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- National Science Foundation
- Society for Biomaterials
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TECHNICAL PROGRAM GRID

MONDAY		TUESDAY		WEDNESDAY		THURSDAY	
	PM	AM	PM	AM	PM	AM	
Industrial Energy Reduction: Materials Opportunity Analysis		The Role of Technology in the Global Primary Aluminum Industry Today and in the Future		Products, Services, Suppliers Showcase			2000
Cast Shop Technology: Cast Shop Safety	Cast Shop Technology: Aluminum Melting: Strategies and Sourcing	Cast Shop Technology: Aluminum Melting: Furnace Design and Refractories	Cast Shop Technology: Melt Treatment: Degassing and Filtration	Cast Shop Technology: DC Casting: Melt Flow and Cooling	Cast Shop Technology: DC Casting: Microstructure and Hot Tearing	Cast Shop Technology: Foundry	2001
Aluminum Alloys For Packaging	Cast Shop Technology: Melt Treatment: Fluxing, Alloying and Grain Refinement				Aluminum Reduction Technology: Cell Stability		2002
Aluminum Reduction Technology: Environmental and Modernization	Aluminum Reduction Technology: Cell Development & Operations - Part 1		Aluminum Reduction Technology: Cell Development & Operations - Part 2	Aluminum Reduction Technology: Pot Control	Aluminum Reduction Technology: Emerging Technologies	Aluminum Reduction Technology: Fundamentals	2003
Magnesium Technology 2005: Magnesium, Primary Production and Environmental	Magnesium Technology 2005: Magnesium and Alloys - Refining, Recycling and Fundamentals	Magnesium Technology 2005: Thermodynamics (Magnesium Alloys)	Magnesium Technology 2005: Magnesium Alloy Development	Magnesium Technology 2005: Magnesium Alloy Processing	Magnesium Technology 2005: Creep Resistant Magnesium Alloys and Welding-Joining	Magnesium Technology 2005: Corrosion and Surface Finishing - Magnesium Alloys	2004
Alumina and Bauxite: Industry Trends and Developments & Bayer Process Chemistry Part I	Alumina and Bauxite: Bayer Process Chemistry Part II & HES and Control & Modelling	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Rapid Prototyping	Alumina and Bauxite: Influences of Alumina on Smelter Performance	Alumina and Bauxite: Bauxite and Bayer Process Red Side	Alumina and Bauxite: Precipitation	Alumina and Bauxite: Alumina Quality	2005
Magnesium Technology 2005: Wrought Magnesium Alloys I	Magnesium Technology 2005: Wrought Magnesium Alloys II	Magnesium Technology 2005: Wrought Magnesium Alloys III	Automotive Alloys 2005: Session I	Automotive Alloys 2005: Session II	Automotive Alloys 2005: Session III	General Abstracts: Mechanical Behavior—Quasi-Static Loading	2006
Carbon Technology: Anode Raw Materials	Carbon Technology: Green Anodes	General Abstracts: Nanostructured and Lightweight Materials		Carbon Technology: Anode Baking	Carbon Technology: Cathode Materials and Corrosion I	Carbon Technology: Cathode Materials and Corrosion II	2007
Shape Casting – The John Campbell Symposium: Liquid Metal Quality	Shape Casting – The John Campbell Symposium: Filling and Feeding	Shape Casting – The John Campbell Symposium: Solidification	Shape Casting – The John Campbell Symposium: Structure and Properties	Shape Casting – The John Campbell Symposium: Modeling	Shape Casting – The John Campbell Symposium: Applications		2008
Globalization of Materials R&D	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Keynote Session	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Forming	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Processing	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Novel Processes I	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Novel Processes II	6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Bulk Metal Processing	2009

TECHNICAL PROGRAM GRID

	MONDAY		TUESDAY		WEDNESDAY		THURSDAY
	AM	PM	AM	PM	AM	PM	AM
2010	Characterization of Minerals, Metals and Materials: Extraction and Processing Applications	Characterization of Minerals, Metals and Materials: Characterization of Structural Engineering Materials – I	Characterization of Minerals, Metals and Materials: Characterization of Structural Engineering Materials – II	Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials – II	Characterization of Minerals, Metals and Materials: Materials Testing and Evaluation	Characterization of Minerals, Metals and Materials: Materials Preparation and Characterization	
2011	General Abstracts: Electronic Materials	General Abstracts: Composites and Coatings	General Abstracts: Advances in Steels	General Abstracts: Environmental Damage and Durability	Recycling - General Sessions: Aluminum and Consumer Goods Recycling	Recycling - General Sessions: Non-Ferrous Recycling	Recycling - General Sessions: Post-Consumer Recycling
2012	General Abstracts: Temperature Treatments and Casting	Characterization of Minerals, Metals and Materials: Characterization of Industrial Products	Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials – I	Metallurgical Technology for Waste Minimization: Session I	Metallurgical Technology for Waste Minimization: Session II	Metallurgical Technology for Waste Minimization: Session III	
2014	Arsenic Metallurgy: Fundamentals & Applications: Plenary Session	Arsenic Metallurgy: Fundamentals & Applications: Removal of Arsenic and its Precipitation from Process Streams I	Arsenic Metallurgy: Fundamentals & Applications: Thermodynamics and Pyrometallurgy	Arsenic Metallurgy: Fundamentals & Applications: Removal of Arsenic and its Precipitation from Process Streams II	Arsenic Metallurgy: Fundamentals & Applications: Process Metallurgy		
2016	Converter and Fire Refining Practices: Plenary	Converter and Fire Refining Practices: Operations and Modernization	Converter and Fire Refining Practices: Process Improvements and Anode Casting	Converter and Fire Refining Practices: Processing Fundamentals	Converter and Fire Refining Practices: Advanced Technologies	TMS Featured Presentations	
2018	Extractive Metallurgy: Pyrometallurgy I	Extractive Metallurgy: Hydrometallurgy	Extractive Metallurgy: Recycling and Waste Minimization	Extractive Metallurgy: Copper	Extractive Metallurgy: Pyrometallurgy II		
2020	Frontiers in Solidification Science: Morphological Evolution and Mushy Zone Phenomena I	Frontiers in Solidification Science: Morphological Evolution and Mushy Zone Phenomena II	Frontiers in Solidification Science: Nucleation	Frontiers in Solidification Science: Crystal-Melt Interfaces: Fundamental Properties and Related Behavior – and – Poster Session	General Abstracts: Mechanical Behavior—Dynamic Loading		
2022	Precious Metals: Au, Ag, Pt, Pd, Os, Rh, Ir, Ru	Surface Engineering in Materials Science III: Laser Processing for Surface Modification	Surface Engineering in Materials Science III: Nanocoatings	Surface Engineering in Materials Science III: Thin Films	Surface Engineering in Materials Science III: Characterization of Surfaces and Films/Coating	Surface Engineering in Materials Science III: Plasma Processing for Surface Modification	Surface Engineering in Materials Science III: Coating Properties and Processing Effects
2024	Mechanical Behavior of Thin Films and Small Structures: Strengthening Mechanisms at Small Length Scale	Mechanical Behavior of Thin Films and Small Structures: Plasticity and Deformation Mechanisms at Small Length Scale	Mechanical Behavior of Thin Films and Small Structures: Stability, Strain and Stress	Mechanical Behavior of Thin Films and Small Structures: Fatigue, Fracture, and Reliability of MEMs and Thin Structures I	Mechanical Behavior of Thin Films and Small Structures: Fatigue, Fracture, and Reliability of MEMs and Thin Structures II	Mechanical Behavior of Thin Films and Small Structures: Advanced Characterization Techniques	
3000	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Dislocation Mechanics of Plasticity	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Impression and Indentation Testing	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Diffusion and Atomistic Modeling	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Microstructure and System Stability	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Mechanics of Nanostructures	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Fatigue, Fracture and Failure	Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday: Thin Films and Multilayers – and – Shock Compression
3001	Materials Processing Fundamentals: Solidification & Casting	Materials Processing Fundamentals: Liquid Metal Processing	Materials Processing Fundamentals: Smelting and Refining I	Materials Processing Fundamentals: Smelting and Refining II	Materials Processing Fundamentals: Powders, Composites & Coatings	Rare Earths, Science, Technology, and Applications V: Reactive Metal Processing	Rare Earths, Science, Technology, and Applications V: Rare Earths

TECHNICAL PROGRAM GRID

MONDAY AM	MONDAY PM	TUESDAY AM	TUESDAY PM	WEDNESDAY AM	WEDNESDAY PM	THURSDAY AM	
Phase Transformations Within Small-Size Systems: Thermodynamics, Phase Equilibria and Kinetics	Phase Transformations Within Small-Size Systems: Order-Disorder Transformations	Phase Transformations Within Small-Size Systems: Phase Separation, Precipitation and Displacive Transformations	Phase Transformations Within Small-Size Systems: Magnetic and Structural Transformations	Phase Transformations Within Small-Size Systems: Amorphous to Nanocrystal Transformations	Phase Transformations Within Small-Size Systems: Transformations in Thin/Thick Films and Multilayers		3002
The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session I	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session II	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session III	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session IV	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session V	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session VI	The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session VII	3003
Neutron Diffraction Characterization of Mechanical Behavior: Facilities, Techniques, and Capabilities	Neutron Diffraction Characterization of Mechanical Behavior: Deformation I	Neutron Diffraction Characterization of Mechanical Behavior: Deformation II	Neutron Diffraction Characterization of Mechanical Behavior: Deformation III	Neutron Diffraction Characterization of Mechanical Behavior: Residual Stress I	Neutron Diffraction Characterization of Mechanical Behavior: Residual Stress II	Neutron Diffraction Characterization of Mechanical Behavior: Phase Transformation	3004
Computational Thermodynamics and Phase Transformations: Grain Boundaries and Interfaces I	Computational Thermodynamics and Phase Transformations: Materials Design and Development	Computational Thermodynamics and Phase Transformations: Grain Boundaries and Interfaces II	Computational Thermodynamics and Phase Transformations: Atomistic and Ab Initio Methods	Computational Thermodynamics and Phase Transformations: Theory and Simulation of Alloys	Computational Thermodynamics and Phase Transformations: Thermodynamic Models and Databases	Computational Thermodynamics and Phase Transformations: Phase Field Models and Related Methods	3005
Bulk Metallic Glasses: Processing and Fabrication I	Bulk Metallic Glasses: Processing and Fabrication II	Bulk Metallic Glasses: Fatigue and Fracture	Bulk Metallic Glasses: Shear Banding and Deformation	Bulk Metallic Glasses: Corrosion, Oxidation and Phase Transformation	Bulk Metallic Glasses: Mechanical Behavior	Bulk Metallic Glasses: Mechanical Behavior and Phase Transformation	3006
Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Analysis of Interdiffusion Microstructures: Session I	Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Analysis of Interdiffusion Microstructures: Session II	Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Experimental Methods for Determining Diffusion Mechanisms	Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Diffusion in Oxide Systems	Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Computational Tools for Understanding Diffusion Mechanisms	Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Applications of Multicomponent Multiphase Diffusion		3007
Hume Rothery Symposium: The Science of Complex Alloys	Hume Rothery Symposium: The Science of Complex Alloys	Hume Rothery Symposium: The Science of Complex Alloys	Hume Rothery Symposium: The Science of Complex Alloys	Powder Metallurgy Research and Development in the Transportation Industry: Titanium Alloys - P/M Developments	Powder Metallurgy Research and Development in the Transportation Industry: Sintering and Densification - P/M Processing	Powder Metallurgy Research and Development in the Transportation Industry: Nano-Matls., Inter-metallics, Amorphous and Composites - P/M Developments	3008
Biological Materials Science and Engineering: Biological Materials I	Biological Materials Science and Engineering: Biological Materials II	Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications I	Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications II	Biological Materials Science and Engineering: Biological Materials Characterization and Biomimetics I	Biological Materials Science and Engineering: Biological Materials Characterization and Biomimetics II		3009
Texture and Microstructure in Thin Films and Coatings: Copper Metallization	Texture and Microstructure in Thin Films and Coatings: Techniques and Coatings	Texture & Microstructure in Thin Films & Coatings: Coatings	Refractory Metals in Electronic Applications: Joint Session with Texture and Microstructure in Thin Films and Coatings: Texture and Thin Films	Refractory Metals in Electronic Applications: Applications	Refractory Metals in Electronic Applications: Processing and Properties		3010
Microstructural Processes in Irradiated Materials: Modelling Defect Evolution	Microstructural Processes in Irradiated Materials: Modelling Defect Evolution and Oxide Dispersion Strengthened Alloys	Microstructural Processes in Irradiated Materials: RPV Embrittlement and Oxide Dispersion Strengthened Alloys	Microstructural Processes in Irradiated Materials: Microstructure Evolution and Segregation – and - Poster Session	Microstructural Processes in Irradiated Materials: He/H Interactions and Ferritic/Martensitic Steels	Microstructural Processes in Irradiated Materials: Carbides, Nitrides and Oxides	Microstructural Processes in Irradiated Materials: Mechanical Behavior of Irradiated Materials	3011

TECHNICAL PROGRAM GRID

	MONDAY		TUESDAY		WEDNESDAY		THURSDAY
	AM	PM	AM	PM	AM	PM	AM
3012	Computational Aspects of Mechanical Properties of Materials: Atomistic Methods	Computational Aspects of Mechanical Properties of Materials: Atomistic Scale Modeling	Computational Aspects of Mechanical Properties of Materials: Nano-Scale and Meso-Scale Modeling	Computational Aspects of Mechanical Properties of Materials: Meso-Scale and Continuum Modeling	Materials Issues for Advanced Nuclear Systems: Materials for Gen IV and Space Nuclear Systems	Materials Issues for Advanced Nuclear Systems: Materials for Nuclear Waste Storage	Materials Issues for Advanced Nuclear Systems: Materials Compatibility
3014	Lead Free Solder Implementation: Reliability, Alloy Development, New Tech.: Interfacial Reactions and Phase Stability in Lead Free Solder Alloys	Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Intermetallic Growth in Lead-Free Solder Joints	Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Lead-Free Solder Alloy Development	Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Electro-migration, & Electrical "Aging" of Lead-Free Solder Joints	Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Thermal Fatigue and Reliability of Lead-Free Solder Joints	Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Mechl. Properties of Lead-Free Solder Alloys and Solder Joints	
3016	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Magnetic and Semiconducting Materials	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Thin Film Stability and Reactions, Electro- and Thermomigration Phenomena	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Aging, Crystallographic Texturing and Characterization of Solder Joints	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Phase Equilibria, Interfacial Energy and Wetting Phenomena in Solder Joints	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Interfacial Reactions and IMC Formation in Solder Joints	Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Effects of Alloying Additions on the Microstructural Evolution of Solders and Solder Joints	
3018	Corrosion Sensors and Monitoring	Applications and Fundamentals of High Aspect Ratio Nanomaterials: Simulation & Control of Carbon Nanotube Formation	Applications and Fundamentals of High Aspect Ratio Nanomaterials: Inorganic Nanostructures	Applications and Fundamentals of High Aspect Ratio Nanomaterials: Applications of Carbon-Based and Inorganic Nanostructures	Applications and Fundamentals of High Aspect Ratio Nanomaterials: Nanostructured Composites	Applications and Fundamentals of High Aspect Ratio Nanomaterials: Monitor and Control of Nanostructure Synthesis	
3020	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Nanostructures and Nanocomposites I	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Nanostructures and Nanocomposites II	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Thin Films, Coatings and Nanostructures	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Semiconductors	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Advanced Technology and Applications I	Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Advanced Technology and Applications II	
3022	Functional Thin Films for Sensors: The Physics and Applications of Functional Thin Films in Sensors	Functional Thin Films for Sensors: Novel Synthesis Methods and Applications of Functional Thin Films	Neutron Scattering in Materials Research: Diffraction, Phases, and Micromechanics	Neutron Scattering in Materials Research: Diffraction: Instruments and Nanostructure	Neutron Scattering in Materials Research: Dynamics and Inelastic Scattering	Neutron Scattering in Materials Research: Diffusion and Other Processes	
3024	The Langdon Symposium: Flow and Forming of Crystalline Materials: Creep	The Langdon Symposium: Flow and Forming of Crystalline Materials: High Temperature Deformation Including Superplasticity	The Langdon Symposium: Flow and Forming of Crystalline Materials: Grain Boundary Properties and Severe Plastic Deformation	The Langdon Symposium: Flow and Forming of Crystalline Materials: Equal Channel Angular Pressing – and – Poster Session	The Langdon Symposium: Flow and Forming of Crystalline Materials: Ultrafine-Grained Materials I	The Langdon Symposium: Flow and Forming of Crystalline Materials: Ultrafine-Grained Materials II	
↓ MARRIOTT HOTEL ↓							
Nob Hill A/B	Superalloys and Coatings for High Temperature Applications: Bond-Coat Technologies – I	Superalloys and Coatings for High Temperature Applications: Bond-Coat Technologies – II	Superalloys and Coatings for High Temperature Applications: Oxidation Behavior – I	Superalloys and Coatings for High Temperature Applications: Ceramic Materials for TBCs	Superalloys and Coatings for High Temperature Applications: Superalloys – I	Superalloys and Coatings for High Temperature Applications: Superalloys – II	Superalloys and Coatings for High Temperature Applications: Superalloys - III
Nob Hill C/D	Friction Stir Welding and Processing III: Aluminum Alloys	Friction Stir Welding and Processing III: High-Temperature Materials	Friction Stir Welding and Processing III: Friction Stir Processing	Friction Stir Welding and Processing III: Process/Applications	Friction Stir Welding and Processing III: Modeling	Friction Stir Welding and Processing III: Microstructure and Texture	
Salon 10/11	Beta Titanium Alloys of the 00's: Applications I	Beta Titanium Alloys of the 00's: Applications II	Beta Titanium Alloys of the 00's: Phase Equilibria	Beta Titanium Alloys of the 00's: Composites and Processing	Beta Titanium Alloys of the 00's: Corrosion and Biomedical	Beta Titanium Alloys of the 00's: Mechanical Response	

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TECHNICAL PROGRAM

Moscone West Convention Center; San Francisco, California USA; February 13-17, 2005

MONDAY

Alumina and Bauxite: Industry Trends and Developments

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday AM Room: 2005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Victorio Siqueira, CVRD, Alumina Business, Rio de Janeiro, RJ 20030 900 Brazil

8:30 AM
Bauxite Mining Sustainably: *Patrick Riley Atkins*[1]; [1]Alcoa, Inc., 390 Park Ave., New York, NY 10022 USA

Beginning in 1990, The international Aluminum Institute began a program to report on the bauxite mineing and rehabilitation activities of the worldwide industry. A survey process was initiated and reports were published in 1992, 1998 and 2004. The recently published 2004 report includes extensive data on mines that represent over 70% of the world's output of bauxite. This paper describe the latest report and will focus on the mine rehabilitation technologies,progress and performance, on company/community interactions and the sustainability goals of the industry.

8:55 AM
Bauxite Supply to the Aluminum Industry - An Update and New Perspective: *William Boyd Morrison*[1]; [1]William B. Morrison & Associates, 3115 Bayo Vista, Alameda, CA 94501 USA

Decisions on bauxite feed sources affect and direct the very foundation of the alumina refinery siteing process. The chemical and mineralogical composition of the bauxite feed determine process configuration and facility design as well as overall project economics. This drive for an assured source of "quality" bauxite is an ongoing one that never ceases. Existing refineries are continually confronted with bauxite supply issues. These issues dictate the needs to a refinery for: Utilization of the geologically best possible sources located at the best geographic position. Delivery at a reasonable cost. Consistency in processability. Stability in supply. Minimization of environmental risk. More and more the emphasis today for existing alumina refineries is on the word "assured." As the industry's alumina refineries age, so do their bauxite sources. What was once good, cheap, and abundant may not be so now. It should be noted that no new "world class" sources on bauxite have been developed recently. This paper revisits a previous study performed five years ago by again focusing upon the bauxite supply situation as it exists in the industry today. The review covers both existing bauxite sources as well as potential new ones. In doing so it addresses bauxite requirements from the refinery perspective and bauxite production from the source perspective. Differing from the previous paper additional emphasis is placed upon the needs and requirements associated with the initial efforts to identify and confirm potential sources of bauxite through more comprehensive, reliable and target oriented geological analysis. The following will focus upon the bauxite supply situation. Identify where supply problems will emerge if they have not already arisen. Revisit the potential for developing new sources. Evaluate these sources much more extensively from a geological perspective. Look at the economic and political issues of supply and finally update the former cost comparison between existing and potential bauxite sources.

9:20 AM
Greenfield Dilemma - Innovation Challenges: *Peter-Hans ter Weer*[1]; [1]TWS Services and Advise, Imkerweg 5, Huizen 1272 EB The Netherlands

After a long period without greenfield projects, the Aluminium industry is currently considering several greenfield Bauxite/Alumina opportunities worldwide. Many of these potential projects however have difficulty to meet threshold criteria used by the industry for their economic evaluation. This paper provides an insight in aspects of this issue, and it compares the economics of brownfield with greenfield Bauxite/Alumina projects. It concludes with suggested directions that could be explored to improve greenfield project economics.

9:45 AM
Complex System of Long-Term Forecasting of World Markets of Primary Aluminium and Alumina: *B. Arlyuk*[1]; [1]Alumconsult Ltd., 2, Shkiperski protok, St. Petersburg 199106 Russia

The complex system of the long-term forecast of the markets of primary aluminium and metallurgical alumina, taking into account their mutual connections and influence is developed. The system is based on the analytical description of the market connections determining a sales volume and the prices of aluminium and alumina. It has allowed to limit the amount of empirical factors in comparison to usually used econometric approach and has provided the comprehensible significance of model. For an estimation the consumption of primary aluminium at the West the forecasts of development the economics in the Western countries is used, which is characterized by cyclic change of indexes of industrial production. The degree of capacity utilizations of smelters and refineries or the volume of supply of the market in turn depends on a level of the world prices for aluminium and spot prices for alumina. Thus, the system of the forecast takes into account presence of a plenty feedback as inside subsystems of the forecast of the market of aluminium, and also alumina, and between these markets. The developed complex system allows to forecast a degree of capacity utilization of aluminium smelters and alumina refineries in Western countries, balance of supply/consumption of metallurgical alumina and aluminium, alumina spot and contract prices and prices of primary aluminium at LME. Thus as the initial information the forecast of input of new capacities and change of the Western world industrial production are used. Identification of the developed system on the actual quarter data for the period since 1990 till 2004 is executed and the empirical factors of model providing forecasting of the world markets of aluminium and alumina with comprehensible accuracy are determined. The developed system is used for a long term forecasts of the world markets of primary aluminium and metallurgical alumina up to 2015.

10:10 AM Break

10:20 AM
Fundamental Research on Alumina Production of the Future in China: *Qingjie Zhao*[1]; *Qiaofang Yang*[1]; [1]Zhengzhou Research Institute Aluminium Corporation of China, Ltd, R&D Dept., 82,Jiyuan Rd., Shangjie Dist., Zhengzhou, Henan 450041 China

Analyzed is the present research situation on production technology and fundamental theory in the alumina industry, and discussed is the future research situation on it. It is proposed that the developing trends of alumina production technology depends on the two factors, one is that the great achievements obtained should be popularized to intensify the process and reduce energy consumption and cost, the other is that new process and technology should be developed rapidly. The core of the fundamental theory is to provide theoretical and technological support for simplifying the process flow, improving efficiency, reducing the enery consumption and optimizing the production target.

10:45 AM
The Comprehensive Energy Saving in China Alumina Industry: *Lijuan Qi*[1]; *Songqing Gu*[1]; [1]Zhengzhou Research Institute Aluminum Corporation of China, Ltd., R&D Dept., 82,Jiyuan Rd., Shangjie Dist., Zhengzhou, Henan 450041 China

In this paper, analysed are both the present energy consumption of chinese alumina production and the factors affecting energy consumption. The trends of energy consumption in the Chinese alumina production are also proposed, and the key technologies to be developed

and the main measurements to be taken are also put forward in the future Chinese alumina production.

Alumina and Bauxite: Bayer Process Chemistry: Part I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday AM Room: 2005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Robert Bitsch, Alcan, Bauxite & Alumina, Cedex, Gardanne, 13541 France

11:10 AM
Layered Double Hydroxides in the Bayer Process: Past, Present and Future: *Steven Philip Rosenberg*[1]; Lyndon Armstrong[2]; [1]BHP Billiton, Bauxite & Alumina Tech. Ctr., PO Box 344, Collie, Western Australia 6225 Australia; [2]Alcan, Queensland R&D Ctr., PO Box 883, Kenmore, Queensland 4069 Australia

Alumina refiners have been aware of some of the potential uses of Layered Double Hydroxides (LDH's) in the Bayer process for more than a decade. By virtue of the lamellar structure, ability to adjust the distance between these layers, and the reactivity of the interlayer region, LDH's can be used for the controlled addition or removal of a variety of species, both organic and inorganic. In this paper, we review the history and present use of LDH's in alumina refining, particularly the most commonly used calcium and magnesium aluminate varieties. We also examine some of the ways in which these LDH's can be produced in the refinery, both intentional and unintentional, as well as some of the possible reasons thay have not found more widespread application. Finally, we discuss how LDH technology could be applied to deal with emerging trends and issues in alumina refining, particularly in reducing the environmental impact of alumina refining.

11:35 AM
Improving the Bayer Process by Power Ultrasound Induced Crystallization (Sonocrystallization) of Key Impurities: *Graham Ruecroft*[1]; David Hipkiss[1]; Martin Fennell[1]; Linda McCausland[1]; [1]Accentus plc, C3 Technology, 551 Harwell Business Ctr., Didcot, Oxfordshire OX11 0QJ UK

Whilst it is known that precipitation of sodium oxalate in supersaturated solutions can be triggered by adding recycled seed crystals of the same to act as initiator, in practice it is found that the surfaces of the crystals become poisoned by other organic materials present in the liquor, and become inactive as crystal growth initiators. The C3 proprietary Sonocrystallization technology [power ultrasound applied to assist crystallization] works by significantly increasing the frequency of nucleation events in the waste liquor stream compared to current operation. Each nucleation event gives rise to a site for an impurity crystal to form. Consequently, the rate of impurity crystal formation and subsequent removal is greatly enhanced. Importantly, the issue of other organic contaminants inhibiting impurity crystal growth and formation, and therefore their removal, is eliminated.

12:00 PM
Theoretical Research on the Precipitation of Sodium Aluminate Solutions Enhanced by Ultrasound: Shugui Hua[1]; [1]Central South University, College of Chmst. & Cheml. Engrg., Cangsha, Hu'nan China

The heat of formation and frontier orbital of possible monomeric aluminate species, hydroxyl radical(¡¤OH), hydrogen radical(¡¤H) and the products of some reactions in caustic aluminate solutions under ultrasound were computed with a semi-empirical quantum chemical method AM1. According the thermodynamics theory and frontier orbital argument, the structure changes of the solution under ultrasound were studied. Conclusions were made that ultrasound can enhance the formation of the growth unit of crystallization [Al(OH)4£-(H2O)2] and accelerate the precipitation of caustic aluminate solution.

Aluminum Alloys For Packaging

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA; Gyan Jha, ARCO Aluminum Inc, Louisville, KY 40223-4032 USA

Monday AM Room: 2002
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Lexington, KY 40511 USA; Gyan Jha, ARCO Aluminum Inc., Louisville, KY 40223-4032 USA

8:30 AM
Aluminium Short Term Price Forecast. Speculations on Forwards, Hedging the Sales: *B. Arlyuk*[1]; [1]Alumconsult Ltd., St. Petersburg 199106 Russia

The system has been developed for quantitative and qualitative forecasting of primary aluminium prices at LME up to 3 months ahead using LME daily parameters, as well as monthly statistic data on industrial production indices in Western countries, IP and Dow Jones indexes at USA. The prices forecasting is the basis of the probability approach for determining optimal moments and volumes of transactions with three-month forwards securing maximum profits. The system has been tested by issuance of daily recommendations to a broker for opening and closing of three-month forward positions at LME during four years since July 2000 till June 2004, the pledge initial volume was $1 M and capitalization 80% realized profit for expansion the investments. As optimal is using the limitation for opening new positions 50-55% of pledge to broker. In the average the system is opening new positions of forwards about 60 times per year. The full fixed profit amounted to 250% annually to the initial investments. The system was used during two years for hedging the sales of aluminium within the quarter forward. It gives the opportunity to increase the price of sales by 50 $/t.

9:00 AM
Investigation of Score Corrosion in Aluminum Can Ends: *Gyan Jha*[1]; W. Yin[2]; [1]Arco Aluminum, 9960 Corp. Campus, Ste. 3000, Louisville, KY 40223 USA; [2]SECAT, 1505 Bull Lea Rd., Lexington, KY 40511 USA

Beverage can ends have utilized vinyl and epoxy coatings to protect the Aluminum from corrosion. The coated Aluminum is post lubricated then fabricated into an end. The coating must withstand the fabrication process without any coating adhesion loss, scuffing and coating fracture. In recent years pitting type corrosion has been observed at the score on Aluminum can ends. Score corrosion can lead to premature failure of the Aluminum can end. This paper will discuss the various factors that can effect score corrosion in Aluminum can ends.

9:30 AM
A Mossbauer Study of 3000 and 5000 Series Packaging Alloys: *Gyan Jha*[1]; S. Jha[2]; W. Yin[3]; Ameer Lahamer[4]; [1]Arco Aluminum, 9960 Corp. Campus, Ste. 3000, Louisville, KY 40223 USA; [2](Retired) University of Cincinnati, Physics, Cincinnati, OH 45208 USA; [3]SECAT, 1505 Bull Lea Rd., Lexington, KY 40511 USA; [4]Berea College, Physics, Berea, KY 40404 USA

Understanding Fe and its surroundings in Aluminum alloys 3104 and 5182 has become more important because of the different types of scrap inputs used to cast these alloys. Fe can have an effect on texture and formability of the final rolled product. It is difficult to characterize the nature of Fe with conventional analysis techniques. The study will present results of the analysis of commercial Aluminum packaging alloys by the Mossbauer technique.

10:00 AM
Simulation of Texture-Dependent Recrystallization in 1050 Aluminum: *Anthony D. Rollett*[1]; Joseph Fridy[2]; Hasso Weiland[2]; Jaakko Suni[2]; Abhijit Brahme[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Alcoa Technical Center, Alcoa Techl. Ctr., PA 15609 USA

In order to verify that we understand the recrystallization process at the microstructural level, Monte Carlo simulation of recrystallization has been undertaken using statistically reconstructed input microstructures to represent the deformed material. This approach has already revealed many gaps in our knowledge of the deformed state, grain boundary properties and so on. A key part of the simulation procedure has been construction of 3D representations of the as-deformed microstructure. The most relevant features are the crystal-

lographic orientations of the grains ("texture") and the size, topology and shape of the grains. In order to perform realistic simulations one needs to specify the initial state of the material with sufficient detail that all these features are reproduced. Cross-sections or surfaces were characterized through automated electron back scatter diffraction (OIM) in the scanning electron microscope (SEM). This approach has shown the importance of capturing the aspect ratio of the deformed grains and the tendency of nuclei to cluster near the prior grain boundaries in order to correctly reproduce the recrystallization kinetics. Work is ongoing to understand the factors that control the marked growth in the cube texture component during recrystallization. Supported in part by the US Department of Energy Aluminum Industry of the Future program.

10:30 AM
Texture-Dependent Recrystallization in 1050 and 5005 Aluminum: Mohammed Haroon Alvi[1]; Hasso Weiland[2]; Jaakko Suni[2]; Soonwuk Cheong[2]; *Anthony D. Rollett*[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Alcoa Technical Center, PA 15069 USA

Recrystallization kinetics have been studied in hot rolled 1050 and 5005 with the objective of understanding the local variation in the rate of recrystallization as a function of texture component. The overall objective is to decrease annealing times by exploiting such variations. Automated electron back-scatter diffraction (EBSD, or OIM) has been used to quantify microstructures at various stages of recrystallization. Analysis of the grain orientation spread (GOS) was found to be the most effective method for partitioning EBSD maps into recrystallized and unrecrystallized regions. The greatest density of nuclei is observed in the S texture component and the recrystallization kinetics are consequently fastest in grains that have this orientation. The cube component is weak in the as-deformed state but increases to between 25% and 40%, depending on annealing temperature. In related work, we are using these experimental results to validate a computer model of recrystallization.

11:00 AM
Microstructure Evolution in Twin Roll Cast AA3105 During Thermomechanical Processing: *Naiyu Sun*[1]; Burton R. Patterson[1]; Jaakko Suni[2]; Eider A. Simielli[2]; Hasso Weiland[2]; Larry Allard[3]; [1]University of Alabama, Matls. Sci. & Engrg., 1530 3rd Ave. S., BEC 254, Birmingham, AL 35294 USA; [2]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6064 USA

This presentation gives an overview of microstructural evolution during thermomechanical processing of twin roll cast (TRC) AA3105, including homogenization, cold rolling and annealing. The resultant processes of constituent particle coarsening, dispersoid precipitation and recrystallization, and their interaction during various annealing conditions are discussed. Time-temperature-transformation curves were determined for precipitation and recrystallization to understand the interaction of these processes. It was found that homogenization prior to cold rolling had a profound effect on recrystallization kinetics, grain size and gradient, as did homogenization time, temperature and cooling rate. Recrystallization kinetics also increased with decreased casting rate, and increased annealing temperature and heating rate.

11:30 AM
HRTEM Study on the Eutectoid Phase Transformation of the Intermetallic Particles from Al6(Fe,Mn) to α-Al(Mn,Fe)Si Phase in AA3003 Alloy: *Yanjun Li*[1]; Arne Olsen[1]; [1]University of Oslo, Ctr. of Matls. Sci. & Nanotech., Dept. of Physics, PB 1126, Blindern, Oslo 0318 Norway

The phase transformation from Al6(Fe,Mn) to α-Al(Mn,Fe)Si phase in a DC-cast 3003 alloy has been studied by TEM and HRTEM at the early stage of the transformation. The 3-D morphology of the transformed α-Al(Mn,Fe)Si particle containing many cylindrical Al channels has been observed by dark field TEM image. The orientation relationships between the Al6(Fe,Mn), Al-spots, α-Al(Mn,Fe)Si phase, and the surrounding Al matrix has been studied. It has been found that the transformed α-Al(Mn,Fe)Si phase has similar orientation relationships with the matrix as the α-Al(Mn,Fe)Si dispersoids precipitated during heat treatment. Semi-coherent interfaces between the transformed α-phase and the Al matrix and between the Al-spots and the surrounding α-phase have been observed by HRTEM. It suggests that the α-phase has heterogeneously nucleated from the aluminium matrix on the interface between aluminium and Al6(Fe,Mn) particle and the Al-spots has nucleated from the α-phase during the phase transformation.

Aluminum Reduction Technology: Environmental and Modernization

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday AM — Room: 2003
February 14, 2005 — Location: Moscone West Convention Center

Session Chair: Margaret M. Hyland, University of Auckland, Dept. of Cheml. & Matls. Engrg., Auckland New Zealand

8:30 AM Invited
Environmental Regulations and Performance for European Smelters: *Eirik Nordheim*[1]; [1]European Aluminium Association, 12, Ave. de Broqueville, Brussels 1150 Belgium

European aluminium smelters are under pressure from tighter environmental regulations and also increasingly Environmental Quality Standards. Emission limits to air, water and waste deposits are set based on common EU regulations. These will generally be framework regulations, leaving the Member States to set detailed limits. But some regional groups like OSPARCOM have in addition to this recommended specific emissions limits to air and water. Increasingly also the Air Quality Standards and Water Quality Standards currently being set at EU level will have an impact on the emission limits set for individual plants, due to the contribution from smelter emissions on the local environment. The presentation will look at the relevant emission limits and Enviromental Quality Standards and where the smelters are in relation to this.

8:55 AM
PFC Emissions Performance for the Global Primary Aluminum Industry: *Jerry Y. Marks*[1]; [1]International Aluminium Institute, 312 NE Brockton Dr., Lees Summit, MO 64064 USA

The global primary aluminum industry, through its new Global Aluminum Sustainable Development Initiative (GADSI), has set a number of challenging environmental targets. Greenhouse Gas Emissions Reduction is one of the focal points of the initiative. A global industry perfluorocarbon (PFC) specific emissions reduction target of 80% has been set for 2010 relative to 1990. To monitor PFC emissions reductions an annual survey of anode effect performance is carried out by the International Aluminium Institute (IAI). IAI is working with a number of world organizations to assure the homogenous application of accurate and transparent GHG inventory methodology. This paper will discuss the results of the anode effect performance surveys through 2003, the latest year for which survey information is available, and will examine benchmark performance by reduction technology. The paper also discusses progress in efforts aimed at establishing common GHG calculation and inventory methods.

9:20 AM
Environmental Challenges in the Prebake Line at Hydro Aluminium Karmøy: *Jørn Tonheim*[1]; Knut Arne Paulsen[1]; Roald Holten[1]; Arnt Helge Fidjeland[1]; Harald Martinsen[1]; Kirsten Louise Bolstad Halvorsen[1]; [1]Hydro Aluminium Karmøy, N-4265 Håvik Norway

It is a challenge for aluminium smelters to reduce emissions and to keep a good working environment. There are still cases of occupational asthma, even in the most modern aluminium smelters. In the Prebake line at Hydro Aluminium Karmøy the emissions of fluoride and dust are within the threshold limits set by the Government according to the OSPAR convention. However, the limits regarding dust and fluoride in the working environment have often been exceeded during anode change. To reduce these emissions and improve the working environment, different measures have been carried out. Forced suction was implemented on 14 test pots in spring 2003 and will be implemented in the whole Prebake line by January 2005. Automatic positioning of the anodes after anode change was implemented during the spring in 2004. A test with reduced manual control in conjunction with use of cavity cleaner before setting the new anode has also been performed.

9:45 AM
Understanding Fugitive Fluoride Emissions at Alumar Consortium: *Nilton Freixo Nagem*[1]; Eliezer S. Batista[1]; Ari F. Silva[1]; Valerio A. Gomes[1]; Luciano J.P. Souza[1]; Luis Carlos A. Venancio[1]; [1]Consórcio

de Aluminio do Maranhão - Alumar, Smelter, Br 135, Km 18 - Distrito Industrial de Pedrinhas, São Luis, Maranhão 65095604 Brazil

One of the most control demanding activities in a Smelter is fugitive fluoride emissions (HF) monitoring and control in Potrooms. A study was performed in Alumar Smelter to map and determine the impact of unit operations (anode setting and tapping, among others) in Potrooms fugitive fluoride emissions (HF). This study consisted on a continuous monitoring of gaseous fluoride through the use of an Open-Path Tunable Diode Laser (TDL) equipment and its correlation with some process variables such as bath chemistry, current level and alumina LOI.

10:10 AM Break

10:25 AM
Why the Søderberg Technology Has a Future in Mini-Smelters Intergrating a Coal Fired Power Plant: *Andre Teissier-duCros*[1]; [1]GEANOVERSEAS, Gean-KTD Mini Smelter Proj., 4434 Covington Hwy., Atlanta, GA 30035-1212 USA

A State of the Art Soderberg incorporating all known, proven technologies, equipment and know how regarding emission control will meet future environment, health and safety standards (OSPAR guidelines) and be bankable for a greenfield, especially if combined with a fluidized bed boiler power plant enjoying itself a low CO2 level on top of other low emission performance. Issues to address are CO2, CF4, HF and mostly PAH (operators' safety & health). We will review the "package" of operating procedures, quality of anode paste, process control settings co-ordinating anode movements and point feeding frequency, and specifications of all equipment. This opens a new market for greenfields in countries suffering of high energy cost and fast growing energy demand.

10:50 AM
CD20 Reduction Cell Upgrade for Dubal's Expansion Project: *Yousef Ali AlFarsi*[1]; Abdelhamid Meghlaoui[1]; Najeeba AlJabri[1]; [1]Dubai Aluminium Company Ltd., Tech. Dvlp., PO Box 3627, Dubai United Arab Emirates

Dubai Aluminium Company Limited (DUBAL) has 480 CD20 reduction cells designed for operation at 200 kA. In planning for a plant expansion, the Technology Development function was entrusted the challenge to effect design criteria changes to the CD20 cell, without prototyping, targeting an operating amperage of 220-230 kA in order to increase the production from 560,000 mt/year to 760,000 mt/year. Such changes to the CD20 technology were critical for the capital cost and the operating cost reduction. The learning curve associated with six years of improvements in operational practices, process control and thermal-electrical design validation of the CD20 and CD26 technologies resulted in appropriate improvements of CD20 cell design and control. Modifications to cathode lining and geometry were supported by mathematical modeling and experimental correlation. Dubal code-named the new reduction cell "D20". In 2003, DUBAL installed and commissioned 212 D20 cells that are operating very satisfactorily at 220 kA, including 50 cells with graphitised cathodes operating at 225 kA in a dedicated booster section. This paper covers the development of the D20 cell, as well as one-year key operating parameters.

11:15 AM
Energy Saving in Hindalco's Aluminium Smelter: *S. C. Tandon*[1]; R. N. Prasad[1]; [1]Hindalco Industries Ltd., Renukoot-231 217, Sonebhadra, UP India

The electrolytic production of primary aluminium metal in Hall-Heroult cells is highly energy intensive and accounts for nearly 40% of the production cost. The role of electric current in aluminium electrolysis cell is two fold; one is the electrolysis of alumina to produce aluminium metal as per Faraday's law of electrolysis and other is maintaining thermal balance of electrolytic cells to make them workable which accounts for more specific energy consumption than theoretically required. Hindalco has made serious efforts to modernize its vintage cells of 1950's & other facilities in order to reduce energy consumption and improve the working environment. Various technological developments have been introduced over the years leading to significant changes in cathode design, alumina feeding, cell process control, operating strategies and ancillary equipment resulting in substantially improved technical results. Recent introduction of slotted anode along with few other initiatives have further reduced power consumption by 0,3 kWh/MT Al, leading to an energy consumption below 14,0 DC kWh/MT Al in vintage cells of the 1950's.

11:40 AM
Alro's Creep Capacity Expansion: *Gheorghe Dobra*[1]; Satish Manaktala[1]; Mihail Atanasiu[1]; Constantin Radulescu[1]; Cristian Theodor Stanescu[1]; [1]ALRO, Techl., 116, Pitesti St., Slatina 230104 Romania

Alro, the only aluminium smelter in Romania, was built under the Romanian socialist system in the 1970s. Transition to a market based economic system, clearly showed that the smelter based on Pechiney AP 9 pre-bake technology had to modernise in order to remain competitive and to meet the EU environmental standards. In the 1990s, a major modernisation program at the cost of over $120 million was undertaken to refurbish the plant. At the completion of this program, the Smelter provided an ideal platform for increasing the electrolytic cell capability significantly and much beyond its original design. A test section with booster power, redesigned cathodes and larger anodes was established and the cell performance results confirmed the potential to increase the Smelter capacity by nearly 30 percent and reduce the energy consumption by over 5%. These results have led to establishment of a Smelter creep capacity expansion program due to be completed by the end of 2005. The test section operation methodology and performance results are the subject of this paper.

Arsenic Metallurgy: Fundamentals & Applications: Plenary Session

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Monday AM Room: 2014
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA; Ramana G. Reddy, University of Alabama, Dept. of Metals & Matls. Engrg., Tuscaloosa, AL 35401-0202 USA

8:30 AM
The Removal of Arsenic from Aqueous Solution by Coprecipitation with Fe(III): *Larry G. Twidwell*[1]; Robert G. Robins[2]; Jacob W. Hohn[1]; [1]Montana Tech of University of Montana, Sch. of Mines & Engrg., 1300 W. Park St., Butte, MT 59701 USA; [2]Murdock University, Chmst., Western Australia 6150 Australia

In treating hydrometallurgical solutions and waste streams for the removal of arsenic, the use of coprecipitation with Fe(III) has been specified by the U.S. EPA as the Best Demonstrated Available Technology. This technology has been widely adopted over the last century, and developments have been well reviewed. This paper is aimed at clarifying a number of uncertainties and misconceptions that result from previously published work by many authors. The clarification will include a discussion of the importance of Fe:As mole ratio, initial arsenic concentration, arsenic valence state, the effect of mixed arsenic valence states, system agitation rate, and possible modifications to the ferrihydrite structure on the effectiveness of arsenic removal. A comparison of the effectiveness of arsenic removal by ferrihydrite coprecipitation, post precipitation, and granulated ferrihydrite will be presented. Also, ferrihydrite long term stability will be discussed.

9:00 AM
On the Preparation and Stability of Scorodite: *George P. Demopoulos*[1]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Scorodite is advocated as a viable option for the fixation of arsenic from aqueous process effluents, especially for arsenic-rich and iron-deficient solutions. It has a high arsenic content, it requires stoichiometric amounts of iron, and it has excellent dewatering and disposal characteristics. Because of its high degree of crystallinity and small specific surface area, scorodite is also, very importantly, thought to have high inherent stability at least from a kinetic point of view. As a mineral, scorodite can be found in a wide variety of geological settings. This suggests that it is stable under specific weathering conditions. Hence our interest in designing a cost-effective process for returning arsenic to the environment in this mineral form. In this paper, (1) the preparation of scorodite in lime neutralisation ?type circuits is discussed and (2) the long term stability of scorodite is evaluated in the light of newly generated accelerated ageing kinetic data.

9:30 AM Break

9:45 AM
Arsenic Capacity of Copper Slags: *R. G. Reddy*[1]; J. C. Font[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Arsenic capacity modeling and experimental study between mattes and slags in copper smelting is reviewed. The arsenic capacities of slags are calculated a priori using Reddy-Blander model. The effect of matte and slag compositions, PSO2 and temperatures on arsenic capacities of copper slags is made. The calculated results for arsenic capacities are in good agreement with the available experimental data. Application of this model for the prediction of Arsenic capacities and distribution ratios in mattes and slags, and removal of arsenic in several industrial smelter processes is discussed.

10:15 AM
Arsenic and Old Waste: *Donald A. Robbins*[1]; [1]ASARCO, Inc., Environmental Services, Phoenix, AZ 85016 USA

Asarco has had a long involvement with arsenic. As far back as the turn of the last century, Asarco was a producer of arsenic trioxide. Later, through the efforts of its Central Research Laboratory, value added products such as high purity metallic arsenic and gallium arsenide were produced. During these years of arsenic production, Asarco was a pioneer in industrial hygiene programs for its workers exposed to arsenic. Biological monitoring programs were initiated and criteria developed to guide industrial hygiene and safety personnel in application of respiratory protection, ventilation, personal protective gear, and education for protecting its workers. With the advent of CERCLA and RCRA in 1980 ASARCO's issues related to arsenic became very different. The company became involved in a significant number of remediation sites where work is on going to prevent arsenic and other metals from being released into the environment. These remediation sites are fertile venues for the application of cost effective and creative control technologies particularly for arsenic.

Beta Titanium Alloys of the 00's: Applications I

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Monday AM Room: Salon 10/11
February 14, 2005 Location: San Francisco Marriott

Session Chairs: Henry J. Rack, Clemson University, Matls. Sci. & Engrg., Clemson, SC 29634-0921 USA; Robert D. Briggs, Boeing Company, Boeing Commercial Airplane, Seattle, WA 98027 USA

8:30 AM Opening Comments

8:40 AM
The Use of Beta Titanium Alloys in the Aerospace Industry: *Rodney Raymond Boyer*[1]; Robert David Briggs[1]; [1]The Boeing Co., Metall./6-20J1, PO Box 3707, MS 73-44, Seattle, WA 98124 USA

Beta titanium alloys have been available since the 1950's (Ti-13V-11Cr-3Mo or B12VCA) but significant applications of these alloys - beyond the SR-71 Blackbird - had been slow in coming. The next significant usage of a beta-alloy did not occur until the mid 1980's on the B-1 bomber. This aircraft utilized Ti-15V-3Cr-3Al-3Sn sheet because of the capability for strip rolling, improved formability and higher strength. The next major usage was on a commercial aircraft, the Boeing 777 which made extensive use of Ti-10V-2Fe-3Al high-strength forgings. Ti-15V-3Cr-3Al-3Sn ducting, castings and springs were also used, along with Ti-3Al-8V-6Cr-4Mo-4Zr (Beta-C) springs. More recent work at Boeing has focused on the development of Ti-5Al-5Mo-5V-3Mo, a high strength alloy which can be used at higher strength than Ti-10V-2Fe-3Al and is much more robust - it has a much wider or friendlier processing window. This, along with additional studies at Boeing, and from within the aerospace industry in general will be discussed in detail, summarizing applications and the rationale for the selection of this alloy system for aerospace applications.

9:10 AM
Military Applications for Beta Alloys: *John C. Fanning*[1]; [1]TIMET, Henderson, NV 89009 USA

Beta alloys are potentially useful for numerous non-aerospace military applications. Topics to be discussed in this paper include: evaluation of TIMETAL 15-3 for body armor; fabrication and evaluation of a TIMETAL 21S mortar barrel; use of TIMETAL 15-3 for tactical knives; and resistance of TIMETAL 21S to erosion in a cannon barrel.

9:40 AM
Application of Ti-10-2-3 in Rotor Hub Parts of EUROCOPTER: *G. Antoine*[1]; Jerome Panter[2]; [1]EUROCOPTER; [2]EADS, Corp. Rsch. Ctr., 12 rue Pasteur, Suresnes 92150 France

The need for maximum lightening in highly fatigue stressed parts of helicopters, e.g. hub bodies, blades sleeves, ..., has led for a number of structural parts of the last EUROCOPTER programs to the use of the Beta titanium alloy Ti 10.2.3 in place of steels. This choice which is motivated by the research of the best weight/cost/strength compromise was favourable to Ti10.2.3 due to its great fatigue behaviour and to the fact that it enable to remove the environmental unfriendly cadmium plating needed on conventional steels. This introduction was possible due to joint researches between EUROCOPTER and EADS Corporate Research Center, especially for metallurgical and mechanical characterizations. This paper will present a summary of the work done in the joint research program and will present the different applications of Ti10.2.3 on the last EUROCOPTER programs as EC120 and NH90.

10:05 AM
Implementation of Dynamic Grade Ti-10V-2Fe-3Al Forgings on the RAH-66 Comanche Helicopter: *Michael J. Lutian*[1]; [1]Sikorsky Aircraft Corp., 6900 Main St., PO Box 9729, Stratford, CT 06615-9129 USA

A number of new materials technologies (metallic and non-metallic) were developed and qualified for the Comanche Program. This paper will focus on one of the primary materials technology developments, triple-melt dynamic grade Ti-10V-2Fe-3Al forgings. A summary of the die forging applications being qualified on critical Comanche main rotor system and airframe interface components, ranging from 10 to 900 pounds, is described. First article qualification and lot acceptance forging data from over twenty destructively-tested forgings representing a dozen part numbers and several forging suppliers, forged over a ten year period during the DVP and EMD Phases of the Comanche Program, will be presented. Data will include analysis of several hundred tensile, fatigue, and fracture toughness coupon tests. Results support triple-melt dynamic grade Ti-10V-2Fe-3Al as a forging material possessing a superior, balanced combination of mechanical properties, producible in a wide range of forging sizes, for optimized designs of critical dynamic components.

10:30 AM Break

10:45 AM
Beta Titanium Springs in the 00's: *Charles Pepka*[1]; [1]Renton Coil Spring, PO Box 880, 425 S. 7th St., Renton, Washington 98055 USA

World class engineers in the new millennium will bring the demands, of cost, reliability, lighter weight, consistent supply, longer service life, and lower cost! These points are all interconnected and cannot be considered separately. Lowering product cost alone will not insure a future in the world marketplace. This market is requiring quality, reliability, at a cost. Beta Titanium like 3-8-6-4-4 (Beta C) springs will provide an innovative engineering team with the tools to meet their needs. AMS spec 4957 Wire and 4958 bar specifications are the most widely used Titanium materials certified for springs. Together they give a stable foundation for titanium's usage into the new millennium. Applications for titanium springs are varied. They range form Space, Commercial Aircraft, Military Aircraft, Military Equipment, many forms of racing (Automotive, Motorcycles, quads, etc.) chemical and industrial applications. The reasons for their usage center around a performance envelope consisting of the smallest lightest package for the force required to do the job. Users report an increase in their equipments performance after switching from other materials to Titanium. The presentation will include a design study and actual applications of Titanium springs.

11:15 AM
Production of Beta Alloys by Electron-Beam Single-Melting: *Michelle McCann*[1]; David Tripp[2]; Michael Cardamone[3]; John C. Fanning[1]; Jon Quinn[3]; [1]TIMET, PO Box 2128, Henderson, NV 89009 USA; [2]TIMET, 900 Hemlock Rd., Morgantown, PA 19543 USA; [3]TIMET, 100 Titanium Way, Toronto, OH 43964 USA

Beta alloys provide useful combinations of mechanical and physical properties, but widespread usage is often limited by the relatively high cost. Production by Electron-Beam Single-Melting (EBSM) may provide opportunities for significant cost reduction related to ingot shape, increased scrap utilization and economies of scale. This technology has already enabled the implementation of Ti-6Al-4V in ground combat vehicle systems and other cost-sensitive applications. Since the EBSM production method is substantially different from the traditional vacuum arc re-melting (VAR) production method, extensive testing and evaluation will be performed on the EBSM product to ensure that the properties and quality are equivalent to those of the traditional product. Results of recent studies on the potential production of beta alloys by EBSM will be discussed in this paper.

11:40 AM
Single Melt Beta-C for Spring and Fastener Applications: *K. O. Yu*[1]; E. M. Crist[1]; R. Pesa[2]; N. Cecchini[2]; C. M. Bugle[2]; [1]RMI Titanium Company, PO Box 269, 1000 Warren Ave., Niles, OH 44446-0269 USA; [2]Dynamet Incorporated, Washington, PA USA

Beta-C is a high strength titanium alloy widely used for spring and fastener applications. Spring and fastener input stock is currently manufactured by rolling billets forged from conventional double melt VAR (2 x VAR) ingots. Recent advances in plasma arc melting (PAM) single melt technology offer a potential to reduce the input stock cost by directly rolling the as-cast near net shape PAM ingots instead of the conventional forged billets. A 5" diameter as-cast PAM ingot was rolled to 0.60" bars which were then processed to smaller diameter centerless ground bars for making springs and fasteners. The evaluation of microstructures and mechanical properties of the bar input stock as well as the finished spring and fastener stock will be presented.

Biological Materials Science and Engineering: Biological Materials I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Monday AM Room: 3009
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Marc Andre Meyers, University of California, Matls. Sci. & Engrg., La Jolla, CA 92093-0411 USA; Julian Vincent, University of Bath, Dept. of Mechl. Engrg., Bath BA2 7AY UK

8:30 AM Keynote
Mechanical Properties of Biological Materials: *Julian Vincent*[1]; [1]University of Bath, Dept. of Mechl. Engrg., Bath BA2 7AY UK

It seems to me that the mechanical properties of biological materials are of interest to the engineer for 3 main reasons: What characteristics do they have? Why and how? How can we benefit from this information? The most versatile material is probably the cuticle of arthropods, which has to be skeleton, skin and sensor, providing support, flexibility, sensitivity, protection, waterproofing, absorption, locomotion, etc. In providing this it is impossible to separate structure and material properties. The properties (examples in parentheses) therefore have to be understood at the level of chemical bonding (epitaxy of chitin-protein interactions via silk-like conformations; incorporation of heavy metals), physical chemistry (control of stiffness achieved by control of water content), micro-morphology (fibre orientations; volume fractions), macromorphology (control of buckling by folding stiffeners) and function (wing foldings, mechanisms for drilling holes). The benefit comes from comparing the design philosophy of the arthropod with what we would do given our technical background and experience. These turn out to be very different (there's only a 10% overlap - by design or coincidence) suggesting that 90% of biological problem-solving remains to be explored and exploited. The last part of the talk will therefore be devoted to techniques of biomimetic data-mining and how to organise biological information in a way which will aid creativity and innovation.

9:15 AM Invited
A Materials Science Approach to the Fracture and Fatigue Resistance of Hard Mineralized Tissue: *R. O. Ritchie*[1]; R. K. Nalla[1]; J. J. Kruzic[1]; J. H. Kinney[2]; [1]University of California, Matls. Sci. & Engrg., 381 Hearst Mining Bldg., MC 1760, Berkeley, CA 94720-1760 USA; [2]Lawrence Livermore National Laboratory, 700 East Ave., Livermore, CA 94550 USA

Despite the clinical interest in the fracture resistance of hard mineralized tissues such as bone and dentin, there is only limited mechanistic information available on how these materials derive their toughness, how it is affected by microstructure and orientation, and how behavior is specifically affected by cyclic fatigue loading. In the present study, crack-growth behavior in dentin and bone is characterized using a fracture-mechanics approach. Significant extrinsic toughening, specifically from crack bridging, crack deflection and (to a lesser extent) microcracking is found; such crack-tip shielding mechanisms naturally induce resistance-curve behavior in an analogous way to the toughening of structural. In this presentation, we discuss the origin of such toughening, e.g., crack bridging from collagen fibrils and uncracked ligaments, and describe how such mechanisms are controlled by the hierarchical microstructure of these biological materials. Moreover, we address the issue of subcritical (time- and cycle-dependent) cracking and why these materials degrade with age.

9:45 AM
Osteoblast-Like Cell Mineralization Induced by Multiphasic Calcium Phosphate Ceramic: *Reed Ayers*[1]; Sheila Nielsen-Preiss[2]; Virginia Ferguson[1]; John J. Moore[1]; Hans-Joachim Kleebe[1]; [1]Colorado School of Mines, CCACS, 1500 Illinois St., Golden, CO 80401 USA; [2]University of Colorado Health Sciences Center, S/M Endocrinology/Metabolism/Diabetes Di, 4200 E. Ninth Ave., CB A009/111H, Denver, CO 80262 USA

The work presented here examines the effect of multiphasic CaP materials on the activity of Saos-2 osteoblast like cells. Heterogeneous calcium phosphate (HCaP) was synthesized using self-propagating high temperature combustion synthesis (SHS). All samples were characterized using XRD, SEM, and FTIR. Cell culture procedures of CaP with Saos-2 cells followed ASTM 813-01. Samples were examined by Environmental SEM (ESEM) and EDS to characterize the mineral content of the cultures. Gold coated samples were used for high resolution imaging. Biomineralization was noted in unconditioned cultures exposed HCaP while the controls (cells only in media and HCaP only in media) showed no mineralization. Calcium phosphate plate-like structures were seen adjacent to cells expressing calcium phosphate containing vesicles. These structures are similar to the organophosphate crystals seen in previous work. Plate-like crystals were also noted in the larger membrane vesicles on the Saos-2 cells indicating the biomineralization was being cellularly mediated.

10:05 AM Break

10:20 AM
Phase Dependent Fracture of Polytetrafluoroethylene (PTFE): *Eric Nathaniel Brown*[1]; G. Rusty Gray[2]; Dana M. Dattelbaum[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., TA-35, Bldg. 455, DCDP 01S, MS E544, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Dynamic Experimentation, MS P952, Los Alamos, NM 87545 USA

Compared with other polymers, polytetrafluoroethylene (PTFE) presents an extremely low coefficient of friction and excellent chemical and thermal stability. This has lead to a wide range of applications for biological implants ranging from single component PTFE structures to sliding contact pads in complex joints, as well as numerous other failure sensitive applications. However, the failure mechanisms of PTFE have received relatively little investigation. Polytetrafluoroethylene is semi-crystalline in nature with its linear chains forming complicated phases near room temperature and ambient pressure. Due to the presence of three unique phases near room temperature, failure during standard operating conditions is strongly phase dependent. This paper presents a comprehensive and systematic study of fracture and damage evolution in pedigreed PTFE 7C to elicit the effects of temperature-induced phase on fracture mechanisms. Fracture behavior is observed to undergo transitions from brittle-fracture below 19°C to ductile-fracture with fibril formation and large-scale plasticity over 30°C.

10:40 AM Cancelled
Electromagnetic-Thermal Responses of Tissues During Microwave Hyperthermia

11:00 AM
Mechanistic Aspects of the Fatigue Behavior of Mineralized Tissues: *Jamie J. Kruzic*[1]; Ravi K. Nalla[2]; John H. Kinney[3]; Robert O. Ritchie[2]; [1]Oregon State University, Dept. of Mechl. Engrg., 204 Rogers Hall, Corvallis, OR 97331 USA; [2]Lawrence Berkeley National Laboratory, Matls. Scis. Div., 1 Cyclotron Rd., Bldg. 62R0100-8255, Berkeley, CA 94720-8139 USA; [3]Lawrence Livermore National Laboratory, 700 East Ave., Livermore, CA 94550 USA

The failure of cortical bone and dentin due to repetitive cyclic loading is a problem of considerable clinical significance. Accordingly, in vitro fatigue-crack growth experiments were performed on human cortical bone (34-41 year-old humeri) and elephant dentin specimens in Hanks' Balanced Salt Solution with the goal of understanding the mechanisms of fatigue-crack propagation. Fatigue-crack growth rates, da/dN, were measured and characterized in terms of the stress intensity range, ΔK. Additionally, subcritical crack-growth experiments were conducted under sustained (static) loading to determine if fatigue-crack growth is due to repetitive loading and unloading, or simply a function of the maximum stress intensity. In both cases, a regime was found where a true cyclic-fatigue mechanism exists; however, for human cortical bone at growth rates above ~ 5 x 10^{-7} m/cycle, both cyclic loading and sustained loading at the maximum stress intensity yielded similar behavior. For the regime of behavior where cyclic loading is required to achieve crack propagation, a fatigue mechanism of alternating crack blunting and resharpening is proposed for both tissues.

11:20 AM
Mechanical Behavior and Structure of a Toco Toucan Beak: *Yasuaki Seki*[1]; Matthew S. Schneider[1]; Marc A. Meyers[1]; Bimal Kad[2]; Franck Grignon[1]; [1]University of California, Dept. of MAE 0411, 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]University of California, Dept. of Structl. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0085 USA

Composite sandwich structures of biomaterials are found throughout nature. Toucan beaks are an excellent example of this. The beaks are 1/3 the length of the bird, yet only make up about 1/20 of the birds mass. In this study, the structure and mechanical properties of a Toco Toucan beak was studied. It was found to be a sandwich composite with an exterior of keratin and a fibrous network of open cells made of calcium rich proteins. The keratin layer was comprised of many small hexagonal bricks glued together. The mechanical properties of the keratin layer revealed a tensile strength of about 50 MPa and a Young's modulus of 2 GPa. Additional measurements taken using micro- and nanoindentation hardness corroborated these values. The keratin layer also had a viscoplastic behavior with changing modes of deformation from slippage due to release of the organic glue at low strain rates to fracture of the keratin bricks at higher strain rates. The fracture surfaces were images with SEM and models of failure behavior are presented.

11:40 AM
Viscoelastic Behavior of Human Stratum Corneum: *Kenneth S. Wu*[1]; Eilidh Bedford[2]; David J. Moore[3]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305-2205 USA; [2]Unilever Research and Development, Port Sunlight CH63 3JW UK; [3]Unilever Research and Development, Edgewater, NJ 07020 USA

The outermost layer of skin, or stratum corneum (SC), provides mechanical and permeability barrier protection from the external environment. The mechanical behavior of SC is crucial to its function and related to underlying cellular microstructure. The influences of temperature, hydration, and tissue treatments have received some attention, but investigations frequently ignore underlying viscoelastic and molecular relaxation processes. These processes are systematically explored here using dynamic and transient mechanical tests involving creep-recovery and stress-relaxation experiments. The influences of hydration, temperature, and chemical modification of the SC tissue were explored. Chemical treatment included delipidization and exposure to buffered pH solutions that modify intercellular lipid properties. Time scales associated with SC viscoelasticity are reported and related to underlying molecular relaxation processes. Stretched exponential modeling of the behavior provides a measure of the distribution of relaxation time scales and their dependence on conditioning parameters. Relaxation processes in the SC are shown to be dominated by long time scales with softening effects from increasing SC hydration significantly decreasing relaxation times.

12:00 PM
Mechanisms Governing the Inelastic Deformation of Cortical Bone: *Christopher Mercer*[1]; Rizhi Wang[2]; Anthony G. Evans[1]; [1]University of California, Matls. Dept., Engrg. II, Rm. 1355, Santa Barbara, CA 93106 USA; [2]University of British Columbia, Dept. of Metals & Matls. Engrg., Forward Bldg., Rm. 107, 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

A two-part investigation has been conducted to understand the inelastic response of cortical bone. In the first part, a flexural test protocol has been designed and implemented that monitors the axial and transverse strains on both the tensile and compressive surfaces of cortical bovine bone. The results are used to assess the relative contributions of dilatation and shear to the inelastic deformation. A deconvolution procedure has been employed to affirm that the stress/strain curves in tension and compression are consistent with results in the literature. Unload/reload tests have characterized the hysteresis and provided insight about the mechanisms causing the strain. In the second part of the investigation, a model is devised for the intrinsic stress/strain response of bone, based on a recent assessment of the nano-scale organization of the collagen fibrils and mineral platelets. The model is used to rationalize the inelastic deformation in tension, as well as the permanent strain and hysteresis.

Bulk Metallic Glasses: Processing and Fabrication I

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Monday AM — Room: 3006
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Peter K. Liaw, University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; Raymond A. Buchanan, University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA

8:30 AM Opening Remarks

8:35 AM
Prediction of the Constitution of Metallic Glasses: *Daniel Miracle*[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

Previous structural models show broad consistency with selected metallic glass features, but do not provided a convincing predictive capability for important characteristics, including constitution. An earlier model for the stability of metallic glasses by Egami and Waseda enabled a reasonable ability to predict the concentration of binary metallic glasses, but there has been no progress since in predicting or explaining the rich and confusing breadth of metallic glass constitutions for more complex glasses. An atomic structural model for metallic glasses has very recently been established. Using relative atomic sizes as the primary variable, this model provides a convincing ability to predict the constitution of metallic glasses with as many as seven chemically distinct constituents in glasses based on Zr, Pd, Fe, Mg, Ti, Al and rare earth metals. The features of this model will be briefly outlined, and comparison with between predicted and observed glass compositions will be presented and discussed. Predictions for medium-range atomic order and local coordination numbers will also be compared with experiment.

8:55 AM
Ti-Base Bulk Nanostructured Composite: *Jürgen Eckert*[1]; *Jayanta Das*[1]; Guo He[2]; Wolfgang Löser[3]; [1]Technische Universität Darmstadt, FB11 Matl. und Geowissenschaften, FG Physikalische Metallkunde, Petersenstrasse 23, Darmstadt, D-64287 Germany; [2]National Institute for Materials Science, Light Matl. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [3]Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden, Postfach 270016, D-01171 Dresden Germany

The in situ formation of bulk metallic glass based composites has recently been demonstrated in several multi-component alloy systems, where a strong glassy matrix is combined with a ductile dendritic ß-Ti type solid solution as the toughening phase. Taking advantage of the hints provided by this successful approach, we developed a strategy for the design of primary-dendrite/nano-eutectic in situ Ti-base bulk composites that can lead to simultaneous high strength and ductility. Our approach employs copper mold casting for the production of bulk alloys, and the solidified microstructure is designed to be composed of micrometer-sized ductile dendrites uniformly distributed inside a ma-

trix of nanoscale eutectic reaction products. The nanostructured matrix is achieved at a relatively deep eutectic, which facilitates the formation of a nano/ultrafine eutectic microstructure over a range of cooling rates. The multicomponent recipe stabilizes a ductile solid solution as the toughening phase and helps reduce the eutectic spacing down to nanometer scale. The observed microstructures will be described under consideration of the solidification parameters and the conditions required to form a nanostructured composite, and we will also correlate the microstructure with the mechanical properties to demonstrate the desirable high strength and plasticity.

9:15 AM
Development of Fe-Based Bulk Amorphous Alloys: From Theory to Experiment: *Z. P. Lu*[1]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA

Glass formation is always a competing process between liquid phase and resulting crystalline phases. Either enhancing the liquid phase stability or suppressing the formation of competing crystalline phase would greatly enhance the glass-forming ability (GFA) of glass-forming liquids. Based on these concepts elaborated in our previous publications,[1] we have successfully synthesized several novel Fe-based bulk metallic glasses (BMGs) which can be cast to fully glassy rods with diameters up to 12 mm.[2] In this talk, we will present the experimental results of these newly developed BMGs, together with the alloy design schemes, origins of the high GFA and implications of this study. Our work also demonstrated that minor additions of adequate elements are very powerful in designing new BMGs with superior GFA. This research was sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR-22725 with UT-Battelle, LLC. [1]Z. P. Lu & C. T. Liu, Phys. Rev. Lett., 91(2003)115505. [2]Z. P. Lu & C. T. Liu, Phys. Rev. Lett., 92(2004)245503.

9:35 AM
In Situ Synchrotron Study of Multiscale Phase Transformations in Bulk Metallic Glass: *Jonathan Almer*[1]; Xun-Li Wang[2]; Alexandru D. Stoica[2]; Ling Yang[4]; C. T. Liu[4]; [1]Argonne National Laboratory, Advd. Photon Source, 9700 S. Cass Ave., Bldg. 431, Argonne, IL 60439 USA; [2]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN USA; [4]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridg, TN USA; [4]University of Cincinnati, Cheml. & Matls. Engrg., Cincinnati, OH USA

We report on in situ measurements of wide-angle x-ray scattering (WAXS) and small-angle x-ray scattering (SAXS) during annealing of Zr-based bulk metallic glasses. The combination of WAXS and SAXS permits material investigation over length scales ranging from Angstroms to microns, respectively. Measurements were carried out at Sector1 of the Advanced Photon Source, using E=80keV x-rays. Such high-energy x-rays offer penetration comparable to neutrons, ensuring that the probe volume is bulk-representative, and provide access to a large volume of reciprocal space. By using an undulator and specialized optics, coupled with area detectors, we demonstrate temporal resolutions on the order of 15 seconds, sufficient for kinetic observations. We find that phase transformations proceed in stages, with distinctively different kinetics. Nanometer sized clusters form first, as revealed by SAXS, followed by an abrupt amorphous-to-crystalline phase transformation, as revealed by WAXS. These results are compared with complementary DSC measurements, and related to transformation theory.

9:55 AM
Role of Ti Replacement for Zr in the Formation of ZrTiCuNiAl Bulk Metallic Glasses: *D. Ma*[1]; *H. Cao*[1]; L. Ding[1]; K. C. Hsieh[2]; Y. Austin Chang[1]; [1]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Sun Yat Sen University, Inst. of Matls. Sci. & Engrg., Kaohsiung, Taiwan 80424 Taiwan

We present a series of ZrTiCuNiAl alloys, which can readily form glassy rods with at least 2 mm in diameter using a conventional copper-mould casting method. Replacement of Zr with Ti, while maintaining the compositions of the other elements constant, appears to decrease the glass-forming abilities (GFA), which eventually vanished at a critical Ti content of 7 at%. To gain an understanding of the GFA of these alloys, we calculated a temperature versus Ti composition section, or an isopleth, using our thermodynamic description recently developed. These thermodynamic calculations suggest that bulk glass formation of these alloys are associated with the suppression of an invariant eutectic reaction, i.e., L —> Cu2TiZr + t3_AlCuZr + NiZr + Cu10Zr7 + AlCu2Zr. The decrease in the GFA with the replacement of Zr by Ti could be attributed to increasing liquidus temperatures as well as competition between the formation of glassy and crystalline phases.

10:15 AM Break

10:35 AM
On the Origin of High Glass Forming Ability in Bulk Metallic Glasses: *Ralf Busch*[1]; [1]Oregon State University, Dept. of Mechl. Engrg., 204 Rogers Hall, Corvallis, OR 97331 USA

Bulk metallic glass forming liquids are alloys with typically three to five elemental components that have a large size mismatch.[1] They are dense liquids with small free volumes and viscosities that are several orders of magnitude higher than in pure metals or known alloys. In addition these melts are energetically closer to the crystal than other metallic melts due to their high packing density in conjunction with a tendency to develop short range order. These factors lead to slow crystallization kinetics. The crystallization kinetics is very complex especially in the vicinity of the glass transition due to the influence of phase separation and the decoupling of the diffusion constants of the different species. Recent developments, such as shear rate depend viscosity are discussed. [1]R. Busch, JOM-J Min. Met. Mat. S. 52, 39 (2000).

10:55 AM
Thermodynamics and Kinetics of Complex Liquids: Bulk Metallic Glass: *Hans J. Fecht*[1]; Rainer Wunderlich[1]; Guojiang Fan[2]; [1]University of Ulm, Dept. of Matls., Faculty of Engrg., Albert-Einstein-Allee 47, Ulm D-89081 Germany; [2]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

With complex alloys forming BMG crystallization can be avoided over a broad temperature/time window. As such, the relevant thermodynamic properties of the metastable glassy and undercooled liquid state can be directly measured below and above the glass transition temperature before crystallization on a nanometer scale sets in. The results for various thermodynamic functions, such as S(T), H(T), G(T) and V(T) together with the measurement of the viscosity (T) give new insight into the nature of the highly undercooled liquid state and the glass transition itself. The results on Zr-Ti-Ni-Cu-Be and Pd-Ni-Cu-P melts suggest a scenario where the glass transition in a metallic alloy is not a phase transition in the classical sense but kinetic freezing triggered by an underlying entropic instability. Further results are discussed within the framework of a new fragility concept. The financial support by the European Space Agency ESA (ThermoLab program MAP) is gratefully acknowledged.

11:15 AM
Atomic Size Effects on Glass Formability: *Mo Li*[1]; Payman Jalali[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA

Effects of atomic size difference on glass formability is investigated in this work using both numerical simulations and analytical approaches. In a model system where atomic size effects are separated from other factors such as enthalpy of formation, we found that atomic size difference plays a role in kinetics for various competing phases, including glass. It is the competition of these different kinetic processes that eventuall leads to glass formation in the system which has longer times for crystallization.

11:35 AM
Thermodynamics and Kinetics of Pd43Ni10Cu27P20 Bulk Metallic Glass-Forming Liquid: *Guojiang Fan*[1]; H. Choo[1]; P. K. Liaw[1]; H. J. Fecht[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Dougherty Engrg. Bldg., Knoxville, TN 37996 USA; [2]University of Ulm, Matls. Div., Ulm 89081 Germany

Recently, several families of multicomponent alloys with a complex chemistry show a much sluggish kinetics for the crystal nucleation during undercooling, which enables the formation of bulk metallic glasses (BMGs) at relatively slow cooling rates. In order to gain an insight on the BMGs formation and also develop BMGs with new compositions, it is essential to understand the thermodynamics and kinetics of bulk metallic glass-forming liquids, which dominate the glass formation. In this study, the thermodynamics and kinetics of the Pd43Ni10Cu27P20 bulk metallic glass-forming liquid have been investigated over a wide temperature range. The thermodynamic functions for this liquid have been calculated, based on the measured specific heat capacity for the liquid and crystal as well as the enthalpy of fusion. The fragility of the Pd43Ni10Cu27P20 bulk metallic glass-forming liquid has been determined, which indicates that this liquid is relatively fragile, despite the best glass-forming ability found thus far. The excellent glass-forming ability of this alloy is attributed to a smaller thermodynamic driving force and larger diffusion length for crystallization than in other bulk-metallic-glass-forming alloys.

Carbon Technology: Anode Raw Materials

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland

Monday AM Room: 2007
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Bruce Goddard, ConocoPhillips, Ponca City, OK 74602-1267 USA; Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA

8:30 AM
Characterization of Reactivity of Green and Calcined Petcoke with Oxygen for Application to Combustion Systems: *Constance L. Senior*[1]; David O. Lignell[1]; Zumao Chen[1]; Adel F. Sarofim[1]; Todd W. Dixon[2]; [1]Reaction Engineering International, 77 W. 200 S., Ste. 210, Salt Lake City, UT 84101 USA; [2]ConocoPhillips, Borger Refinery, Spur 119 N., Borger, TX 79007 USA

Petcoke combustion differs from coal combustion because of the inherent properties of the fuel. The lower volatile content of petcoke and the lower surface area of the char relative to coal make petcoke more difficult to ignite than coal and require longer burnout times relative to coal. Green cokes generally have volatile matter in the range of 8-10%, whereas calcined cokes have on the order of 1% volatile matter. Measurements of green petcoke reactivity have not been previously reported in the literature, particularly measurements made at the high temperatures characteristic of a flame. This paper presents information on petcoke combustion kinetics that was obtained from combustion of coke samples under well-controlled conditions. Green petcoke reactivity was higher than that of calcined coke, but lower than coal. Measured reactivities were used to model the combustion of petcoke and to explore the effects of particle size and temperature on burnout time.

8:55 AM
Comparison of Physical Properties of Finer Grains of Calcined Cokes Generated During Calcination with Those Originally Present in Green Cokes: *Ravindra Narayan Narvekar*[1]; [1]Goa Carbon Limited, Production, Dempo House, Campal, Panaji, Goa 403001 India

Green coke is an aggregate of various grain sizes ranging from few microns to few inches.The finer particles present in green cokes are more susceptible to burn inside the kiln or get drifted outside the kiln alongwith combustion gases.Simultaneously finer particles are generated inside the kiln during the calcination process because of intense heat and abrasion during transportation from feed end to discharge end of the kiln.Now Goa Carbon Limited has tried to find out whether rrrrrthe generated finer grains of calcined coke differ from originally present finer grains in physical characteristics.There are indications that the generated finer grains are significantly denser than originally present finer grains as seen from bulk density,apparent density etc.

9:20 AM
A Tool for Predicting Anode Performance of Non-Traditional Calcined Cokes: *Keith J. Neyrey*[1]; Les Edwards[1]; J. Anthony Ross[2]; Franz Vogt[1]; [1]CII Carbon, LLC, Techl., 1615 E. Judge Perez, 4th Fl., Chalmette, LA 70043 USA; [2]Century Aluminum of WV, Inc., PO Box 98, Ravenswood, WV 26164 USA

The supply of traditional calcinable green coke is not meeting demand, which has resulted in calciners using green cokes that are typically sold to the fuel market. In order for calciners to more thoroughly evaluate non-traditional cokes (higher sulfur and metals and higher volatile content), a simple microscopic method was developed to make an empirical determination of coke quality. Microscopic analysis of calcined coke is not a new technique, and many papers have been published on the subject. Sophisticated software and equipment have been developed to perform such analyses. The simpler method used in this study is economically feasible for a production laboratory. The paper describes the microscopic analysis of a number of traditional and non-traditional cokes. Bench scale anodes were prepared and evaluated for critical properties. These properties were correlated to the results of the microscopic evaluations and other common coke quality measures. Comments about the performance of these cokes in anodes are given.

9:45 AM
Multivariate SPC Applications in the Calcining Business: *Michael Marcon*[1]; Todd W. Dixon[2]; Arvin Paul[3]; [1]InControl Technologies, Inc., 3845 FM, 1960 W. Ste. 318, Houston, TX 77068 USA; [2]ConocoPhillips, Borger Refinery, Spur 119 N., Borger, TX 79007 USA; [3]ConocoPhillips, Lake Charles Calcining Plant, PO Box 3187, Lake Charles, LA 70602-3187 USA

The introduction of Advanced Process Control (APC) has changed the way process engineers view the operating principals of industrial control schemes. While the major benefit of APC is to provide tighter control by reducing control variable variation, one of the side benefits of APC is they collect data on all phases of the operation. A tool to extract additional information from the available data and further reduce the operational region is multivariate statistical process control (MVSPC). This is a statistical procedure based on the correlations that exists between the process variables. By taking advantage of these correlations, it is possible to maintain production within this multivariate region and further reduce overall variation and lower production cost. Advanced statistical analysis of a commercial, rotary kiln can provide a useful, quantitative measure of process stability and, more importantly, allow on-line fault detection of process control elements.

10:10 AM Break

10:25 AM
Physical Characterization and Reactivity Measurerments of Anode Butt Cores: *John Marthin Andresen*[1]; [1]Pennsylvania State University, The Energy Inst., 407 Academic Activities Bldg., State College, PA 16802 USA

The recycling of anode butt material is frequently used improve the economics of the Hall-Héroult process, where alumina is reduced by carbon to produce aluminum. Since about 15-30wt% of a new anode can consist of recycled butt material this fraction could have a significant impact on the performance of the new anode in the bath. However, very little is known about the variation in anode butt materials chemistry where major differences can have been developed during electrolysis. On one hand, the lower part has been in contact with the hot electrolyte experiencing temperatures in the range of 930 to 980°C and may also have been modified by CO2 burn where typically the binder coke is attacked selectively. On the other hand, the top part of the anode reaches a temperature in the range of 350 to 600°C depending on the protective cover of electrolyte material and air burn may take place. Further, the middle part of the anode butt may not be affected by any of the above. This work focuses on the variation of physical properties of anode butt cores using non-destructive and destructive methods. X-ray computed tomography scanning was used as a non-destructive method to study the overall structure of the anode butt cores from the air burn side to the work face. Parallel to the non-destructive study, conventional destructive data, including apparent and absolute densities, and specific pore volume, were obtained for the anode butt cores representing different position in the reducing pot. The data indicate that the carbon near the air-burn side as well as that of the working face have somewhat higher porosity than the center of the core, that may lead to higher reactivity.

10:50 AM
Studying Mesophase Contents in Pitches from Different Sources: *A. S. Tayanchin*[1]; Y. D. Kratzova[2]; V. S. Biront[2]; J. A. Johnson[1]; [1]RUSAL Engineering and Technological Center, Krasnoyarsk Russia; [2]Krasnoyarsk University of Non-Ferrous Metals and Gold, Krasnoyarsk Russia

The ultimate goal for the present work was to study the pitch anisotropic content and liquid/solid phase formation in different Russian pitch supplies and to identify and compare the contents of fine anisotropic inclusions and mesophase areas. The study showed that mesophase contents and sizes of mesophase areas have reasonable correlation with the pitch softening point whereas the probability of mesophase generation increases with the increase in softening point. Mesophase content is minimal in the as produced pitches. However, an intensive ordering of structures in the pitch, which is associated with the growth of mesophase spheroids, starts at the pitch temperatures in the range of 420-450°C. Several consecutive “coke over coke” phases have been identified for creation of mesophase starting from nucleation, growth of mesophase spheroid that further coalescences into the mesophase package (particle). Correlations have been established between mesophase and size of the mesophase areas.

11:15 AM
Procedure for Testing Infiltration Property of Pitches: *V. K. Frizorger*[1]; Y. D. Kratzova[2]; A. S. Tayanchin[1]; [1]RUSAL Engineering

and Technological Center, Krasnoyarsk Russia; [2]Krasnoyarsk University of Non-Ferrous Metals and Gold, Krasnoyarsk Russia

A simple test procedure was developed for testing pitch infiltration (pitch wetting) in to the coke, which is based on using pencils of pitch specifically made for the tests. The test procedure avoids some of the issues with existing pitch wetting procedures related to the time consumed in the test, degree of variation of test results and sensitivity to even minor changes in the experimental lay-out. The test procedure looks for the coefficient of pitch infiltration which is not dependent on such factors as the mass of pitch or coke, the shape or size of the pitch or coke and it eliminates the influence of the test container material with the coke and pitch placed for experiments. The procedure is highly sensitive to changes in the pitch properties and the resultant rate of pitch infiltration into the coke is independent of the coke and pitch properties.

Cast Shop Technology: Cast Shop Safety

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday AM Room: 2001
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Seymour G. Epstein, The Aluminum Association Inc, Washington, DC 20006 USA

8:30 AM
A Summary of Findings from Twenty Years of Molten Metal Incident Reporting: *Seymour G. Epstein*[1]; [1]The Aluminum Association, Inc., 900 19th St. NW, Ste. 300, Washington, DC 20006 USA

For more than 50 years the aluminum industry has had an ongoing effort to gain an understanding of molten aluminum-water explosions and how they may be prevented. In spite of these extensive efforts, explosions continue to occur. Following the occurrence of several devastating explosions in the early 1980s, The Aluminum Association established a formal world-wide program of reporting molten metal incidents to enhance awareness, to complement the industry's research efforts and to provide guidance for the Association's safety efforts. Much has been learned from this program, and the findings from 20 years of reporting will be summarized in this paper.

8:55 AM
Scrap Melting Safety - Improving, But Not Enough: M. D. Bertram[2]; *F. R. Hubbard*[2]; D. C. Pierce[1]; [1]Consultant, 8408 Twin Lake Ct., Richmond, VA 23229 USA; [2]IMCO Recycling, 397 Black Hollow Rd., Rockwood, TN 37854 USA

Over the years scrap suppliers and processors have learned many lessons about improving safety in melting operations. Most plant personnel are aware of the typical hazards involved in melting scrap aluminum. However, there are issues that challenge the best efforts of management to keep their plants and people safe. Tightening scrap supplies, small items that create large problems (e.g., air bag cylinders) and very dense scrap containing contaminants are three issues that can make safety a challenge in the melting facility. Equipment upgrades plus continued education/training and communication help, but the fact that serious molten metal incidents continue to occur, prove that there is a need for additional vigilance in this arena.

9:20 AM
Safe Charging of RSI (Remelt Secondary Ingot) and Other Ingot Shapes into Melting Furnaces: *John L. Zeh*[1]; [1]Logan Aluminum Inc., U.S. Hwy. 431 N., Russellville, KY 42276 USA

Safety is the first requirement when charging a melting furnace. Moisture and other contaminants have repeatedly shown that their presence on or in RSI or other charge materials can cause catastrophic explosions. Logan Aluminum Inc. is one of the largest users of RSI thus has extensive experience in safe use of this material. Other charge materials such as magnesium and magnesium alloy ingot present similar dangers. Steps to ensure safe use of sows or ingots include communication to suppliers of safety requirements, incoming material specifications, inspection and test, quarantine and rejection procedures, safe pre-melting processing (drying, storage and inventory tracking) and melt furnace charging. This system significantly reduces the risk of explosion during melting.

9:45 AM
Cause and Prevention of Explosions Involving DC & EMC Casting of Aluminum Sheet Ingot: *Ray T. Richter*[1]; J. Martin Ekenes[2]; [1]Alcoa Inc., Alcoa Techl. Ctr., Alcoa Ctr., PA 15069 USA; [2]Hydro Aluminum

The casting of aluminum alloy sheet ingot and T-bar presents the potential for some of the most volatile situations that can occur in DC (direct chill) and EMC (Electromagnetic) casting processes. Aluminum Association explosion incident data from over 300 explosions spanning a period of more than twenty years was reviewed and analyzed looking for common factors and repetitive reasons for explosions. Analysis of explosions occurring during the three stages of sheet ingot casting, 'start of cast', 'steady state' and 'end of cast', were examined and prioritized. Case studies illustrate the need for understanding both technical and non-technical factors contributing to explosions involving molten metal. This paper identifies the major causes of explosions involving DC casting of aluminum alloy sheet ingot and makes recommendations for how to prevent the recurrence of such events and minimize the risk of injury.

10:10 AM Break

10:20 AM
A Safe Cast Abort System for D.C. Sheet Ingot with Non-Tilting Furnaces: *John A. Paris*[1]; Michael J. Matisko[1]; [1]Alcoa Inc., 4879 State St., Bettendorf, IA 52722 USA

During DC casting operations, emergency situations arise that require the ability to safely terminate the casting operation when the trough and filtering systems are full of metal from furnace to casting table. Safe termination includes stopping the platen and draining away the metal without putting personnel at risk to injury. Older casting facilities often do not have the systems and equipment capable of performing a safe termination and space is generally very limited. A forty-year old DC casting pit at Alcoa Davenport Works was modified to provide this capability. This paper discusses the design criteria, space and equipment constraints, and procedural changes that resulted in a successful retrofit to allow safe casting aborts on this older DC casting station.

10:45 AM
Preventing Molten Metal Explosions Related to Skim Tools and Salt: *Edward M. Williams*[1]; Ray T. Richter[2]; Donald L. Stewart[2]; Jake J. Niedling[3]; [1]Alcoa Inc, Hwy. 66, PO Box 10, Newburgh, IN 47629 USA; [2]Alcoa Inc, Alcoa Techl. Ctr., Alcoa Ctr., PA 15069 USA; [3]Alcoa Inc, 900 S. Gay St., Riverview Tower, Knoxville, TN 37902 USA

In 2003, Alcoa Inc. experienced a number of molten aluminum explosions that were associated with skim tools used for cleaning in-line metal treatment units. These explosions were investigated and the incidents appeared to be related to the residual skim and dross on the tools from previous skimming operations. Visual observations indicated high moisture content on the skim left on the tools once they cooled to room temperature. Analysis of the dross confirmed that it contained varying amounts of magnesium chloride salts, which are highly hygroscopic at room temperature. Testing was performed analyzing the skim and investigating moisture pick-up and release during cooling and heating cycles. Based upon this analysis procedural and equipment modifications were made to minimize the risk of further explosions.

11:10 AM
Casthouse Safety - A Focus on Dust: *David D. Leon*[1]; [1]Alcoa Inc., Alcoa Techl. Ctr., Alcoa Ctr., PA 15069 USA

Information, training and safeguards regarding aluminum dusts and their explosibility has traditionally focused on pure metallic powders, pigments, pastes, and their associated handling facilities. But incidents throughout the aluminum industry has shown that there are hidden dangers in other processes, like casting and metal fabricating, which can generate potentially explosive dusts. This paper will introduce the audience to the hazards associated with potentially explosive dusts, where they can be found in the casthouse, prevention methods, and actions to be taken in case of an incident.

11:35 AM Panel Discussion

Characterization of Minerals, Metals and Materials: Extraction and Processing Applications

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Monday AM Room: 2010
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; John E. Dutrizac, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada

8:30 AM Invited
Formation of Mg-Al Layered Double Hydroxide Modified by Organic Acid Anions and its Application to Wastewater Treatment: *Tomohito Kameda*[1]; Masami Saito[2]; Shingo Saito[2]; Yoshiaki Umetsu[1]; [1]Tohoku University, IMRAM, 1,1 Katahira 2-Chome, Aoba-ku, Sendai 980-8577 Japan; [2]Tohoku University, Dept. of Applied Chmst., Grad. Sch. of Engrg., Aoba 07, Aoba-ku, Sendai 980-8579 Japan

Development of scavengers to remove heavy metal ions and organic pollutants from wastewaters has been attempted by coupling Mg-Al layered double hydroxide (Mg-Al LDH) with organic acid anions having functional groups in their structure, such as EDTA and organic sulfonic acid. These organic-modified Mg-Al LDHs were synthesized by adding a solution of magnesium and aluminum nitrates to alkaline solution containing the organic acid anion. The solid product was confirmed to be hydrotalcite-like compounds with Al/Mg mole ratio of 1/3 and to have intercalated organic anions in the interlayers. The EDTA-modified Mg-Al LDH was found to remove rapidly Cu2+ and Cd2+ from aqueous solution. The organic sulfonate-modified Mg-Al LDH could uptake organic pollutant exampled by bisphenol A. This organic sulfonate-modified Mg-Al LDH was regenerated by extraction of the uptaken organic pollutant with ethanol, and confirmed to have a high potentiality for the repeated use.

8:55 AM Invited
Characterization of Iron(III) Oxyhydroxides in Hydrometallurgical Residues: *Mitch Loan*[1]; William Richmond[2]; Tim St. Pierre[3]; Gordon M. Parkinson[2]; [1]University of Limerick, Matls. & Surface Sci. Inst., Limerick Ireland; [2]Curtin University of Technology, AJ Parker CRC for Hydrometall., Nanochmst. Rsch. Inst., GPO Box U 1987, Perth, W.A. 6845 Australia; [3]University of Western Australia, Sch. of Physics, Mailbag Delivery Point M013, Crawley, Perth, W.A. 6009 Australia

The characterization of iron(III) oxyhydroxides in hydrometallurgical residues can be a complicated task. Iron-phases in residues commonly arise after neutralization and precipitation of liquors to remove dissolved iron or after addition of soluble iron to remove dissolved impurities. Iron-phases also precipitate in storage ponds, mine drainage and waste streams where air or bacteria oxidize dissolved Fe(II). The low solubility of iron creates a high supersaturation environment favouring the formation of nanoscale and metastable phases. These phases often have poor physical properties and contain high loadings of adsorbed components - often the element being refined. Importantly, low proportions of iron(III) oxyhydroxides (~ 20 wt%) can still control the physical properties of a residue, and also release adsorbed toxins on transformation to more crystalline phases. In this study, the application of multiple techniques to differentiate among the various iron(III) oxyhydroxide phases, and the complications involved in characterizing the poorly crystalline ferrihydrite, schwertmannite, akaganeite and goethite in a heterogeneous sample environment are demonstrated.

9:20 AM Invited
Influence of Platinum Group Metals Mineralogy on their Leachability During a Chloride-Assisted Pressure Oxidation Process: *C. Joe Ferron*[1]; C. C. Hamilton[1]; O. Valeyev[1]; N. Davidson[1]; [1]SGS Lakefield Research Limited, Metallurgl. Tech., PO Box 4300, 185 Concession St., Lakefield, Ontario K0L 2H0 Canada

The PLATSOL process was developed to extract simultaneously, base metals, gold and platinum group metals from various materials. Extensive research has indicated that most PGM's were amenable to the PLATSOL process, with the notable exception of cooperite PtS. Further work showed that a thermal pretreatment at 500-700ºC transformed the structure of the cooperite and similar refractory minerals (Pt, Pd) S into Pt metal and Pt-Pd alloys that responded very well to the PLATSOL process. Examples are presented of the mineralogy of PGM concentrates as produced, in the residue from the PLATSOL leach and after pre-treatment.

9:45 AM
Storing Capacity of Copper Ions in Vermiculite and Its Relations to the Preparation Conditions: *Bowen Li*[1]; *Janny-Yang Hwang*[1]; Shuhui Yu[2]; [1]Michigan Technological University, Inst. of Matls. Procg., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]China University of Geosciences, Matl. Dept., 29 Xueyuan Rd., Beijing, Beijing 100083 China

The storing capacity of copper ion in vermiculite was investigated and its relations to the preparation conditions were determined based on the ion exchange reaction between vermiculite and copper chloride solution. The factors that effecting the carrying capacity of vermiculite with copper ions, such as particle size of vermiculite, concentration of the copper ion in the solution, pH value, reaction temperature and reaction time, etc. were investigated individually. The best reaction condition for preparing Cu-vermiculite is 0.1M copper ion concentration, pH 3.0, 80-90°C reaction temperature and 3 hours reaction time. The Cu-vermiculite carrying 5.5wt% copper was prepared with vermiculite powder of 4.1 microns in average diameter.

10:10 AM Break

10:20 AM Invited
Developing an Atomic-Level Understanding of the Mechanisms that Govern CO2 Mineral Carbonation Reaction Processes: *Michael J. McKelvy*[1]; Andrew V.G. Chizmeshya[1]; Jason Diefenbacher[2]; Hamdallah Béarat[2]; R. W. Carpenter[1]; George Wolf[3]; [1]Arizona State University, Ctr. for Solid State Sci., Sci. & Engrg. of Matls. Grad. Prog., Tempe, AZ 85287 USA; [2]Arizona State University, Ctr. for Solid State Sci., Tempe, AZ 85287 USA; [3]Arizona State University, Dept. of Chmst. & Biochmst., Tempe, AZ 85287 USA

Mineral carbonation is an intriguing CO2 sequestration candidate technology, which produces environmentally benign and geologically stable materials. The primary challenge is economically viable process development. Serpentine and olivine minerals are exciting candidate feedstock materials, due to their wide availability, low-cost, and rapid mineral carbonation potential. Cost-effectively enhancing their carbonation rate is critical to reducing mineral sequestration process cost. We will discuss our recent research into the mechanisms that govern serpentine/olivine mineral carbonation reaction processes, including in situ observations of the mineral carbonation process and a novel mechanistic approach to enhance carbonation reactivity that avoids the cost of mineral activation. Our goal is to develop the necessary atomic-level understanding to engineer improved carbonation materials and processes to reduce process cost.

10:50 AM Invited
Alkaline Leaching for Mixture of Nickel Hydroxide and Nickel-Rare Earth Alloy: *Masao Miyake*[1]; Masafumi Maeda[1]; [1]University of Tokyo, Inst. of Industl. Sci., Internatl. Rsch. Ctr. for Sustainable Matls., 4-6-1 Komaba, Meguro, Tokyo 153-8505 Japan

A leaching treatment was investigated to recover metals including rare earths from the mixture of Ni hydroxide and hydrogen storage alloy. By leaching with an ammoniacal alkaline aqueous solution, only Ni hydroxide was dissolved from the mixture and the hydrogen storage alloy could be recovered without decomposing. The effects of leaching conditions such as pH and temperature on the dissolution rate of Ni hydroxide were examined.

11:15 AM Invited
Development and Application of Laser Ablation Microprobe (LAM)-ICP-MS for Analysis of Trace Precious Metals: *Louis J. Cabri*[1]; Paul J. Sylvester[2]; Mike N. Tubrett[2]; Anna Peregoedova[3]; Greg McMahon[4]; J.H. Gilles Laflamme[5]; [1]Cabri Consulting Inc., 99 Fifth Ave., Ste. 122, Ottawa, Ontario K1S 5P5 Canada; [2]Memorial University of Newfoundland, Dept. of Earth Scis., St. John's, Newfoundland A1B 3X5 Canada; [3]McGill University, Dept. of Earth & Planetary Scis., 3450 Univ. St., Montreal, Quebec H3A 2A7 Canada; [4]Fibics Incorporated, 556 Booth St., Ste. 200, Ottawa, Ontario K1A 0G1 Canada; [5]CANMET, MMSL, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

In-situ analyses of trace precious metals at low levels (10s to 100s of ppb) are important for quantifying mineralogical balances and for solving process problems when used together with detailed mineralogical study. This will be a focus of academic research and contractual work for industry in the new Inco Innovation Centre located at Memorial University of Newfoundland. Sulfide standards were developed and tested for LAM-ICP-MS analyses of potential precious metals carriers in sulfides (e.g. pentlandite, pyrite, pyrrhotite, and chalcopyrite). After use of sintered single-element platinum-group element (PGE) and Au standards (Ballhaus & Sylvester, 2000) and fused FeS single-PGE standards (Cabri et al., 2003), we have successfully characterized fused FeS standards containing six PGE+Au and applied them in several projects ranging from determination of PGE partitioning in experimental sulfide charges (Mungall et al., in press) to exploration samples and process products for mass balance calculations.

Computational Aspects of Mechanical Properties of Materials: Atomistic Methods

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Monday AM Room: 3012
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Christopher Woodward, Air Force Research Laboratory, MLLMD, Dayton, OH 45433 USA; Vaclav Vitek, University of Pennsylvania, Matls. Sci. & Engrg., Philadelphia, PA 19104 USA

8:30 AM Invited
Modeling Nanoscale Elastic Instabilities: *Krystyn J. Van Vliet*[1]; [1]MIT, DMSE, 77 Mass. Ave., Rm. 8-214, Cambridge, MA 02139 USA

Computational modeling of inelastic deformation is complicated by the range of length and timescales involved in nucleation, motion and interaction of defects such as dislocations in crystalline materials. Here, we present a computational approach that combines the atomistic detail of molecular dynamics with the large structural length scale of continuum models. We find that the application of this atomistically informed continuum approach is well-suited to the study of elastic instabilities in crystals, in particular under concentrated surface loads as typical of nanoindentation. With this computational tool, we consider the effects of microstructural and macrostructural dimensions on the elastic limit of crystals and crystalline structures of nanoscale physical dimensions.

9:05 AM
Ab-Initio Computational Calculations of Elastic Constants of Titanium Boride (TiB) Using Density Functional Theory: *K. S. Ravi Chandran*[1]; Krutibas Panda[1]; [1]University of Utah, Metallurgl. Engrg., # 412, 135 S., 1460 E., Salt Lake City, UT 84112 USA

Ab-initio computational calculations of anisotropic elastic constants of titanium boride, TiB have been performed using the computational implementation of Density Functional Theory (DFT) in Wien2k. TiB has orthorhombic crystal structure, thus nine independent elastic constants are to be determined to completely understand its elastic behavior. TiB has attracted attention as reinforcement in metal matrix composites, wear resistance coating and monolithic ceramic due to its high hardness and elastic modulus. The elastic constants were determined using the Full-Potential Linearized Augmented-Plane-Wave (FLAPW) method with the generalized gradient approximation (GGA) by employing specific distortions of the unit cell. The single crystal elastic constants were determined using both the unrelaxed and relaxed atomic positions. It has been found that relaxation has significant effect on the single crystal elastic constants. The nature of chemical bonding and electronic charge transfer in TiB has been studied to provide insight into its superior mechanical properties such as high hardness and high stiffness values.

9:25 AM
Bond-Order Potentials for Magnetic Transition Metals: Application to Defect Behaviour in Iron: *Duc Manh Nguyen*[1]; Guo Qiang Liu[2]; David G. Pettifor[3]; [1]UKAEA Fusion, Theory & Modlg., Culham Sci. Ctr., Abingdon, Oxfordshire OX14 3DB UK; [2]Chinese Academy of Science, Inst. of Physics, PO Box 603, Beijing 100080 China; [3]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK

Ferritic steels are the prime candidate materials for structural components of future fusion power plants. The development of genetic methods for modelling iron-based alloys under neutron irradiation is an issue of significant importance for the international fusion programme. The conventional molecular dynamic treatments of transition metals involve embedded atom models or Finnis-Sinclair potentials. However, ferromagnetic properties of iron are poorly described by such central force schemes, because the stability of the bcc crystal structure arises from magnetic effects, which in turn depend on local environments. In this talk, a robust and real space tight-binding bond-order potential is formulated for transition metal alloys by including both directional bonding and magnetism to describe accurately the complexities introduced by ferromagnetism. Physical properties are determined self-consistently within the Stoner model of band magnetism. The constructed BOP was applied in modelling point defects with special focus on magnetic and energetic properties of different interstitial configuration.

9:45 AM
Molecular Dynamics Study of Mutli-Cycle Single Asperity Contact: *Jun Song*[1]; Pil-Ryung Cha[2]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Bowen Hall, 70 Prospect Ave., Princeton, NJ 08540 USA; [2]Kookmin University, Sch. of Metallurgl. & Matls. Engrg., 861-1, Chongnung-Dong, Songbuk-Gu, Seoul 136-702 Korea

Many MEMS devices employ mechanical contacts as high frequency electrical switches. These contacts occur on the nano- and micro-scales. Elastic/plastic deformation, adhesion and morphology evolution all play important roles in the operation of these devices. We perform a molecular dynamics simulation of single asperity (EAM) Au contacts under cyclic loading and unloading. We monitor the full force-displacement curve, the evolution of the atomic structure/asperity morphology, dislocation nucleation and motion. We correlate features in the force-displacement relation to both dislocation motion and morphology evolution. One common feature of the simulations is the transfer of atoms from one side of the asperity contact to the other. The material transport is related to both adhesion and plastic deformation. We separate these effects by modify the interatomic potential to controllably reduce adhesion.

10:05 AM
Impurity Effects on Grain Boundary Fracture at the Atomic Scale: *Diana Farkas*[1]; Brian Hyde[1]; Margarita Ruda[2]; [1]Virginia Tech, Dept. of Matls. Sci., 201 Holden Hall, Blacksburg, VA 24060-0237 USA; [2]Centro Atomico Bariloche, Bariloche Argentina

We investigate the mechanisms of fracture behavior in bi-crystals and nano-crystalline bcc Fe at the atomic scale using empirical force laws and molecular level simulations. The simulations are focused on modeling how the presence of interstitial impurities affects the behavior of grain boundaries during fracture. The results show that C interstitial impurities strengthen the grain boundaries in both bi-crystals and the nano-crystalline material, inhibiting grain boundary failure. For very small grain sizes, the presence of interstitial C also inhibits the process of grain boundary sliding. H impurities, on the other hand, strongly promote grain boundary fracture in bi-crystals.

10:25 AM Break

10:35 AM Invited
Smoothed Atom Mechanics: A Meshless Quasicontinuum: *Ronald E. Miller*[1]; Ellad Tadmor[2]; [1]Carleton University, Mech. & Aero. Engrg., 1125 Col. By Dr., Ottawa, ON K1S 5B6 Canada; [2]Technion, Mechl. Engrg., Technion City, Haifa 32000 Israel

The Quasicontinuum (QC) method is a multiscale technique based on the idea of representative atoms and finite element interpolation, which greatly reduces the number of required degrees of freedom in an atomistic problem without significant loss of accuracy. The method offers a seamless transition from fully-atomistic regions to coarsened continuum regions by changing the density of atoms visited in the calculation, i.e. the representative atoms. The QC method has been very successful in treating two-dimensional (2D) problems, however when extended to three dimensions (3D) it is found that the gain in degree-of-freedom reduction can sometimes be offset by the cost of the 3D meshing associated with the finite element interpolation. This is a significant shortcoming, since most problems of interest in atomic-scale mechanics are 3D in nature. In this talk, we introduce a reformulation of the QC method within a meshless framework using the recently developed "local optimal point interpolation" (LOPI) scheme. We refer to the new approach as Smoothed Atom Mechanics (SAM) to

highlight its connection with smoothed particle hydrodynamics. SAM provides a more natural framework than QC for adaptive refining and coarsening of the model. In 3D, it is significantly faster than QC due to the elimination of the need for mesh generation. It is also a framework that can be easily and efficiently parallelized. Details of the new formulation will be presented, along with preliminary examples that highlight the capabilities of the method.

11:10 AM Invited
Dislocation Structure, Phase Stability and Yield Stress Behavior of Platinum Group $L1_2$ Intermetallics: Combined Ab-Initio-Peierls-Nabarro Model Approach: *Oleg Y. Kontsevoi*[1]; Yuri N. Gornostyrev[1]; Arthur J. Freeman[1]; [1]Northwestern University, Physics & Astron., 2145 N. Sheridan Rd., Evanston, IL 60201 USA

The dislocation structure and mobility are among the key phenomena governing the deformation and fracture behavior of intermetallic alloys. We present the results of fundamental comparative studies of the dislocation properties and the mechanical behavior for a class of intermetallic alloys based on platinum group metals (PGM) which are being developed for ultra-high temperature applications: Ir_3X and Rh_3X (where X = Ti, Zr, Hf, V, Nb, Ta). To connect the microscopic and mesoscopic scales in the analysis of dislocation structure and mobility, we employ a combined approach based on highly accurate first-principles calculations of the shear energetics and the modified semi-discrete 2D Peierls-Nabarro model with an ab-initio parametrization of the restoring forces. Based on our analysis of dislocation structure and mobility, we provide predictions of temperature yield stress behavior of PGM-based intermetallics, show that their dislocation properties are closely connected with the features of electronic structure and $L1_2$ -> $D0_{19}$ structural stability, and demonstrate the dramatic difference in dislocation structure and the mechanical behavior between PGM alloys with IVA and VA group elements. Supported by the AFOSR (grant No. F49620-01-1-0166).

11:45 AM
Atomistic Predictions of Non-Planar Dislocation Core Structures in fcc Iridium Using Bond-Order Potentials with Greens Function Boundary Conditions: *Marc J. Cawkwell*[1]; Duc Nguyen-Manh[2]; David G. Pettifor[3]; Vaclav Vitek[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Engrg., 3231 Walnut St., Philadelphia, PA 19104 USA; [2]UKAEA Fusion, Culham Sci. Ctr., Abingdon OX14 3DB UK; [3]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

Iridium is a promising material for use in aggressive environments due to its corrosion resistance and high melting temperature. However, unlike any other fcc metal, it can undergo brittle transgranular cleavage at temperatures up to 500°C, the origin of which is likely hidden in the atomic level properties of dislocations. We have developed Bond-Order Potentials for atomistic simulation of extended defects in iridium that are an excellent description of cohesion. Simulation of the core structure of the screw dislocation in iridium using Greens function boundary conditions has highlighted two possible configurations; a planar core dissociated into Shockley partials and a metastable non-planar core spreading into intersecting {111} planes. We have evaluated the Peierls stress of these configurations and suggest the limited mobility of the non-planar core could contribute to the strong work hardening and brittle cleavage of iridium. Research supported by the DOE BES grant. No. DE-PG02-98ER45702 (MJC, VV).

12:05 PM
Yield Criterion for Plastic Flow in Body-Centered Cubic Metals Based on Atomistic Modeling of Glide of Screw Dislocations: *Roman Gröger*[1]; Vaclav Vitek[1]; Vikranth Racherla[2]; John L. Bassani[2]; Luzhong Yin[3]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Engrg., 3231 Walnut St., Philadelphia, PA 19104 USA; [2]University of Pennsylvania, Dept. of Mechl. Engrg. & Applied Mech., 297 Towne Bldg., 220 S. 33rd St., Philadelphia, PA 19104 USA; [3]Rensselaer Polytechnic Institute, Scientific Computation Rsch. Ctr., 110 8th St., CII-7013, Troy, NY 12180 USA

Plastic deformation of body-centered cubic (bcc) metals is controlled by the glide of a/2<111> screw dislocations owing to their low mobility resulting from non-planar cores. Upon loading such cores transform and become asymmetric which is responsible for complex asymmetries and orientation dependencies of the yield and flow stress. In order to elucidate these phenomena we performed molecular statics modeling of the motion of an a/2[111] screw dislocation in bcc molybdenum loaded by combination of shear stresses perpendicular and parallel to the Burgers vector. While shear stresses perpendicular to the slip direction cannot induce glide, they alter the core structure and affect significantly the glide process. Based on these results, we have formulated a general yield criterion that includes both shear stresses parallel and perpendicular to the slip direction. This criterion is verified by comparing its predictions with experimental observations of slip traces in single crystals of molybdenum at low temperatures.

12:25 PM
Solid-Solution Softening Trends in BCC Mo by First Principles: *Dallas R. Trinkle*[1]; Richard G. Hennig[2]; Thomas J. Lenosky[2]; Satish Rao[1]; Christopher Woodward[1]; [1]Air Force Research Laboratory, MLLMD, Bldg. 655, 2230 10th St., Wright Patterson AFB, OH 45433-7817 USA; [2]Ohio State University, Dept. of Physics, 174 W. 18th Ave., Columbus, OH 43210 USA

Solid solution softening observed in the group VA and group VIA transition metals has traditionally been attributed to either extrinsic—such as interstitial scavanging—or intrinsic—direct solute/dislocation interaction—effects. We investigate intrinsic mechanisms using first principles methods. First, density functional theory calculates the change in the primary Peierls barrier when Re, Hf, Os, W, Ir and Pt solutes are introduced along a straight a/2<111> screw dislocation in Mo. Here the local strain field associated with the dislocation core is self-consistently coupled to the long-range elastic field using the recently developed lattice Greens Function Boundary Condition method. We compare with classical solute potential results and the work of Fleischer to understand the effect of size and modulus misfit on softening in bcc transition metals. The connection of classical potentials with ab initio data allows the extension of chemically accurate calculations to physically relevant length scales.

Computational Thermodynamics and Phase Transformations: Grain Boundaries and Interfaces I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Monday AM Room: 3005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Corbett C. Battaile, Sandia National Laboratories, Matls. & Process Modlg. Dept., Albuquerque, NM 87185-1411 USA

8:30 AM Keynote
Anisotropic Grain Boundary Properties: *David J. Srolovitz*[1]; Hao Zhang[1]; Mikhail I. Mendelev[1]; [1]Princeton University, Mechl. & Aeros. Engrg., Princeton, NJ 08540 USA

Grain boundary structure and properties depend on five distinct crystallographic variables: three to describe the relative orientation of one grain with respect to the other and two to describe the boundary plane. The evolution of polycrystalline structures may depend upon the anisotropy in grain boundary mobility, grain boundary free energy/stiffness, efficiency with which the boundary absorbs defects. In this presentation, we focus upon grain boundary properties that are important for quantitative modeling of the evolution of polycrystalline microstructures as a function of these crystallographic parameters (i.e., grain boundary mobility and grain boundary stiffness). We discuss how to determine these properties using molecular dynamics simulations. Finally, we compare predicted grain boundary dynamical properties with experimental measurements to draw some conclusions on what controls the rate at which polycrystalline structures evolve.

9:15 AM Invited
Grain Boundary Energy Anisotropy: Modeling and Implications: *James A. Warren*[1]; [1]National Institute of Standards and Technology, Metall./CTCMS, 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20899 USA

The excess free energy associated with a grain boundary depends on both the misorientation and the inclination of the plane between the bounding grains. In two dimensions two parameters are required to specify the energy, while in three dimensions five are required. Here we explore several methods for incorporating this anisotropy into a phase field model of grain boundaries, explore the physical consequences of each approach, compare analytic models with MD simulations, and examine the implications in modeling the dynamics of grain rotation.

9:45 AM
Dependence of Grain Boundary Mobility on Boundary Plane: *Hao Zhang*[1]; Mikhail I. Mendelev[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Bowen Hall, 70 Prospect Ave., Princeton, NJ 08540 USA

Quantitative grain boundary mobility data is a pre-requisite for quantitative predictions of such microstructural processes as grain growth and recrystallization. Such data has never been determined experimentally or via simulation as a function of boundary plane. We present the results of a series of 3-d molecular dynamics simulations in which the grain boundary mobility was determined as a function of boundary plane and temperature. Elastic strains are used to drive the motion of a series of nominally flat Sigma5 <001> tilt grain boundaries in Ni. We also determine the activation energies for boundary migration and compare how they vary with boundary plane with similar data on self-diffusion along the same grain boundaries. This similarity in the data provides some hints to the basic mechanism of grain boundary migration.

10:05 AM
An Atomistic Study of Grain Boundary Wetting: *Ho-Seok Nam*[1]; Mikhail I. Mendelev[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., 70 Prospect Ave., Princeton, NJ 08544 USA

There are many examples in which a liquid metal in contact with a polycrystalline solid of a different composition develops a deep liquid groove at the intersections of the grain boundaries and the solid-liquid interface. In many cases, the liquid film quickly penetrates into the solid along the grain boundary. The rate of propagation of such liquid layers can be greatly accelerated by the application of even modest stresses. This is a form of liquid metal embrittlement. We have performed a series of molecular dynamics simulations using a set of binary embedded atom method potentials that can be adjusted to vary the thermodynamic properties of the system. We report on how the liquid films propagate as a function of temperature, solubility, melting point difference and grain boundary character. These results are compared with general trends gleaned from a series of experimental studies in the literature.

10:25 AM Break

10:40 AM Invited
On Computer Simulations of Zener Pinning: *Mark Andrew Miodownik*[1]; Azmir Harun[1]; Mike Clode[1]; Elizabeth Ann Holm[2]; [1]King's College London, Mechl. Engrg., Strand, London WC2R 2LS UK; [2]Sandia National Laboratories, Matls. & Process Modlg., Albuquerque, NM USA

Zener pinning is an enigmatic phenomenon that has so far resisted a complete theoretical understanding due its inherent multi-scale complexities. In this talk we briefly review the successes and failures of the different theoretical approaches to understanding this phenomenon. We the present new work which compares the ability of three different types of microstructural model to simulate Zener pinning: The Phase Field model, the Front Tracking model and the Monte Carlo Potts model. The same 3D test geometry is simulated using each method. This is an hexagonal network with spherical particles located at the centre of each hexagonal grain. The hexagonal grain network provides a constant driving force for a moving boundary and includes triple line and quadruple point motion. This geometry allows detailed investigation of the boundary/particle interaction. The shape of the boundaries during bypass are compared with theoretical predictions and previous simulations. The pinning force acting on the migrating curved grain boundary is also calculated and compared with theoretical predictions for each model. The advantages and disadvantages of each type of model are highlighted. The ability of the models to simulate the pinning force of non-spherical and incoherant particles is also discussed.

11:10 AM Invited
Insights from Large Scale Modeling of Grain Growth Including Misorientation Dependent Boundary Mobility and Diffusive Solute Drag: *Koenraad G.F. Janssens*[1]; Elizabeth A. Holm[1]; [1]Sandia National Laboratories, PO Box 5800, MS 1411, Albuquerque, NM 87111 USA

In technical materials, grain growth is a microstructure transformation process of high complexity. The evolution of its statistically relevant quantities, such grain size, shape and (mis-)orientation distribution, is highly dependent on locally interdependent variables, such as grain boundary energy and mobility and local solute activity. Adding to the complexity, phenomena like anomalous grain growth may be determined by processes with a low occurrence probability (e.g. 1 in 100000 grains). This fact makes large-scale simulations of the evolution of a large number of grains of interest. We will present the development and applications of a massive-parallel-type, irregular, shapeless cellular automata based computational model for grain growth including crystallography and solute drag. Results provide insight into the collective nature of the grain growth process. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

11:40 AM
Effects of Fe and Mg Co-Segregation on Boundary Migration in Al: *Mikhail I. Mendelev*[1]; David J. Srolovitz[1]; [1]Ames Laboratory, Ames, IA 50011 USA

Even small concentrations of impurities can dramatically modify the mobility of grain boundaries in otherwise pure metal. Analytical models describing this effect consider only one type of impurities while real materials contain several different impurities. We developed an extension of the Cahn-Lucke-Detert continuum model for the case in which a material contains two types of impurities. In order to apply this analytical model to the case of Al containing Fe and Mg as impurities, we need to know the grain boundary mobility in pure Al, the Fe and Mg diffusivities and their heats of segregations to a grain boundary in Al. These data are obtained from molecular dynamics simulation. The interatomic potentials are fit to a wide range of crystal, liquid and defect properties in Al-Fe and Al-Mg alloys. Using these data we predict the dependence of the grain boundary mobility on Mg concentration in Al sample which already contains small amount of Fe impurities.

12:00 PM
Diffusion Mechanism at the Grain Boundaries in Two-Dimensional Metals: Gennady Mikhailovich Poletaev[1]; Roman Juryevich Rakitin[1]; *Mikhail Dmitrievich Starostenkov*[1]; [1]Altai State Technical University, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia

The present paper is concerned with the research of diffusion mechanism at the grain boundaries in two-dimensional metals by the method of molecular dynamics. The diffusion of atoms was studied on the example of three metals: Ni, Al, Cu. The packing of two-dimensional crystals was corresponded to the plane [111] of FCC lattice. The researches were made, using two types of the potentials of interatomic interaction: Morse pair potentials and multipartial Finnis-Sinclair potentials. The mechanism, the dependences of the coefficient of diffusion at the grain boundaries in the dependence on temperature, mutual orientation of grains, free volume, energy of grain boundaries were studied. It was found, that leading diffusion mechanism at the lowangle grain boundaries of two-dimensional metals was the result of interaction of grain boundary dislocation pairs. The creep of dislocation pairs in opposite sides was observed in the experiments.

Converter and Fire Refining Practices: Plenary

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee
Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Monday AM Room: 2016
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Alistair G. Ross, Inco Ltd, Canadian Smelting & Copper Business, Copper Cliff, ON P0M 1N0 Canada; Tony Eltringham, BHP Billiton, Houston, TX 77056-3020 USA

8:30 AM Opening Comments

8:45 AM
Progress in Converting and Casting: *Theo Lehner*[1]; *Alistair G. Ross*[3]; [1]New Boliden, Rönnskar Smelter and Lulea University of Technology, Sweden; [3]Inco Ltd, Copper Cliff Ops., 18 Rink St., Copper Cliff, ON P0M 1NO Canada

Turning matte into anodes continues to be an area to watch: for visitors to see pyrometallurgy in action; for metallurgists to keep up with modern smelting furnaces and ever increasing production rates; for management to remain profitable using more tools than taught in school; and for environmentalists to detect the last small clouds from the ever cleaner smelter. Current converting practices have been screened and analyzed, and today, converting and casting is a modern industry. An impressive, though typical learning curve of converter productivity may be detected. Local conditions such as availability and type of raw materials, products extracted, cost factors, etc., have stimulated the development of add-ons or even alternatives to the still

dominant method of converting by Peirce-Smith technology. Turning blister copper into anodes follows a similar path, namely increased productivity and improved quality. We dare to predict that development will continue.

9:15 AM
Recent Developments on the Peirce-Smith Converting Process at the Rönnskär Smelter: *Magnus Ek*[1]; *Peter Olsson*[1]; [1]New Boliden, Smelter Operations, Se-932 81, Skelleftehamn Sweden

After the installation of three new converters as part of the recent expansion of the Rönnskär smelter completed in 2000 work continues to further improve the converter operation. The converter upgrade led to an increase in blister copper taps from 150 tons per batch to more than 300 tons. The total cathode copper production increased from 130,000 tons in 1999 to 230,000 tons in 2001, the first year in full production. In recent years focus has been to improve operating practice, maximize the blowing time, extend the converter campaigns hence to reduce the specific brick consumption and improve the converter slag composition.

9:45 AM
Developments in Peirce-Smith Converting at Inco's Copper Cliff Smelter During the Past 35 Years: *Tony Warner*[1]; Jin Liu[1]; Frank Javor[2]; Randy Lawson[2]; Warren Shellshear[2]; Tien V. Hoang[2]; Ron Falcioni[2]; [1]Inco Technical Services Limited, 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada; [2]Inco Limited, Copper Cliff Smelter, Copper Cliff, Ontario P0M 1N0 Canada

At the Copper Cliff Smelter, Inco Limited has extensive experience in converting primary smelting mattes over a broad range of nickel/copper ratios and compositions. Over the past 35 years, technology developments at Inco have reduced the number of Peirce-Smith converters in Nickel/Bulk service from thirteen to five. This paper will discuss; the performance and operating life of "stretch" 13.7 m. vessels compared to conventional 10.7 m. converters; process control and information system development from the early 1980's, to monitor converter aisle status on a continuous basis, to the recent central control of the "stretch" converters; nitrogen cooling of "finishing" matte, from plant testing to implementation in the early 1990's, to replace the dedicated cooling converters; the impact on converting due to changes in the smelter flow sheet in the early 1990's; as well as a review of a current program, under consideration, to develop a future converting strategy to address environmental issues and smelter profitability.

10:15 AM Break

10:30 AM
Control and Optimization of Converting Practices: *Florian Kongoli*[1]; Ian McBow[1]; Robert Budd[2]; S. Llubani[1]; [1]FLOGEN Technologies Inc., Metals Dept., 5757 Decelles Ave. # 511, Montreal, QC H3S 2C3 Canada; [2]FLOGEN Technologies Inc., 3422 Old Capitol Trail, #791, Wilmington, DE 19808 USA

Converters have been playing an important role in the extraction and processing of metals ever since the first type of converter was invented about 100 years ago. Historically their operation has been mainly based on the experience of talented operators rather than on strict control and optimization technologies. However, in the new realities of unavoidable necessity to increase productivity and in the conditions of diversification of primary materials from various geographical areas, improving control and optimization techniques have become indispensable. In this paper a review of the authors' previous work in terms of improving control and optimization of converter operational practices related to magnetite content and precipitation, slag and metal blows, problematic and dangerous elements such as As, Sb etc. has been described by highlighting the advantages that the new control and optimization methods and tools bring to everyday converter practices. Several examples are given and future work is underlined.

11:00 AM
High Oxygen Shrouded Injection at Falconbridge: 5 Years of Operation: *Joel P. Kapusta*[1]; *Hans Strickling*[2]; William Tai[2]; [1]Air Liquide Canada Inc., 90, Boul. Marie-Victorin, Boucherville, Québec J4B 1V6 Canada; [2]Falconbridge Limited, Falconbridge, Ontario P0M 1S0 Canada

Although Peirce-Smith converting remains the workhorse in matte converting, the copper and nickel industries are still confronted with production hindrances such as tuyere blockage, excessive refractory wear and limits in the oxygen enrichment levels of the blast air. The knowledge acquired on high-pressure gas injection technology as well as the invention of the concentric tuyere for steelmaking led to the development of the Air Liquide Shrouded Injector (ALSI) technology for copper and nickel converting. Following a technology demonstration in 1997-98 with four shrouded injectors installed in a Peirce-Smith converter, Falconbridge Limited implemented ALSI Technology on a commercial scale in May 1999 as an integral part of its new Slag Make Converter at Sudbury, Ontario, Canada. The paper presents the various aspects of commissioning ALSI Technology, as well as the operating changes and injector design modifications over the five years the shrouded injectors have been in operation.

11:30 AM
Improving Inco's MK Reactor Performance Through Application of Praxair's Coherent Jet (CoJet) Technology: Tony Warner[1]; *Jin Liu*[1]; Dave McCann[2]; Dave E. Hall[2]; Dave Malette[2]; Jennifer A. Bradley[2]; Eric Mackenzie[3]; Adrian Deneys[4]; [1]Inco Technical Services Limited, 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada; [2]Inco Limited, Copper Business, Copper Cliff Smelter, Copper Cliff, Ontario P0M 1N0 Canada; [3]Praxair Canada Inc., 1 City Centre Dr., Ste. 1200, Mississauga, Ontario L5B 1M2 Canada; [4]Praxair Metals Technologies, 1500 Polco St., Indianapolis, IN 46224 USA

A novel low-pressure adaptation of Praxair's coherent jet technology, utilizing "CoJet" lances, was developed for application at Inco's Copper Cliff Smelter where oxygen is available in large tonnage quantities, but only at relatively low supply pressures (< 28 psig header pressure). The project objectives were to evaluate the effect of coherent jet technology on controlling accretion formation at the tip of the oxygen lance and also to increase the oxygen utilization efficiency in the Inco "Oxygen Top-Blowing Nitrogen Bottom-Stirring" (TBBS) MK Reactor, used for converting nickel-contaminated chalcocite to semi-blister copper. This paper describes the development and subsequent implementation and testing of two CoJet lances in this application.

Corrosion Sensors and Monitoring

Sponsored by: Structural Materials Division, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS)
Program Organizers: James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA; Raúl B. Rebak, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

Monday AM Room: 3018
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Raúl B. Rebak, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

8:30 AM
Microelectromechanical Systems (MEMS) for Detecting Corrosion: *Mario H. Castro-Cedeno*[1]; [1]Rochester Institute of Technology, Mfg. & Mechl. Engrg. Tech., 78 Lomb Memorial Dr., Rochester, NY 14623-5604 USA

Microelectromechanical systems (MEMS) are devices that extend integrated circuits (IC) technology by adding sensing elements and/or actuators to the silicon chip. The goals are a complete system on a chip and significant reductions in system size, weight and cost. MEMS are ideal for detecting and monitoring corrosion because they enable real-time health monitoring of structures or components under operational conditions. They can use well-known techniques for detecting corrosion such as linear polarization resistance, acoustical noise and electrochemical measurements. In this paper, examples of MEMS sensors for corrosion detection and monitoring are proposed and test results of a prototype device based on linear polarization resistance are presented.

9:00 AM
Electrochemical Characterization of Nanocrystalline Surface of Alloy 22: *Krishnan Selva Raja*[1]; Manoranjan Misra[1]; Shantanu A. Namjoshi[1]; [1]University of Nevada, Metallurgl. & Matls. Engrg., 1661 N. Virginia St., MS 388, Reno, NV 89557 USA

A nano-crystalline surface with well defined grain size of less than 20 nm could be achieved by localized surface deformation followed by a low temperature annealing. This paper will discuss the processing steps of creating an optimized nano-crystalline surface layer on Alloy-22, a Ni-Cr-Mo-W alloy. Preliminary studies of surface modification of Alloy 22 by conventional shot peening followed by annealing at 300-350°C for 1-2 h showed improved passivity and increased charge density of the passive film. Scanning Kelvin Probe studies are being carried out to investigate the potential gradients across the passive

films of nano-crystalline surface. Results of these studies coupled with TEM analyses will help understand the underlying mechanism for improved corrosion resistance of nano-structured surface.

9:30 AM
Monitoring the Evolution of the Corrosion Potential and Corrosion Rate of Alloy 22 Immersed in Various Electrolyte Solutions: John C. Estill[1]; Gary A. Hust[1]; Kenneth J. King[1]; *Raul B. Rebak*[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

Alloy 22 (N06022) is a Nickel-Chromium-Molybdenum (Ni-Cr-Mo) alloy, which has excellent general and localized corrosion resistance. Depending on the exposed electrolyte composition and temperature, the corrosion potential (Ecorr) of Alloy 22 may gradually increase as the time increases. The raise in Ecorr (ennoblement) is generally accompanied by a reduction in the corrosion rate, suggesting the formation of a protective oxide film on the alloy surface. Results are presented to show the correlation between Ecorr and the corrosion rate in various electrolyte solutions and temperatures. Studied variables included ratio of chloride to nitrate, pH and creviced vs. non-creviced testing specimens.

10:00 AM Break

10:15 AM
Early Corrosion Detection by Magnetoelastic Techniques and Electrochemical Impedance Spectroscopy: Francisco Rumiche[1]; Alberto Polar[1]; Varsha Singh[1]; George M. Lloyd[1]; Ming L. Wang[1]; *J. Ernesto Indacochea*[1]; [1]University of Illinois, Dept. of Civil & Matls. Engrg., 842 W. Taylor St., MC 246, Chicago, IL 60607 USA

A sensor based on magnetoelastic techniques was developed as a nondestructive technique to detect early corrosion in carbon steel cables and tendons used in stay bridges and reinforcements. The magnetic response in terms of the effect of corrosion on conductivity and permeability of the material was monitored by adjusting the input current signal to localize the induced magnetic field in a depth between 0.01 and 1.5 mm. Conventional electrochemical techniques and Electrochemical Impedance Spectroscopy were used to follow the development and establish the early stages of corrosion. The results of these techniques were correlated to the measurements of the magnetoelastic sensor. Furthermore, optical microscopy, X-Ray analysis, electron scanning microscopy with the electron dispersion spectroscopy probe and Raman spectroscopy were used to characterize systematically the passive films and corrosion products correlating the results with the measurements of the magnetic response of the surface of the carbon steel specimens.

10:45 AM
The Use of Electrochemical Impedance Spectroscopy (EIS) to Measure the Corrosion of Metals in Contact with Wood: *Samuel L. Zelinka*[1]; Douglas R. Rammer[1]; [1]USDA Forest Products Laboratory, Condition Assessment & Rehabilitation of Struct., 1 Gifford Pinchot Dr., Madison, WI 53726 USA

Recent replacement of traditional preservative treatments with newer, more corrosive chloride based preservatives highlight the ineffectiveness of current fasteners to resist corrosion and the need for tools to predict the service life (durability) of a bare or coated fastener in preservative treated wood. One possible technique to quantify the increased corrosivity of the new preservative treatments is use of Electrochemical Impedance Spectroscopy (EIS). EIS is well suited to measuring the corrosion in wood because it can be used in situ, works well in high resistance media, and does not permanently polarize the ions in the wood. While EIS is used to measure corrosion in other materials, this technique has not been developed for the wood environment. We will present preliminary results on the utilization of EIS to measure the corrosion rate of metals in wood.

11:15 AM
Corrosion Monitoring in Kraft Digesters with EN Probes: *Steven J. Pawel*[1]; [1]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Bldg. 4500-S, MS-6156, Oak Ridge, TN 37831 USA

Electrochemical noise (EN) probes were deployed in two continuous kraft digesters at a variety of locations representative of corrosion throughout the vessels. Current and potential noise, the temperature at each probe location, and the value of up to 60 process parameters (flow rates, bulk liquor chemistry, etc.) were monitored continuously during each experiment. The results indicate that changes in furnish composition and process upsets were invariably associated with concurrent substantial changes in EN activity throughout the vessels. Post-test evaluation of the mild steel electrode materials in both vessels confirmed general corrosion of a magnitude consistent with historical trends in the respective vessels as well as values qualitatively (and semi-quantitatively) related to EN current sums for each electrode pair. Stainless steel electrodes representing 309LSi and 312 overlay repairs exhibited zero wastage corrosion - as did the actual overlays - but the EN data indicated periodic redox activity on the stainless steel that varied with time and position within the vessel. Little or no correlation between EN probe activity and other operational variables was observed in either vessel.

Extractive Metallurgy: Pyrometallurgy I

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Monday AM — Room: 2018
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Edgar E. Vidal, Colorado School of Mines, Metall. & Matls. Engrg., Golden, CO 80401 USA; Tom Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA

8:30 AM
Separation of Al_2O_3 Inclusions Across Interfaces Between Molten Steel and Ladle-, Tundish- and Mold-Slags: *George N. Shannon*[1]; S. Sridhar[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The modeling of inclusions separating across a steel-slag interface previously introduced by Nakajima/Okamura and Bouris/Bergeles is expanded to include different inclusions shapes (octahedral and plate-like, besides spherical) as well as comparison between model ladle-, tundish-, and mold-slags. Investigation of the relation of these shapes with the interfacial capillary force - which arises due to the dynamic interfacial energy during separation - has shown that the force has a significant effect on whether the inclusion separates rapidly or settles at the interface. For the slags considered, the most significant physical property is found to be viscosity, which results in fast separation and dissolution times for the mold and ladle slags, relative to the more viscous tundish slag. Particle dissolution (using experimental data) is included in separation modeling. Deformable inclusions are also discussed, wherein the shape-change force deflects some of the inclusion's inherent driving force to separate.

9:00 AM
Process for Converting SO_2 to Sulfur Without Generating Pollutants Through Reactions Involving BaS and $BaSO_4$: *Hong Yong Sohn*[1]; Marijanka Savic[1]; Rafael Padilla[2]; Gilsoo Han[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [2]University of Concepcion, Dept. of Metallurgl. Engrg., Concepcion, Chile

Nonferrous smelters and coal gasification processes generate sulfur dioxide streams, most of which are treated to produce sulfuric acid with the accompanying problems of market shortage and transportation difficulties. A method for converting sulfur dioxide to elemental sulfur by a cyclic process involving barium sulfide and barium sulfate without generating solid wastes has been developed. In this process, barium sulfate is reduced by a suitable reducing agent such as hydrogen to produce barium sulfide, which is used to reduce sulfur dioxide and produce elemental sulfur vapor and regenerate barium sulfate. The latter is then reduced to reproduce barium sulfide. Thermodynamic analysis and experimental results indicated that the BaS-SO_2 reaction produces mainly sulfur vapor and solid barium sulfate and that the gaseous product from the $BaSO_4$-H_2 reaction is mainly water vapor. The rates of the two reactions are reasonably rapid in the temperature range 700-1050°C.

9:30 AM
A Thermodynamic Model of Combustion Zone in Lead Blast Furnace: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

A thermodynamic model has been developed to model the distribution behaviors of Cu, Fe, S, O, Pb, Zn, As, and heat balance in lead blast furnace. The model results have been validated by the industrial data of Kazzinc in Kazakhstan. It can be predicted for any set of controllable

process parameters such as feed composition, smelting temperature, degree of oxygen enrichment and volume of oxygen-enriched air. The effects of the blast air, industrial oxygen, and coke charge on the distributions of Cu, Fe, S, O, Pb, Zn, As, and heat balance, and lead loss in slag have been presented and discussed.

10:00 AM Break

10:15 AM Cancelled
The Interaction of a Highly Soluble Gas Jet and a Liquid

10:45 AM
Preparation of Magnesium Oxide with Low Calcium Oxide Content and High Specific Surface Area from Low-Grade Dolomite: *Xiang-Yang Zhou*[1]; *Jie Li*[1]; *Hong-Zhuan Liu*[1]; *Ye-Xiang Liu*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China

The low energy consumption preparation technology of magnesium oxide with low calcium oxide content and high specific surface area from low-grade dolomite has been investigated. The better magnesium oxide preparation technology conditions are the followings: (1)dolomite powder is partially calcined at 750-800°C for 2h, (2) solid-liquid ratio in calcined dolomite slurry should be 20~30g/l, (3) carbonation temperature, carbon pressure and carbonation time are less than 40°C,0.4~0.6Mpa and 1.5~2h respectively during carbonation process, (4) decomposition temperature of Mg(HCO3)2 solution should be more than 90°C, (5) calcination temperature of precursor is less than 850. More than 90% of magnesium can be extracted by the technology. The MgO product (CaO<0.38%) with a specific surface area of near 50m2-g-1 also can be obtained by the technology.

11:15 AM
Further Improvements and Applications of the Altair Hydrochloride Pigment Process: *Dirk Verhulst*[1]; Bruce J. Sabacky[1]; Bob Wang[1]; Douglas K. Ellsworth[1]; [1]Altair Nanomaterials, Inc., 204 Edison Way, Reno, NV 89502 USA

Altair owns a 5 t/day-feed pilot plant for the processing of ilmenite ores. It involves digestion in concentrated hydrochloric acid, solvent extraction to concentrate Ti in a purified stream, and spray hydrolysis to produce, after calcination and milling, a high quality pigment. All chloride streams are recycled. Our latest work includes a systematic study of the solvent extraction step via small-scale tests and modelization. Feedstocks other than ilmenite ores are also being explored. We are working on the treatment of oil sand tailings for the recovery of Ti and Zr and on supplying electrodes for the manufacture of Ti metal by molten salt electrolysis.

Friction Stir Welding and Processing III: Aluminum Alloys

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Monday AM Room: Nob Hill C/D
February 14, 2005 Location: San Francisco Marriott

Session Chair: Kumar V. Jata, Air Force Research Laboratory, WPAFB, OH 45433 USA

8:30 AM Welcome/Opening Remarks

8:40 AM Keynote
Commercialization of Friction Stir Welding - A Lab to Production Success Story: *Doug Waldron*[1]; Keith McTernan[1]; [1]Advanced Joining Technologies, 3030 Red Hill Ave., Santa Ana, CA USA

While the benefits of friction stir welding (FSW) have been widely published since 1995, demonstrations of its end use in production applications have been limited. This paper will present several examples of production transitions within several market sectors such as aerospace, marine, transportation (road), oil and gas, and extrusions. Further, the process of transitioning from research studies to production applications will be presented. Examples of product implementation begin with the first successful implementation of FSW into aerospace on The Boeing Company's Delta launch vehicle program to the first US marine implementation in both commercial and military market vessels. Transportation applications will also be discussed illustrating a high volume application potential supported by the FSW process. This paper provides an overview on a typical FSW project lifecycle that includes process development, mechanical testing, certification issues, and an economic business case for production implementation.

9:00 AM
Microcharacterization and Texture Analysis of Friction Stir Processed AA 5052 Alloy: *Michelle N. Adams-Hughes*[1]; Peter N. Kalu[1]; Marwan K. Khraisheh[2]; Namas Chandra[1]; [1]FAMU- FSU College of Engineering, Mechl. Engrg., 2020 Pottsdamer Rd., Rm. 229, Tallahassee, FL 32310 USA; [2]University of Kentucky, Mechl. Engrg., 210C Ctr. for Robotics & Mfg. Sys., Lexington, KY 40506- 0108 USA

Friction Stir Processing (FSP) was employed in the fabrication of AA 5052 alloy (0.11 Si, 0.25 Fe, 0.17 Cu, 0.03 Mn, 2.2 Mg, 0.25 Cr, 0.02 Zn, balance Al - all compositions are in wt.%). The material was characterized using, SEM, OIM and X-Ray diffraction. A correlation was made between the processing parameters (rotation and translation speeds) and the microstructure and the texture developed in the material. The rotation speed had a greater effect on the development of the microstructure than the translation speed. Lower rotation speed resulted in a finer grain size. The grain orientation, average grain misorientation, and grain misorientation spread for each processing condition is presented in this paper.

9:30 AM Invited
Influence of Thermal Environments on Friction Stir Welding Operating Windows: *R. J. Lederich*[1]; J. A. Baumann[1]; [1]The Boeing Company, PO Box 516, MC S245-1003, St. Louis, MO 63166-0516 USA

Successful FSW requires maintenance of the appropriate hot working temperature range and sufficient forging force for the duration of the weld. For a given FSW tool and weld setup (parts to be welded, anvil, and clamps), temperature is determined by the spindle speed (rpm), travel speed (ipm), and to a lesser extent, the forging force. We have produced lap welds joining 3.2 mm (0.125") thick 2024Al sheets to substrate configurations having widely different heat conduction characteristics. Temperatures were measured by installing thermocouples at identical locations along the length of the weld. We have determined the appropriate operating window for each weld setup and related it to its heat conduction characteristics. Welds were sufficiently long to enable determination of the steady-state operating window; this window was found to be much narrower than windows determined by shorter welds. Joint properties were determined at various points within the operating windows.

9:50 AM
Unpredictable Stress Corrosion Crack Growth in Friction Stir Welded 7075 Al: *Christian Fuller*[1]; Murray W. Mahoney[1]; Jesse B. Lumsden[1]; Leanna M. Micona[2]; Mike Hyatt[2]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]The Boeing Company, Seattle, WA USA

Stress corrosion (SC) crack growth behavior of friction stir (FS) welded 7075 Al was investigated with constant displacement double cantilever beam specimens. The chevron notch was located at the interface between the FSW nugget and the heat-affected zone. As-FS welded specimens were loaded at one of four locations: advancing side of tool and parallel to the weld direction; retreating side of tool and parallel to the weld direction; advancing side of tool and opposite the weld direction; or retreating side of tool and opposite the weld direction. SC crack growth depended on the loading location. Cracks loaded parallel to the weld direction ran straight providing both crack growth rate and threshold data, while cracks loaded opposite to the weld direction turned 90° from the loading axis. This work illustrates that SC crack growth in as-FS welded 7075 Al can be unpredictable and depends on loading direction.

10:10 AM
Effect of Processing Variables and Tool Design on the Corrosion Properties of FSW AA7050: *Jesse B. Lumsden*[1]; Christian Fuller[1]; Murray W. Mahoney[1]; [1]Rockwell Scientific Co., Matl. Scis., 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

Although melting does not occur during friction stir (FS) welding, temperatures are sufficiently high and times at temperature are long enough to cause changes in the microstructure. The altered microstructures in FS welded high strength Al alloys are sensitized making them more susceptible to stress corrosion cracking (SCC), intergranular corrosion, and pitting corrosion than the parent material. Recent investigations indicate that tool design and processing variables can have a significant impact on the SCC susceptibility of FSW AA7050. Pronounced improvements in SCC resistance are obtained by altering

the conventional threaded pen and flat shoulder geometries when the travel speed is optimized. These SCC results will be discussed and related to temperature measurements and an evaluation of the microstructure. This work was sponsored by the Office of Naval Research, Project N00014-02-0212.

10:30 AM Break

10:50 AM Cancelled
Progress in Friction Stir Welding of Thick Section Aluminium Materials

11:10 AM
Microstructure, Fatigue Crack Growth, and Corrosion in Friction Stir Welded Al 5456: *Peter S. Pao*[1]; Richard W. Fonda[1]; Harry N. Jones[1]; Brian J. Connolly[2]; Alison J. Davenport[2]; [1]Naval Research Laboratory, Washington, DC 20375 USA; [2]University of Birmingham, Birmingham UK

The microstructure, mechanical properties, fatigue crack growth and corrosion behavior of friction stir welded Al 5456 were investigated. TEM studies reveal substantial decreases in dislocation density in the HAZ and weld nugget, as compared to the base plate, giving rise to a uniformly lower microhardness across those regions. Longitudinal yield strength also decreases toward the center of the weld. In the nugget region, serrated flow initiates immediately upon yield, while in the base plate such serrated flow does not occur until a critical strain is reached. Fatigue crack growth rates in the HAZ are significantly lower and the fatigue crack growth threshold significantly higher than those in the weld nugget and the base plate. Anodic scans across the weld region, using a microelectrochemical cell, indicate the highest anodic reactivity in the nugget region. These results will be discussed in terms of the observed microstructure in various regions of the weld.

11:30 AM
Friction Stir Welding of Dissimilar Aluminum Alloys: *Robert Cook*[1]; William J. Arbegast[1]; Sean Long Fox[2]; Tito Handby[2]; [1]South Dakota School of Mines and Technology, Advd. Matl. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]Oglala Lakota College, PO Box 370, Kyle, SD 57752 USA

Friction stir welding has been shown to be successful in the joining of dissimilar aluminum alloys in longitudinal butt joints. The results of process parameter optimization studies for 0.25 inch thick 2024-T3 to 7075-T73 butt joints alternating on the advancing side and the retreating side are presented. The influence of processing parameters on advancing and retreating side alloy selection on X-, Y-, and Z- axes forces, observed metal flow patterns, and defect formation are presented in terms of the FSW Metalworking Model. Mechanical property, bend test, and intergranular corrosion testing of the dissimilar metal couples are discussed in terms of processing parameters.

11:50 AM
Fatigue of Pre-Corroded 2024-T3 Friction Stir Welds: Experiment and Prediction: *Ulises Alfaro*[1]; Tommaso Ghidini[1]; Claudio Dalle Donne[1]; [1]DLR -German Aerospace Center, Inst. of Matls. Rsch., Linder Hoehe, Koeln, Germany 51147 Germany

Friction Stir Welding has been identified as "key technology" by aerospace industry, due to the high mechanical properties of the joint. However, there is still lack of information about the damage tolerance of corrosion susceptible aluminum alloy FSW-joints (i.e. 2xxx, 7xxx). In this investigation 2024-T3 base metal and FSW-joints were pre-corroded with an alternate immersion technique (ASTM G44) in a 3.5% NaCl aqueous solution for 100, 250 and 1000h. SN fatigue strength of the pre-corroded specimens were carried out in laboratory air using different R-ratios (R=-1, 0.1, 0.5). Maximal pit geometry (width and depth) criteria were used as initial crack in a linear-elastic model to predict the fatigue life of the specimens. Numeric simulations were realized with help of AFGROW and NASGRO software. The obtained prediction results are quite close to the experimental data.

12:10 PM
The Effect of Spot Friction Welding (SFW) Parameters on the Strength and the Microstructure of Aluminum 6111-T4 Lap Joints: *David Mitlin*[1]; Tsung-Yu Pan[2]; Michael L. Santella[3]; Zhili Feng[3]; [1]University of Alberta, Cheml. & Matls. Engrg., Edmonton, Alberta T6G 2G6 Canada; [2]Ford Research and Advanced Engineering, Mfg. & Processes, Rm. 2345-1, M/D 3135, SRL Bldg., Dearborn, MI 48124 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6096 USA

Spot friction welding (SFW) is a new solid-state welding process that utilizes a rotating tool with a probe pin to create spot welds in lap configurations. Unlike the linear friction stir welding (FSW) process, where the tool moves in the transverse direction to form a continuous linear butt joint, in SFW the tool is not translated. Instead, the tool is retracted from the workpiece when the stirring process is finished at a particular spot. We examined the influence of weld parameters such as tool rotation speed, tool hold time and tool downward force on the strength of aluminum 6111-T4 lap-joints. We have also characterized the subsequent microstructures near the weld nugget, the thermo-mechanically effected zone and the heat affected zone using scanning and transmission electron microscopy. Specific issues addressed are the effect of weld parameters on texture evolution, on precipitate/zone reversion and subsequent re-formation, and on the dislocation content of the affected areas.

12:30 PM
Precision Friction Stir Welding of Vacuum Processing Chambers: *Jack Thompson*[1]; [1]General Tool Company, 101 Landy Ln., Cincinnati, OH 45215 USA

Processing chambers of high-vacuum semiconductor fabrication equipment have typically been machined from solid billets of aluminum, machined from aluminum castings, or dip-braze fabricated from pre-machined aluminum plates. Fusion weldments of aluminum plates have typically been avoided because of porosity in the welds, particularly when welds are machined for o-ring grooves. Friction Stir Welding is an attractive new alternative means of chamber fabrication because it combines the best attributes of solid wrought aluminum, including the absolute lack of porosity, with significant reductions in machining and wasted material. General Tool has recently completed a production run of these chambers, and has developed means of holding machining tolerances on the pre-machined internal features of the chamber.

Frontiers in Solidification Science: Morphological Evolution and Mushy Zone Phenomena I

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Monday AM Room: 2020
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Alain Karma, Northeastern University, Boston, MA 02115 USA; Peter W. Voorhees, Northwestern University, Evanston, IL 60208 USA

8:30 AM Opening Remarks

8:40 AM Keynote
Conduction-Limited Melting of Mushy Zones: *Martin E. Glicksman*[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg. Dept., 110 8th St., CII-9111110 8th St., Troy, NY 12180-3590 USA

The formation and melting of dendritic mushy zones of pivalic acid were observed under convection-free conditions as part of NASA's United States Microgravity Payload Mission, flown on the space shuttle Columbia in 1997. Video data show that in microgravity dendrites melt without relative motion or settling with respect to the quiescent melt phase. Individual mushy-zone fragments follow a square-root time-dependence as predicted using quasi-static conduction-limited theory. Agreement between theory and experiments is found when the melting of individual crystallites occurs under shape-preserving conditions (constant axial ratio). Interactions among crystallites in the mushy zone, however, inevitably result in thermal shielding and shape changes and, as melting progresses, other complicating effects such as capillarity enter the process. Our recent shape analysis of melting dendritic fragments show that slender crystallites (axial ratios up to 20) exhibit strong capillary effects when their major axis dimensions decrease below 5 mm.

9:25 AM Invited
Adiabatic Remelting of the Mushy-Zone During Rapid Solidification: *Douglas M. Matson*[1]; Robert W. Hyers[2]; [1]Tufts University, Mechl. Engrg., 025 Anderson Hall, 200 Coll. Ave., Medford, MA 02155 USA; [2]University of Massachusetts, Dept. of Mechl. & Industl. Engrg., Engrg. Lab Bldg., 160 Governers Dr., Amherst, MA 01003 USA

Following primary metastable recalescence, the first stage of microstructural evolution involves growth of the stable phase into the mushy-zone. The stable phase growth rate can be predicted using a simple dendrite growth model if the heat balance is modified to include

isothermal melting of the pre-existing solid. This adiabatic remelting model successfully predicts the growth rates for the stable phase as measured experimentally in the Fe-Cr-Ni alloy system. The growth rate of the stable phase depends strongly on composition, but this dependence is countered by a reduction in the heat absorbed through melting of the metastable solid. Due to the small variation in thermophysical properties over a wide range of compositions, the net result of these two competing effects is a constant heat flux which is independent of composition.

10:00 AM Invited
Transition of the Mushy Zone from Continuous Liquid Films to a Coherent Solid: *Michel Rappaz*[1]; Stephane Vernède[1]; [1]EPFL, Inst. of Matls., Computational Matls. Lab., Lausanne CH-1015 Switzerland

While studies of solidification microstructures have focused mainly on the tips of the dendrites, the last stage solidification is equally important from the point of view of defect formation (porosity, hot tearing), mechanical strength build-up and precipitation of phases. In particular, the transition from continuous liquid films to a coherent solid in low concentration alloys is of crucial importance for hot tearing formation, and more generally speaking for liquid feeding ability and coherency development. Based on a fairly recent theoretical model of coalescence which will be recalled briefly, new results obtained for a population of equiaxed grains will be presented. A granular-type model based on a Voronoï tessellation has been used for the description of the gradual disappearance of liquid films and the clustering of equiaxed grains. This percolation-type approach has been used then to calculate the pressure drop in the mushy zone on the assumptions of a Poiseuille flow in between the grains and a Kirchoff model for the connectivity of the liquid films including the Losses associated with solidification shrinkage (i.e, PKL model). Comparison with a standard average pressure drop calculation based on Carman-Kozeny's relationship will be presented.

10:35 AM Break

10:55 AM Invited
Phase-Field Simulations of Coupled Columnar and Equiaxed Dendritic Growth: *Christoph Beckermann*[1]; Arnoldo Badillo[1]; Juan C. Ramirez[1]; [1]University of Iowa, Dept. Mechl. & Industl. Engrg., 2412 SC, Iowa City, IA 52242 USA

Two-dimensional phase-field simulations of coupled columnar and equiaxed dendritic growth are performed for a binary alloy solidifying unidirectional under an imposed temperature gradient. The equiaxed grains nucleate and grow inside the constitutionally undercooled zone ahead of the columnar front. The phase-field model reduces to the sharp-interface equations in a thin-interface limit where kinetic and solute trapping effects are negligible. The computations are performed using an adaptive grid that moves with the solidification front. The competition between the columnar and equiaxed dendrites is investigated as a function of the nucleation undercooling, the equiaxed grain density, and the imposed cooling rate and temperature gradient. The conditions leading to a columnar-to-equiaxed transition are examined in detail and compared to theoretical models. Future needs in simulating grain structure development in solidifying alloys are discussed.

11:30 AM Invited
Cellular Pattern Selection in Directional Solidification: *Rohit Trivedi*[1]; Shan Liu[1]; Blas Echebarria[2]; Alain Karma[2]; [1]Iowa State University, Matls. Sci. & Engrg., 100 Wilhelm Hall, Ames, IA 50014 USA; [2]Northeastern University, Dept. of Physics, 110 Forsyth St., Boston, MA 02115 USA

Although significant advances have been made in the planar and dendritic interface growth, our understanding of cellular pattern formation has remained incomplete. Due to the strong coupling of solute field between the neighboring cells, significant variations in cell shapes and cell spacing are present for different cells in an array that has precluded a proper quantitative description of cellular growth. Through detailed experimental studies and phase-field modeling, we have found that all cells in an array follow a definite scaling law in that all cell shapes collapse (within optical resolution) onto a single shape when they are scaled with the local spacing. This shape interpolates between the two known limits of this problem: the Scheil equation and the 2D Saffman-Taylor (ST) equation which describes the Laplacian limit of directional growth. The former predicts the asymptotic shape of the interface far from the tip while the latter is found here to predict reasonably well the cell tip of experimental and numerical cell shapes. An expression for the cell tip undercooling is obtained from the results of the phase-field model, which is found to be in good agreement with the result based on the ST shape and with the result obtained experimentally in the succinonitrile-salol system. In the limiting case, when an intercellular eutectic is present, the cell shape is shown to follow accurately the solution of the 3D ST problem. Finally, it is found that the phase-field results are strikingly different for 2D shapes and 3D axisymmetric shapes: steady-state cells exist in 2D over a very wide range of spacing, but they only exist in 3D up to a certain maximum spacing only. The existence of this band gap in 3D cell solutions is consistent with previous numerical solutions of the 3D ST problem. Experimental results for 3D cells and phase-field results on 3D non-axisymmetric cells will be presented to give insight into the complex nature of the stability of cellular arrays in 3D.

12:05 PM
Crossover Scaling of Wavelength Selection in Directional Solidification of Binary Alloys: Michael Greenwood[1]; Mikko Haataja[1]; *Nikolas Provatas*[1]; [1]McMaster University, Matls. Sci. & Engrg. & Brockhouse Inst. for Matls. Rsch., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

The selection of microstructure length scales in metal alloys is critical to their mechanical properties. Understanding the kinetics of dendritic pattern selection during solidification is therefore essential to be able to optimize their performance. In this talk we report on simulated cellular and dendritic spacing selection in directional solidification of dilute binary alloys using a phase-field model solved with adaptive-mesh refinement. The spacing of primary branches is examined for a range of pulling velocities, thermal gradients and alloy compositions. It is found to undergo a maximum as a function of pulling velocity, in agreement with experimental observations. Using power spectral analysis we demonstrate that wavelength selection is unambiguously described by a crossover scaling function from the emergence of cellular growth to the onset of dendritic fingers. The construction of the crossover scaling function and length scales is discussed, and our results are validated using previously published experimental data on directional solidification.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Nanostructures and Nanocomposites I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Monday AM — Room: 3020
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Sungho Jin, University of California, Matls. Sci., La Jolla, CA 92093 USA; Anthony T. Fiory, New Jersey Institute of Technology, Physics, Newark, NJ 07102 USA

8:30 AM Keynote and Introduction by John C.C. Fan, Chairman and CEO, Kopin Corp. Highlights of J. Narayan's Contribution to Materials Science and Engineering

8:45 AM Invited
Laser Processing of Polymeric and Living Biomaterials: How Can We Leverage Natures Nanotechnology?: *Douglas B. Chrisey*[1]; [1]Naval Research Laboratory, 4555 Overlook Ave., Washington, DC 20375-5345 USA

Today, there are countless applications for thin film coatings made from novel biocompatible materials including medical implants, drug coatings, and bioelectronic interfacing and the implementation of these new materials is expected to improve both device capabilities and performance. Pulsed lasers have unique qualities that can be applied to process biomaterial thin films and they have proven to be an invaluable tool in the research and development. At NRL, we have developed advanced laser-based processing technologies for the deposition of biomaterials including polymers and living biomaterials. We have demonstrated the ability to fabricate novel 3-D tissue constructs using a unique laser forward transfer process that deposits cells using a CAD/CAM process engineered tissue constructs cell-by-cell in order to simulate native structured tissue. But we don't have to re-create the tissue construction with complete fidelity for once the cells communicate, they ride the path evolution has taught. Powered by this break-

through in biomaterial processing, we can now enhance understanding, development, and exploitation of the field of tissue engineering by the ability to group and order specific, defined populations of cells and bioscaffolding with precision. The goal is to demonstrate specific biological function by engineering tissue constructs consisting of well-defined heterogeneous mammalian cell populations.

9:15 AM Invited
Laser Preparation and Characterization of Different Iron-Based Core-Shell Nanostructures: Foreseen Applications: Josef Pola[2]; *Rodica Alexandrescu*[1]; Ion Morjan[1]; Ion Voicu[1]; Florian Dumitrache[1]; Iuliana Soare[1]; Lavinia Albu[1]; Monica Savoiu[1]; [1]National Institute for Lasers, Plasma & Radiation Physics, Lab. of Laser Photochmst., 111 Atomistilor St., PO Box MG 36, Bucharest 76900 Romania; [2]Academy of Sciences of the Czech Republic, Inst. of Cheml. Process Fundamentals, Laser Chmst. Grp., Prague 6 165 02 Czech Republic

The processing of coated magnetic nanoparticles raises great technological interest. Among others, coating provides a matrix for binding of the particles, prevents grain growth and agglomeration, prevents the surface oxidation of the nanoparticles and environmental degradation, would likely enhance the stiffness, toughness, and service life of the composite coating. As such they are potential candidates for applications in data storage, computing, sensing, medicine etc. Among different methods used for Fe encapsulation the laser pyrolysis method is one of the few techniques able to produce nanostructured composites in the nanometer size domain (around 10 nm or even less), with controlled size distribution and chemical composition. This presentation will focus on two examples in which the laser pyrolysis technique is used in a non-conventional geometry of irradiation for the synthesis of i) siloxane polymer-iron/iron oxide shell-core nanostructures (prepared by IR laser-induced and ethylene-photosensitized co-decomposition of iron pentacarbonyl and methoxytrimethylsilane) and ii) iron-carbon core-shell nanoparticles (obtained from ethylene/acetylene/Fe(CO)5 mixtures). The morphology; size distribution chemical content of the nanocomposites are demonstrated by different performant characterization methods.

9:45 AM Invited
Breakthroughs in Optimization of Mechanical Properties of Nanostructured Metals and Alloys: *Carl C. Koch*[1]; *Khaled M. Youssef*[1]; Ronald O. Scattergood[1]; K. Linga Murty[1]; Donald W. Brenner[1]; [1]North Carolina State University, Matls. Sci. & Engrg., 233 Riddick Bldg., 2401 Stinson Dr., Raleigh, NC 27695-7907 USA

The mechanical behavior of nanostructured materials has been a topic of great interest in recent years for the promise of superior properties. While it was predicted that both strength and ductility would be dramatically increased by decreasing grain size to the nanoscale, in general, the strength and hardness were indeed found to be greatly enhanced, but ductility was disappointingly low. Hardness increases of 5 to 10 times that of conventional grain size metals were observed, but ductilities were reduced to very low values, typically less than 2% elongation in tension for metals with grain sizes < 25 nm. Recently, however, some examples of optimized strength and ductility have been reported in nanostructured metals and alloys, in the authors' laboratory and in the literature. This talk will describe these breakthroughs in the mechanical behavior of nanostructured materials and discuss the possible reasons for these superior properties.

10:15 AM Break

10:30 AM
Syntheis and Properties of Nanostructured Magnetic Materials: *D. Kumar*[1]; [1]North Carolina A & T State University, Dept. of Mechl. Engrg., Greensboro, NC 27411 USA

The key to the successful fabrication of nanostructured magnetic materials with improved properties is the development of smart material systems by material-engineering and understanding the fundamentals of materials science. It is in this context that we have developed a novel smart thin film processing method based upon pulsed laser deposition to process nanocrystalline materials with accurate size and interface control with improved magnetic properties. Using this method, single domain nanocrystalline Fe and Ni particles in 5-10 nm size range were embedded in amorphous and crystalline alumina and TIN matrices. By controlling the size distribution in confined layers, it was possible to tune the magnetic properties from superparamagnetic to ferromagnetic in a controlled way. Such growth processing is driven by an underlying instability, such as misfit strain in heteroepitaxial systems. The uniformity in particle size distribution arises from a competition between the thermodynamic instability and kinetic effects. In this talk we will also demonstrate that in multilayer structures, buried dots can influence the nucleation in subsequent layers, leading to self-organization of a more ordered and uniform array. Another interesting accomplishment of our present work that we will like to share is structural characterization of nanostructured magnetic materials using techniques with probe size of atomic dimension so that an unambiguous information are obtained. Understanding the atomic structures and chemistry of nanoparticles and particle-host matrix interfaces is often critical to structure-property relationship in solid state structures of nanoscale dimension. We have employed scanning transmission electron microscopy with atomic number contrast (STEM-Z) and energy loss spectroscopy (EELS) to understand the atomic structure of Ni and Fe nanoparticles and interface between the nanoparticles and the surrounding matrices. Since Z-contrast imaging and EELS could be performed simultaneously, we were able to make direct correlations between structure and chemistry of the Ni and Fe nanoparticles. It was interesting to learn from EELS measurements at individual grains and interface planes that Ni in alumina matrix does not from an ionic bond at the interface indicating the absence of metal-oxygen bond at interface. The absence of metal-oxygen bond, in turn, suggests the absence of any dead layer on Ni nanoparticles even in an oxide matrix. This talk will also highlight some of the research activities pioneered by Prof. J. Narayan in the area of nanomaterials.

11:00 AM
Strengthening Mechanisms in Nanostructured Layered Materials: *Xinghang Zhang*[1]; Amit Misra[2]; Haiyan Wang[2]; Richard G. Hoagland[2]; [1]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

Sputter-deposited multilayer composites exhibit very high strength as the bilayer period approaches a few nanometers. Strengthening at layer thickness of greater than tens of nm can be described by dislocation pile-up model, whereas hardening of multilayers at smaller layer thickness, from tens of to a few nm, is not well understood. I will briefly review the strengthening mechanisms in multilayer films that are currently believed to be operative at nanometer length scale. Specific examples on strengthening in Cu/austenitic stainless steel multilayers are given in an attempt to explore the role of interface on strengthening at nanometer length scales. Apart from layer interface induced strengthening, we have recently demonstrated twin interface induced strengthening in single-phase 330 stainless steel films. The high strength is attributed to the resistance to single dislocation transmission across twin interfaces, which are parallel to the surface of substrates and separated a few nm spacing. A model is deve loped that accounts for the formation of nanoscale twins during sputter deposition in terms of the twin boundary or stacking fault energy and deposition rate. Molecular dynamics simulations confirm that at nanometer length scales where plasticity is controlled by the motion of single rather than pile-ups of dislocations, twin boundaries are very strong obstacles to slip. These observations provide a new perspective to producing ultra-high strength metals by utilizing growth twins with nanometer-scale spacing.

11:30 AM
Growth Textures of Nanocrystalline Fe-Ni Alloy Foils: *Yong Bum Park*[1]; [1]Sunchon National University, Matls. Sci. & Metallurgl. Engrg., Maegog-dong 315, Sunchon, Chonnam 540-742 Korea

The texture evolution due to grain growth that takes place during annealing was investigated in nanocrystalline Fe-Ni alloys fabricated by using a continuous electroforming method. In the current materials, grain growth occurred in annealing at much lower temperatures than in conventional coarse-grained counterparts. With regard to the macrotextures, the as-deposited textures were of fibre-type characterized by strong {100}and weak {111}components, and the occurrence of grain growth resulted in the strong development of the {111}fibre texture with the minor {100}components. It was clarified using orientation imaging microscopy that abnormal growth of the {111} grains in the early stages of grain growth plays an important role on the texture evolution. The origin of the abnormal grain growth has been discussed in terms of the orientation dependence of energy density.

12:00 PM
Directed Assembly of Metal Nanostructures by Laser-Induced Rapid Spatio-Temporal Surface Modulations: *Chi Zhang*[1]; Wei Zhang[1]; Adam Bauer[1]; Ramki Kalyanaraman[2]; [1]Washington University, Dept. of Physics, St. Louis, MO 63132 USA; [2]Washington University, Ctr. for Matls. Innovation & Dept. of Physics, St. Louis, MO 63130 USA

Thin film nucleation and growth is a self-assembly process where the initial stages naturally consist of nanometer sized clusters. On defect-free isotropic surfaces, these nanoclusters are a result of random walk surface diffusion and binding. The coupled time and length scale in nucleation, as determined by nucleation rate and saturated

cluster density, are determined primarily by growth rate and substrate temperatures. Therefore, appropriate surface temperature modulations could induce morphology changes. In recent work, we presented novel studies showing that the application of a laser-induced repeated and rapid spatio-temporal surface perturbation in-situ with film deposition dynamically directs the assembly of periodic structures. Evidence shows that the assembly is a result of an anisotropic diffusion flux, reevaporation and suppressed nucleation. The experiment consists of physical vapor deposition simultaneous with pulsed laser interference irradiation of a Si(100) surface. Deposition of metal films like Co and Ag or metal oxides like TiOx result in periodic arrays of dot or line-like features whose periodicity and spacing resemble the interference patterns. The size scale of the resulting dot or line-like features can be varied from the nanometer to micron scale by deposition parameters. The rapid spatio-temporal modulation comes from the short fringe spacing (200 to 2000 nm) and short laser pulse width (9 ns). In this work, we present fundamental experimental studies of the influence of these rapid surface modulations that lead to directed assembly of nanostructures. This experimental approach is a simple way to assembly large-area arrays in a single-step parallel process that is likely to lead to a cost effective manufacturing process.

12:15 PM
Self-Assembled Magnetic Nanostructures: Epitaxial Ni on TiN (001) Surface: *Honghui Zhou*[1]; Jagdish Narayan[1]; [1]North Carolina State University, Matls. Sci. & Tech., 2153 Burlington Lab, CB 7916, Raleigh, NC 27695 USA

Self-assembled nickel magnetic nanostructures were grown on epitaxial titanium nitride (001) surface by pulsed laser deposition (PLD) technique. These nanostructures were characterized by conventional and high-resolution transmission electron microscopy (TEM and HRTEM), scanning transmission electron microscopy (STEM) Z-contrast imaging, and X-ray diffraction (XRD) techniques. The results have shown that the growth of nickel on epitaxial titanium nitride (001) surface follows Volmer-Weber growth mode (3D island growth). The predominant orientation of nickel islands observed is Ni (100) // TiN (100), the so-called "cube-on-cube" orientation relation. The islands are faceted with a truncated pyramidal shape and bounded by (111) planes at sides and (100) plane at the top. The two principal axes of the rectangular island bases are close to two orthogonal <011> directions. The island size analysis showed a narrow size distribution, similar to that found in the systems with coherent self-assembled semiconductor islands grown via Stranski-Krastanov mode (2D followed by 3D growth). The islands were not completely randomly distributed; some islands chains were formed along the directions close to <011>. The interaction between neighboring islands through the island-induced strain field is believed to be responsible, to a large degree, for the island size uniformity and the lateral ordering.

Functional Thin Films for Sensors: The Physics and Applications of Functional Thin Films in Sensors

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: Anis Zribi, General Electric Global Research Center, Niskayuna, NY 12309 USA; Jeffrey Fortin, GE Global Research, Niskayuna, NY 12309 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Choong-Un Kim, University of Texas, Materials Science and Engineering, Arlington, TX 76019 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Gerald Schultz, GE Infrastructure, Sensing, Wilmington, MA 01887-4498 USA

Monday AM Room: 3022
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Anis Zribi, GE Global Research, Micro & Nano Struct., Niskayuna, NY 12309 USA; Jeffrey Fortin, GE Global Research, Micro & Nano Struct., Niskayuna, NY 12309 USA; C. U. Kim, University of Texas, Arlington, TX USA

8:30 AM Opening Comments: Chairperson

8:35 AM Invited
Micromachined Scanning Thermal Probes and Probe Arrays for High Speed and High Resolution Diagnostics of Thin Films: *Yogesh B. Gianchandani*[1]; [1]University of Michigan, Ann Arbor, MI 48109 USA

Scanning-probe microscopy is emerging as a technique of increasing importance in high-resolution diagnostics of thin films. Thermal microscopy, in particular, offers a number of uses beyond the obvious ability to map temperature variations, including, for example, the measurement of Tg on sub-micron features, non-destructive mapping of sub-surface defects in thin films, and non-lithographic thermal patterning of materials. This presentation will review the development and application of micromachined thermal probes and multi-probe arrays that use polyimide as the structural material and thin-film metal for the probe tips and temperature sensors. Both thermistor and thermocouple probes have been developed. The high mechanical compliance afforded by the use of polyimide as a structural material facilitates their use without mechanical feedback, giving high-throughput parallelism a real chance.

9:00 AM Invited
Thin Film Capacitive Micromachined Ultrasonic Transducers: *B. T. Khuri-Yakub*[1]; [1]Stanford University, E. L. Ginzton Lab., Rm. 11, Stanford, CA 94305-4088 USA

Capacitive Micromachined Ultrasonic Transducers (CMUTs), using thin film membranes, have been developed for generating and detecting ultrasound.. Silicon micromachining allows the manufacture of capacitors with thin membranes, very thin gaps, and with electric fields of the order of 109 V/m that enable their competitive performance. It is possible to make CMUTs with over 100% fractional bandwidth, with an electromechanical coupling coefficient close to unity, and to make single element, one-dimensional (1D), and two-dimensional (2D) arrays of thousands of elements, as well as annular arrays. CMUTs have been operated in the frequency range of 100 kHz to 50 MHz, and with a dynamic range of 150 dB/V/Hz. This paper will first review the operation and technology for making CMUTs. The performance and technology will be compared and contrasted to the technology for making piezoelectric transducers and arrays. Finally, examples of 2D and 3D imaging will be presented to confirm the performance of these transducers.

9:25 AM
Nano-Tunable Infrared Thin Film Filters and Emitters for Sensing of Trace Gases: *Lawrence Domash*[1]; Brian Kinkade[1]; [1]Aegis Semiconductor Inc., R&D, 78A Olympia Ave., Woburn, MA 01801 USA

The mid IR contains spectral signatures of atmospheric trace gases including carbon dioxide, carbon monoxide, sulphur dioxide, hydrogen cyanide, water vapor, nitric oxide or methane, whose sensing and measurement are important for pollution control, HVAC, and home and automotive safety. Optical spectroscopic detection is known to be the most sensitive and precise of any detection technique but has been considered too expensive for consumer applications. We describe a new family of miniature nano-tunable narrowband infrared filters based on the thermo-optic properties of thin film semiconductors. Originally developed for fiber optic telecommunications networks at 1.5 µm, the technology has now been extended to the 3-5 µm range, leading to very compact tunable filters with passbands on the order of 0.5% of center wavelength and tuning ranges up to 4% of center wavelength. We describe a prototype carbon monoxide sensor testbed based on a 4550-4650 nm tunable filter and show it is capable of detecting 10 ppm of CO. This level of low-noise detection results from the ability of nano-tunable optical elements to enable the application of advanced signal processing methods. Second, we show how nano-tunable thin film filters can be integrated with miniature blackbody sources in TO-5 cans to create a new family of ultra low cost integrated tunable IR emitters (named Firefly(tm)).

9:50 AM
Temperature Dependent Infrared Properties of InP, AlN and Al_2O_3: *N. M. Ravindra*[1]; Anthony T. Fiory[1]; Stephen Rubin[1]; Sudhakar Shet[1]; Vishal R. Mehta[1]; Sreeya Srivatsa[1]; [1]New Jersey Institute of Technology, Newark, NJ 07102 USA

A spectral emissometer operating in the wavelength range of 1 to 20 microns and temperature range of 25 to 1000ºC has been deployed to measure the optical properties of InP, AlN and Al2O3. The measured reflectance, transmittance and emittance of these materials have been compared with available optical properties in the literature. Phenomenological approaches are subsequently employed to deconvolute the measured optical properties to yield wavelength and temperature dependent fundamental optical constants.

10:15 AM Break

10:35 AM
Interaction of Metal Oxide Sensitive Layer With Ambient Atmosphere at Varying Sensor Working Temperature: *Tadeusz*

Pisarkiewicz[1]; Wojciech Maziarz[1]; Krzysztof Hajduk[1]; [1]AGH University of Science and Technology, Dept. of Elect., Al. Mickiewicza 30, Krakow 30-059 Poland

Semiconductor resistive gas sensors are known from their poor selectivity and resistance drift. The exact knowledge of the mechanism of gas/ oxide semiconductor surface interaction enables the optimal selection of gas sensor working regime. The authors have performed the analysis of semiconductor surface reactions with oxygen atmosphere and the influence of chemisorbed oxygen concentration on sample conductance was calculated. Further the influence of reduction and other oxidizing gases on the sensor surface has been envisaged. The model calculations were performed for different temperature operation modes, i.e. sinusoidal, pulse and triangle and the resuls compared with experimental observations. The adequate choice of model parameters results in output signals with good agreement with experimental sensitivity characteristics for thin SnO2 sensitive layers deposited onto multilayer ceramic or micromachined silicon substrates.

11:00 AM

Novel Single Crystal Model for Semiconducting Oxides Thin Film Gas Sensors: *Satyajit Shukla*[1]; Sudipta Seal[1]; [1]University of Central Florida, AMPAC & MMAE Dept., Engrg. 381, 4000 Central Florida Blvd., Orlando, FL 32816 USA

It has been recognized that the gas sensitivity, as well as the response and the recovery time of a semiconducting oxides thin film sensor, depend on number of parameters, such as nanocrystallite size, film thickness, nature and amount of dopants, surface catalyst and foreign oxides, amount of film porosity, and operating temperature. Interestingly, a theoretical model, which would give the effect all these variables on the gas sensitivity has not been yet reported in the literature. In view of this, we propose here a new constitutive equation for the gas sensitivity of nanocrystalline semiconducting oxide sensors based on a single crystal model. The present theoretical model describes the relationship between the gas sensitivity, nanocrystallite size, film thickness, Debye length, operating temperature, surface coverage, and lattice oxygen-ion-vacancy concentration. The validity of the proposed theoretical model is justified by comparing the model predictions with the reported experimental results.

11:25 AM Invited

Water Thin Film Nano-Scale Transistor for Chemical Sensing: *M. Willander*[1]; Z. Chiragwandi[1]; O. Nur[1]; [1]Chalmers University of Technology and University of Göteborg, Physl. Elect. & Photonics, Physics & Engrg. Physics, SE-412 96 Göteborg Sweden

In this invited paper, we present a Si compatible nano-scale water based transistor. The device composed of two nano-electrodes and a third electrode for controlling and manipulating the pH of water The water film thickness can be as thin as 1 µm. By controlling the base electrode, the pH of the water can be manipulated, and a transistor action is observed between two nano-electrodes (emitter and collector). Different device configurations are investigated in view of the effect of the nano-electrodes spacing (emitter-collector spacing) as well as the position and design of the pH electrode (base). The DC characteristics will briefly be discussed in view of the water ions decomposition and it's dependence on the transistor configuration. Time dependant measurement will be presented and discussed both theoretically and correlated to experimental results. Dynamics of ions in water and it's relation to pH will also be discussed. Conversion of chemical reaction(s) to an electrical signals in a well controlled way will be shown and discussed. Finally, and due to the presence of water in almost most chemical reactions as well as biological activities, the importance of the newly presented device will be highlighted.

11:50 AM

Room Temperature Hydrogen Gas Sensitivity of Nanocrystalline Doped-Tin Oxide Sensor Under Ultraviolet Light: *Satyajit Shukla*[1]; Lawrence Ludwig[2]; Rajnikant Agrawal[1]; Julian Duarte[1]; Hyoung Cho[1]; Sudipta Seal[1]; [1]University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. (AMPAC) & Mechl. Matls. Aeros. Engrg. (MMAE) Dept., Engrg. 381, 4000 Central Florida Blvd., Orlando, FL 32816 USA; [2]Kennedy Space Center, KSC-NASA, FL 32899 USA

Nanocrystalline indium oxide doped-tin oxide thin film is sol-gel dip-coated on microelectromechanical-systems (MEMS) device. Effect of ultraviolet (UV) radiation on room temperature hydrogen sensitivity of the present sensor is systematically studied. Freshly prepared sensor exhibits higher hydrogen sensitivity under UV-exposure than that without UV-exposure, in agreement with earlier reports. However, sensor behavior is observed to reverse after initial UV-exposure, which is attributed to the burning of carbonaceous impurities from the sensor-surface. It is observed that subsequent exposure to UV-radiation results in deterioration of hydrogen sensitivity of the present sensor. Very high hydrogen sensitivity as high as 110000 is observed, for 900 ppm hydrogen in dark condition. However, in the presence of UV-radiation, hydrogen sensitivity is reduced to 200. The drastic reduction in hydrogen sensitivity under UV-exposure is explained on the basis of the constitutive equation for the gas sensitivity of nanocrystalline semiconducting oxides thin film sensor proposed by present authors.

General Abstract Session: Electronic Materials

Sponsored by: TMS

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Monday AM Room: 2011
February 14, 2005 Location: Moscone West Convention Center

Session Chair: James C. Earthman, University of California, Dept. of Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA

8:30 AM

Wafer Integration of GaN/GaN and Al0.25Ga0.75N/GaN Through Wafer-Bonding Technology: *Frank F. Shi*[1]; [1]University of Illinois, Micro & Nanotech. Lab., 208 N. Wright St., Dept. of Electl. Engrg., Urbana, IL 61801 USA

Group III-nitride-based wide-bandgap semiconductors, such as GaN, AlN, and their ternary alloy AlxGa1-xN have emerged as the leading materials for a variety of key applications in advanced integrated optoelectronics. One of the major challenges in group-III nitride semiconductors is the heterointegration of free-standing GaN-based devices with dissimilar substrates. Wafer bonding, as one of the encouraging material and device integrating techniques, has become an enable approach to fabricate hybrid-integrated heterostructures with multiple bandgaps. In this study, the wafer integration of GaN/GaN and Al0.25Ga0.75N/GaN semiconductors through direct bonding technology was reported. The surface morphologies of pre-bonding and debonded wafers were characterized by Atomic Force Microscope (AFM). Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) were employed to study the epitaxial structures and the bonded interface micromorphologies. The interface adhesion was estimated based upon the measured interface fracture energy Go from double cantilever beam (DCB) technique, and the interface fracture energies of several different wafer-bonded III-V semiconductors were also compared. By comparing the atomic chemical bond energy Eo with the interface fracture energy Go, the bondability of a few major III-V semiconductors were analyzed. The potential effects from surface and interface microstructures on the wafer bondability and interface adhesion were discussed. It has been found that the relatively strong wafer bonding of group III-nitride was typically realized at 1000°C. From AFM scanning, it was found that the debonded wafer surfaces generally have much higher surface roughness than the pre-bonding wafers. The wafer bondability and bonding process conditions depend heavily on the surface topography of the pre-bonding wafer surfaces. Lower wafer surface roughness is expected to provide better bondability and demand less stringent bonding conditions. The Ga droplets due to GaN dissociation and Ga extraction were observed on the debonded GaN wafer surface. Interface fracture energies Go of bonded group III- nitrides are higher than those of other bonded III-V semiconductor wafers due to the nature of their stronger chemical bonds and the closer-packed plane at (0001) direction. It was also observed that the measured interface fracture energies of water-bonded semiconductors are generally about 40% of the overall atomic chemical bond energy on the same unit are a, which mainly resulted from the large density of dangling bonds and other imperfections at the wafer-bonded interfaces. High resolution SEM and TEM images on the wafer-bonded interfaces of GaN/GaN and Al0.25Ga0.75N/GaN show an uniformly distrusted amorphous thin layer and the previously observed nanoinclusions in other wafer-bonded semiconductors were not found due to the fact that it was thermally difficult to rearrange group III-nitride atoms and to recrystallize the bonded interface between the two group III-nitride wafers.

9:00 AM
Hot-Wall CVD Epi-Growth of 4H-SiC Using PVT Buffer Layer: *Ying Gao*[1]; [1]Bandgap Technologies, Inc, 1428 Taylor St., Columbia, SC 29201 USA

Physical vapor transport (PVT) has been successfully used for SiC bulk growth. Recently, this method has been proposed for the growth of thick epilayer, however, due to the extremely high temperature required for implementing this process, it is very hard to control the doping level during epi-growth. Therefore, chemical vapor deposition (CVD) is still the main approach to achieve high quality epitaxial film in SiC for device application. But good quality CVD film requires very high quality substrate and is very sensitive to surface preparation. The defects existing in the substrate, such as micro-pipes, dislocations, scratch marks will propagate to the epilayer during growth. Moreover, a great number of new defects will be generated in the initial epi-growth due to the existence of the huge thermal stress formed by different growing temperatures for substrate (more than 2300°C) and epilayer (less then 1700°C). In this work, we attempt to make use of PVT to grow a thin buffer layer with high n-type doping, and then grow regular epilayer by CVD on top of it. The PVT buffer layer was grown at 2050°C in the ambient of Ar, which will significantly reduce the thermal stress from substrate and close most of the micro-pipes. Atomic force microscopy (AFM) images showed a bunch of scratch marks appearing on the original commercial wafer surface. After PVT buffer growth, the surface became perfect with no scratch marks owing to the initial etching process of PVT growth and a true micro-pipe free surface was obtained. The RMS roughness was reduced from more than 2 nm to 0.6 nm. In the second step, homoepitaxial growth was carried out in the home-built vertical hot-wall CVD system. SiH_4-C_3H_8-H_2 system was used in CVD growth. The chamber pressure was maintained at 300 Torr. A 10 μm thick epilayer was grown at a growth rate of 15 μm/hr. The epilayer was examined by high resolution optical microscope (HROM), AFM and scanning electron microscopy (SEM). Excellent surface morphology was obtained without features. Furthermore, with the aid of KOH etching and back-reflection X-ray topography, the substrate, the PVT buffer layer and the CVD epilayer will be investigated to determine the variation in defect density, particularly threading and screw dislocations. Further improvement of the epitaxial layer quality will be discussed and suggested.

9:30 AM
Effects of Holmium Oxides on Electrical Properties of BaTiO3 Sputtered Thin Films: *Z. H. Wu*[1]; J. P. Chu[1]; S. F. Wang[2]; T. N. Lin[1]; C. H. Lin[1]; [1]National Taiwan Ocean University, Inst. of Matls. Engrg., 2, Pei-Ning Rd., Keelung 20224 China; [2]National Taipei University of Technology, Dept. of Matls. & Minerals Resources Engrg., Taipei 20224 China

The holmium-doped $BaTiO_3$ thin films deposited on Pt/Ti/SiO_2/Si substrate by r.f magnetron sputtering method were investigated. The effects of post- annealing temperature and dopant concentration were studied. The compositions of thin films measured consist of BT2 (Ba/Ti=0.8543), BT (Ba/Ti=0.9681), BT-0.05Ho, BT-1.1Ho, BT-1.8Ho and BT-2.9Ho where Ho-content was in at%. The dielectric constant of all compositions thin films increased with increasing annealing temperature, presumably due to better crystallinity and large grain sizes. The BT2 film with 200nm thickness annealed at 700°C showed the high dielectric constant of ~287 measured at 100 KHz. In addition, a significant role of the Ho on reduction of leakage current density is observed for the 700°C annealed samples. The BT-2.9Ho film showed the low leakage current density of 1.27×10^{-8} A/cm^2 at an electric field of 100KV/cm.

10:00 AM Break

10:20 AM
Mechanosynthesis Technique to Improve Superconductor Properties of MgB2: Mechanical Alloying-Ball Milling: *Ely X. Colon*[1]; [1]University of Wisconsin and University of Puerto Rico, Applied Superconductivity Ctr., Matl. Sci. & Engrg., PO Box 779, Sabana Seca, P.R. 00952 USA

The present article explains the process used in the development of the mechanosynthesis of superconductor, MgB2 and MgB2 + MB2 using diborides such as ZrB2, AlB2 and TiB2, in order to improve its superconducting properties. These milling devices include the vibrational mills. X-ray diffraction (XRD) and Superconducting Quantum Interface Device measurements, (SQUID) were used in order to identify the phases presented and the characterization of the final product of the samples after conducting mechanical alloying process. The SQUID measurements showed a decrease of the Tc in samples of 10at% TiB2 with 20K to 5K transition. 10at% AlB2 shows a wide transition and lower Tc in comparison with pure MgB2 bulk with a transition Tc from 15K to 5K. The XRD patterns show amorphization behavior for all of the compounds and many impurities and secondary phases that could be the cause for the decrement of Tc.

10:50 AM
True Image Light Amplifier: *Phillip Kornreich*[1]; *Akshob V. Bangle*[1]; [1]Syracuse University, EECS, 121 Link Hall of Engrg., Syracuse, NY 13244 USA

A feeble light with appropriate amplification improves visibility in dark places. This has led to considerable research and development of Night Vision Techniques. Here at Syracuse University, it has been successfully demonstrated that the Semiconductor Cylindrical Fiber Light Amplifiers (SCFLA) provides net gain for optical communication wavelength, which are in the infrared region. For light amplification in the visible spectrum, we have selected a semiconductor compound that would amplify light in this region. We found that the semiconductor Cadmium Germanium Phosphide ($CdGeP_2$) can be used for light amplification since it covers almost the whole visible spectral band from a wavelength of 414nm to about 700nm. We have calculated the light amplification for various pump light induced charge carrier densities. An UV pump laser is used in this application. A graph of the gain produced to the wavelength is shown in the figure. As seen, when the pump induced charge carrier surface density in the semiconductor film of 1.6 X 10^{17} per m2, the gain region covers almost the whole visible spectrum. This semiconductor fiber can amplify light over the whole visible spectrum using only one pixel per image point unlike the usually display devices that uses three different colors to present as an image to a human eye, there by requiring three pixels for each image point. Thus one can build light amplifying plates that are about 4 to 10 mm thick and have sufficient gain.

11:20 AM
A Cellular Automata Model for Dendritic Crystal Growth: *Liang Yu*[1]; Yuanchi Dong[1]; Liaosha Li[1]; [1]Anhui University of Technology, Anhui Provincial Key Lab. of Metallurgl. Engrg. & Resources Recycling, Hudong Rd., Ma anshan, Anhui 243002 China

A cellular automata model was proposed as a simple simulation model for crystal growth patterns in a diffusion field. Parabola-like and dendritic patterns were obtained in two-dimensional cellular automata. The crystal grain growth and its pattern were controlled by parameters of the diffusion field as well as the surface tension effect introduced through parameter in the surface process. As a simple model, the simulation could be easily performed and a variety of growth patterns were possible to be generated by changing the parameters in the model.

11:50 AM
A Comparative Study of Ferroelectric Lanthanide Doped Bismuth Titanate Films Prepared by Sol-Gel and Pulsed-Laser-Ablation: *Ashish Garg*[1]; X. Hu[1]; Z. H. Barber[1]; [1]Indian Institute of Technology, Dept. of Matls. & Metallurgl. Engrg., Kanpur 208016 India

Thin films of ferroelectric Bi-layered Aurvillius perovskite oxides have been an active area of investigation due to their potential applications in non-volatile ferroelectric random access memories (FRAM) and ferroelectric field effect transistor (FET) devices. Among various materials, SrBi2Ti2O9 (SBT) and La-doped bismuth titanate (BLT) have been intensively investigated due to their excellent fatigue resistance on Pt electrodes. In this paper, we present the results of lanthanide–doped (Sm and Nd-doped) bismuth titanate ferroelectric (BLnT) thin films deposited on platinized Si substrates. To study the influence of type process of the film properties, the films were grown by pulsed laser deposition (PLD) and chemical solution deposition (CSD) and structural and ferroelectric properties of the films were evaluated in detail. The structural characterization was done using X-ray diffraction (XRD), Raman spectroscopy and atomic force microscopy (AFM). Detailed ferroelectric measurements were performed to study hysteresis behavior (P-E loops), dielectric constant, leakage behavior (J-V plots), and polarization fatigue. The films deposited by both processes were polycrystalline but the film morphology was dependent on the type of process. Pulsed-laser-ablated Sm-doped Bi4Ti3O12 films on Pt/Si substrates show a remanent polarization (2Pr) as high as ~45 C/cm2. In case of chemical-solution-derived Nd- and Sm-doped Bi4Ti3O12 films grown on Pt/Si substrates, the crystallinity and ferroelectric properties were strongly dependent upon the annealing temperature. All films demonstrate fatigue-free behavior up to 109 read/write switching cycles. Conduction mechanism of the films is found to exhibit a dependence on the type of deposition process.

General Abstract Session: Temperature Treatments and Casting

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Monday AM Room: 2012
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Alan W. Cramb, Carnegie Mellon University, Pittsburgh, PA 15213 USA

8:30 AM
Investigation of Recrystallisation Resistance in Al-Hf-(Sc)-(Zr) Alloys: *Haakon Hallem*[1]; [1]NTNU, Dept. of Matls. Tech., Alfred Getzv 2b, Trondheim 7491 Norway

Highly stable microstructures are required to cope with the demand for extruded aluminium alloys tolerant of exposures to temperatures of up to 600°C. In order to achieve this Hf, Sc and Zr were added to Al alloys in different concentrations and combinations. These elements form dispersoids of the type Al3X (X=Hf,Sc,Zr) upon annealing. Cold rolling was applied to the alloys in order to investigate the recrystallisation resistance after deformation. The Al-Sc-Zr alloy has been found to stay unrecrystallised with a heat treatment of 1 hr at 600°C even after 80% reduction. One of the Al-Hf-Sc-Zr alloys did also resist recrystallisation, except for a thin surface layer. Despite the recrystallisation resistance the heat treatments applied cause a considerable loss in strength of all the alloys. But still the unrecrystallised alloys have a higher hardness.

8:55 AM
The Effect of Tempering on Low Cycle Fatigue Behavior of Polycrystalline Al2024: *Aezeden Omar Mohamed*[1]; [1]University of Manitoba, Mechl. & Mfg. Engrg., Mechl. Engrg. Dept., Rm. 356, Winnipeg, Manitoba R3T 2N2 Canada

A low cycle fatigue study on Al2024 polycrystalline alloy was conducted under symmetric tension-compression at room temperature, using a servo-hydraulic testing machine, to investigate the effect of tempering on cyclic deformation. The tests were conducted at constant frequency for O-temper condition and a constant strain rate for T6-temper condition. The fatigue response of the alloy was evaluated macroscopically in terms of cyclic stress strain response and microscopically in terms of appearance of cyclic slip bands. It was found that the cyclic stress strain response of O-tempered alloy exhibited an increase in saturation stress with plastic strain whereas the cyclic stress strain response of T6- tempered alloy exhibited a definite plateau region where the saturation stress remained constant with plastic strain. The T6-temper alloy showed the presence of persistent slip bands (PSBs), which were extending across the grain at 45° orientations from the grain boundary. No (PSBs) were observed in the microstructure of the O-temper alloy; however, precipitate free zone (PFZ) was observed adjacent to the grain boundaries. Needle-like precipitates were also observed within the grains with two different orientations and the angle between them was 65?a in the O-tempered alloy.

9:20 AM
A Differential Scanning Calorimetry Study on Crystallization of Sputtered Ba1-x Sr x TiO3 Thin Films: *Tai Nan Lin*[1]; Jinn P. Chu[1]; Sea-Fue Wang[2]; [1]National Taiwan Ocean University, Inst. of Matls. Engrg., 2 Pei Ning Rd., Keelung 202 Taiwan; [2]National Taipei University of Technology, Dept. of Matls. & Mineral Resources Engrg., Taipei 202 Taiwan

Crystallization behavior of sputtered BST, $(Ba_{0.3}Sr_{0.7})TiO_3$, thin films has been characterized by means of differential scanning calorimetry(DSC) and transmission electron microscopy(TEM). The crystallization of as-deposited amorphous structure to the equilibrium crystalline structure is confirmed as an irreversible, exothermic, first order transition. DSC result confirming the metastable nature of sputtered films is in agreement with that of a conventional, ex-situ X ray diffraction result. At a heating rate of 20°C/min, the crystallization peak temperature of BST film is found to be 697.9°C and to increase with increasing heating rate. Activation energy for crystallization is determined, by Kissinger's method, to be 447.7 kJ/mol. TEM results reveal the presence of amorphous and crystalline phases at temperatures below 750°C, indicating that the substrate temperature below 750°C is not sufficiently high for the full crystallization of the film. Diel ectric constant value increases with increasing substrate temperature, reaching the highest value of ~160 at 750°C.

9:45 AM
Effect Appreciation of Jominy End Quench on the Microstructure and Mechanical Properties of A356 Cast Aluminum Alloys: *Qingcai Liu*[1]; Yungui Du[1]; Xin Huang[1]; [1]Chongqing University, 174 Shapinba St., Chongqing 400044 China

The review to date examines the use of the Jominy End Quench specimen and its application to the examination of the effects of quench rate and subsequent processing on A356 aluminum alloys. Heat treatment for aluminum alloys broadly covers the following: homogenization to eliminate coring developed during solidification; annealing for recovery, recrystallization and grain size control; solutionizing of precipitation strengthening solutes in solid solution; quenching to supersaturate the solid solution for subsequent aging; and aging to precipitate the strengthening phases. The review collected and compiled the current knowledge related to the relationship between the heat treatment process and the microstructure and mechanical properties of A356 aluminum alloys. It was extended to the alloying elements and heat treatment effects on precipitate and microstructure. The experimental technique of the Jominy End Quench was concerned in this work.

10:10 AM Break

10:30 AM
Substitution of Malleable Cast Iron with Ferritic Ductile Cast Iron at Low Temperatures: *Mehran Tadayonsaidi*[1]; Nima Baghersaiee[2]; Naser Varahram[3]; [1]AZAD University, PO Box 31535-3453, Karaj Iran; [2]Engineering Research Institute, Control Dept., Ministry of Jihad Agriculture, Tehran Iran; [3]Sharif University of Technology, Dept. Met. Sci. & Engrg., Tehran Iran

In the past 40 years the use of ductile cast iron has grown rapidly, mainly through conversions from gray and malleable iron casting. In many cases malleable cast iron replaced with ductile iron but it has a higher mechanical property such as elongation and toughness at low temperature. In this study the properties of ferritic ductile cast iron with different percent of silicon and the effect of heat treatment annealing cycles and microstructure on the mechanical properties, as compared to malleable ductile iron were investigated. The results show that Ferritic ductile iron has higher mechanical properties such as yield stress and hardness, which is rised by increasing the amount of silicon contents, also when designing for low temperature application the amount of silicon content should be less than 2% due to the similar impact strength to malleable cast iron at low temperature. Mechanical tests and SEM and optical microscopic investigate the accuracy of these results. The result of this research was so benefit for different industry applications mainly in railway parts which cause to lower costs with the same property comparing to another structure.

10:55 AM
The Effect of Mold Flux on Radiative Heat Transfer: *Wanlin Wang*[1]; Alan W. Cramb[1]; [1]Carnegie Mellon University, 5000 Forbes, Pittsburgh, PA 15213 USA

As an important factor in the moderation of heat transfer in continuous casting mold, the mold flux has been researched widely for varied purposes. However, the study of effect of mold flux on radiative heat transfer has not been conducted much. Through using an infrared radiation emitter, which is developed at Carnegie Mellon University to allow about 1 MW/m2 heat flux to produce, the radiative heat flux is applied to copper mold to simulate the heat transfer phenomena in continuous casting. The effect of adding a thin slag disc on top of copper mold on radiative heat transfer has been analyzed. It has been found that the mold flux plays a very different role by enhancing radiative heat transfer instead of retarding conductive heat transfer. The specific effect of crystalline and glassy parts of the mold flux on radiative heat transfer, and the influence of their properties on heat transfer rate will be discussed in this paper.

11:20 AM
Castability and Microstructure Characterization of Reduced and Lead Free Brasses for Sanitary Applications: *C. Vilarinho*[1]; D. Soares[1]; J. J. Barbosa[1]; F. Castro[1]; [1]University of Minho, Mechl. Engrg., Campus de Azurem, Guimarães 4800 Portugal

As a result of environmental concerns, brass products, namely those which are in direct contact with drinking water, must be manufactured from reduced or lead free alloys. For that purpose a set of brasses, that could substitute the traditional ones, has been developed and cast and

its chemical composition determined by XRF spectrometry. Castability and solidification shrinkage of the alloys were studied using appropriate devices and melting and transformation temperatures were determined by Differential Scanning Calorimetry (DSC). Each alloy was observed by Scanning Electron Microscopy (SEM) to identify the phases present and its chemical analysis was determined by Electron Probe Microanalysis (EPMA). A comparison between the results concerning the developed alloys and the traditional leaded brasses is established.

Globalization of Materials R&D

Sponsored by: National Materials Advisory Board, Public & Governmental Affairs Committee
Program Organizers: Toni G. Marechaux, National Research Council, National Materials Advisory Board, Washington, DC 20418 USA; Warren H. Hunt Jr., Aluminum Consultants Group Inc., Murrysville, PA 15668-2002 USA

Monday AM Room: 2009
February 14, 2005 Location: Moscone West Convention Center

Program Moderator: Warren H. Hunt Jr., Aluminum Consultants Group Inc., Murrysville, PA 15668-2002 USA

Join us for a variety of presentations and a roundtable discussion on the globalization of materials research and development. Topics and participants include:

8:30 AM
National Policy: *Toni Marechaux*[1]; [1]The National Academies, 2101 Constitution Ave. NW, Washington, DC 20418 USA

8:50 AM
Corporate Research Management: *Dianne Chong*[1]; [1]The Boeing Company, PO Box 516, St. Louis, MO 63166-0516 USA

9:10 AM
Metals Industry: *Diran Apelian*[1]; [1]Worcester Polytechnic Institute, Metal Procg. Inst., 100 Institute Rd., Worcester, MA 01609-2280 USA

9:30 AM
Electronics Industry: *Darrel Frear*[1]; [1]Freescale Semiconductor, 2100 E. Elliot Rd., Tempe, AZ 85284-1801 USA

9:50 AM Break

10:00 AM
The Role of the Materials Societies: *Brajendra Mishra*[1]; [1]Colorado School of Mines, Kroll Inst. for Extractive Metals, Dept. of Metallurgl. & Matls. Engrg., 1500 Illinois St., Golden, CO 80401-1887 USA

10:20 AM
University Perspective: *Henry Rack*[1]; [1]Clemson University, Sch. of Matls. Sci. & Engrg., 208 Rhodes Hall, Clemson, SC 29634-0921 USA

10:40 AM
Government Perspective: *Sylvia Johnson*[1]; [1]NASA Ames Research Center, USA

11:00 AM
Roundtable Discussion

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Monday AM Room: 3008
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Thaddeus B. Massalski, Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15213 USA; Kenneth F. Kelton, Washington University, Dept. of Physics, St. Louis, MO 63130 USA

8:30 AM Opening Remarks

8:40 AM Keynote
Hume-Rothery Rule in Structurally Complex Alloy Phases: *Uichiro Mizutani*[1]; [1]Nagoya University, Dept. of Crystalline Matls. Sci., Sch. of Engrg., Furo-cho, Chikusa-ku, Nagoya 464-01 Japan

Special emphasis will be put on our understanding of why nature is able to stabilize structurally complex alloy phases like quasicrystals and gamma-brasses, the latter of which has been known as one of the typical Hume-Rothery phases for many years. Most of these structures have been synthesized by using the Hume-Rothery rule that relates fundamental aspects of electronic structure to stability. The Hume-Rothery rule related electronic structure studies on structurally complex alloy phases will be reviewed, followed by its new interpretation based on the first-principle band calculations, which hopefully goes beyond the first naive free electron picture put forward by Mott and Jones in 1936.

9:30 AM Invited
Spherical Periodicity, A General Feature of Matter at its Early Stages of Formation: *Peter Häussler*[1]; [1]Chemnitz University of Technology, Inst. of Physics, Reichenhainer Str.70, Chemnitz 09107 Germany

Structure formation, the transition from the completely disordered state to the long-ranging order of a crystal, is even for simple inorganic systems under many aspects still a mystery. Both, liquid and amorphous systems, are right on the way to form structural elements, both are precursors of the crystalline state. Understanding their fundamental structure-forming processes may once help a modern materials design. For very early stages of structure formation, within the liquid and amorphous state, local spherical periodicity around any adatom was observed due to the minimization of the total energy along a self organizing spherical-periodic resonance. The resonance arises between two macroscopic subsystems, namely the valence electrons in total as the one and the forming static structure as the other one. It is based on characteristic momenta of both subsystems and hence is a global effect. As a consequence, a pseudogap gets formed at the Fermi energy (Hume-Rothery-, Peierls-like) stabilizing the system, as well as affecting any electronic transport property. To find the optimum the forming static structure as well as the electronic system both may mutually adjust. The more degrees of freedom exist, the more optimal becomes the resonance. We are able to describe the static structure of the glassy and the liquid state of metals, ionic glasses, amorphous quasicrystals, as well as glassy semiconductors on the same roots. Due to the resonance gaps at the Fermi energy, electronic transport will strongly depend on the strength of the resonance. We propose the spherical resonance model as an autonomous missing link between the microscopic description of atoms/molecules, described by Schrödinger's equation, and the crystals where global concepts as planar resonances exist and Bloch's theorem applies. We report on different scenarios how the total system is able to optimize the resonance, give an overview on the structural features of many complex phases as liquid and amorphous systems of different type and show systematics in the structure of liquid and amorphous elements along the periodic table. Systematics in the electronic transport properties and their evolvement during the structure formation are also briefly reported.

10:00 AM Break

10:20 AM Invited
Complex Metallic Alloys: A New Frontier in Condensed Matter Physics: *Jean-Marie Dubois*[1]; [1]CNRS, LSG2M, Ecole des Mines, Parc de Saurupt, Nancy 54042 France

Aside quasicrystals, which were a focus of interest for the last two decades and obliged us to forge new tools able to solve complex crystallographic structures, many crystals based on metals present giant unit cells and intriguing properties. Whereas a few tens are known in binary compounds (e.g. Al3Mg2 with 1148 atoms per unit cell), these so-called "Complex Metallic Alloys" are supposed to be hidden in the many, as yet essentially unexplored, ternary, quaternary, etc. phase diagrams of metallic elements alloyed with metalloids and/or rare earths. The new spring board that complex intermetallics offer to metal physicists and material scientists is to understand in great detail the relationships between structural complexity and lattice and electronic excitations on the one hand and the role and nature of chemical bonding in the formation and stability of such compounds. In this respect, the seminal work of Prof. U. Mizutani on the Hume-Rothery mechanism in CMAs and quasicrystals is of key importance and wil l be illustrated by results of the author and collaborators. The talk will also put emphasis on two examples of properties, namely surface energy and reduced friction in vacuum, with a view at using selected complex metallic alloys as model systems for a deeper understanding of properties that are of technological relevance.

10:50 AM Invited
Electron Concentration Variation and Local Environment Effects in Complex Materials: *Igor A. Abrikosov*[1]; [1]Linköping University, Dept. of Physics & Measurement Tech., Campus Valla, Fysikhuset, F308, Linköping SE-58183 Sweden

The ability of the first-principles theory to describe the structural transitions in complex materials upon the change of the electron concentration will be illustrated by several examples, including the study of electronically induced phase transitions in ternary transition metal distannide systems, electronic topological transitions in Al-Zn alloys, and phase stability of Li(Mn-Co)O2 oxides. We will also reiterate that simple arguments that consider a filling of the rigid band by electrons upon alloying often fail to predict electronic and structural properties of materials. Particular attention will be paid to the so-called local environment effects, i.e. a dependence of the electronic properties of each atom in a system on its local chemical environment. We will discuss recent experimental studies where the effect is detected, and compare it with our theoretical results. Considering complex magnetic structures of Fe-Ni alloy as an example, we will show how the interplay between a change in valence electro n concentration and local environment effects can lead to a formation of a glass-like state.

11:20 AM Invited
From Electronic Structure to Phase Equilibria Calculations: *Tetsuo Mohri*[1]; Ying Chen[2]; Munekazu Ohno[3]; [1]Hokkaido University, Grad. Sch. of Engr., Div. of Matls. Sci. & Engrg., Kita-13 Nishi-8, Kita-ku, Sapporo 060-8628 Japan; [2]University of Tokyo, Dept. of Quantum Engrg. & Sys. Sci., Hongo 7-3-1, Bunkyo-ku, Tokyo 113-8655 Japan; [3]Technical University, Clausthal, Inst. fuer Metall., Robert Koch Str. 42, Clausthal-Zellerfeld D-38678 Germany

By combining electronic structure calculations with statistical mechanics calculations, phase stability and phase equilibria of a given alloy system can be predicted from the first-principles. The authors have been attempting first-principles calculations on a phase diagram for a series of Fe-based alloy systems and reproduced the transition temperature with high accuracy. Moreover, by combining with Phase Field Method, it is demonstrated that even ordering dynamics is well reproduced from the first-principles. In those calculations, Cluster Variation Method(CVM) plays a central role in evaluating a configurational free energy. The applicability of the conventional CVM, however, is limited to a system in which global lattice symmetry is preserved at each unit cell. Hence, the structural stability of a complex system such as topologically disordered systems has not been well studied by the conventional CVM. The recent progress of Continuous Cluster Variation Method (CCVM) opens up a new possibility of exploring the topological relaxations. The recent progress of the CCVM is reviewed.

Industrial Energy Reduction: Materials Opportunity Analyses

Sponsored by: TMS
Program Organizer: Sara Dillich, EERE-ITP, US Department of Energy, Washington, DC 20585 USA

Monday AM Room: 2000
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Sara Dillich, EERE-ITP, US Dept. of Energy, Washington, DC 20585 USA

8:30 AM
Refractories for Industrial Processing: Energy Reduction Opportunities: James G. Hemrick[2]; *H. Wayne Hayden*[1]; Peter Angelini[2]; Robert E. Moore (Deceased)[3]; William L. Headrick[3]; [1]MMPaCT, Inc., 123 Lake Hills Dr., Oak Ridge, TN 37830-4231 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6069, Oak Ridge, TN 37831-6065 USA; [3]R. E. Moore and Associates, PO Box 314, Rolla, MO 67402 USA

Refractories are used in most manufacturing industries as insulation and/or containment vessel linings for high temperature and corrosive environments. In addition, refractory components often have load bearing or heat transfer functions which contribute further to their performance requirements. Reduction of energy losses and increased productivity in processing could be realized by development of higher strength refractories capable of operating at higher temperatures, development of refractories with lower thermal conductivity to reduce wall losses at higher service temperatures, improved control of thermal expansion of current refractories, and development of refractories with greater resistance to degradation. Fifteen industrial application types were analysed. Four types, boilers and reaction systems, gasifiers, reverbatory furnaces, and kilns and calciners, have been identified as areas where significant energy savings (trillions of BTUs) could be realized as a result of refractory improvement.

9:00 AM
Materials for Separations Technologies: Energy and Emission Reduction Opportunities: *William T. Choate*[1]; Robert Jubin[2]; Sharon Robinson[2]; [1]BCS, Incorporated, 5550 Sterrett Pl., Ste. #306, Columbia, MD 21044 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN USA

Separation processes are the most energy intensive industrial operations. They consume over 5,000 trillion Btu/year, more than a third of the energy consumed within manufacturing facilities. Mass transfer operations (i.e., distillation, evaporation and drying) account for 80% of the separations energy consumed. These operations are inherently high-energy consumers, utilizing "heat of vaporization" as the separating phenomena. Materials used as separation agents such as adsorbents, absorbents, ion-exchange resins, ionic liquids and membranes perform low-energy intensive "heats of solution" separations. The greatest potential for reducing industrial separations energy is the development of new and enhanced materials that will enable a shift from high-energy to low-energy technologies. Materials with improved selectivity, capacity, stability, morphology, and ability to regenerate will move these low-temperature technologies from niche applications into broader use. This study examined the largest industrial separation operations and identified over 220 trillion Btu/year of potential savings that would result from enhanced materials.

9:30 AM
Impacts of Condition Assessment on Energy Use: Selected Applications in Chemical Processing and Petroleum Refining: *Emory A. Ford*[1]; Joan Pellegrino[2]; [1]Materials Technology Institute, 1215 Fern Ridge Pkwy., St. Louis, MO 63141-6078 USA; [2]Energetics, 7164 Columbia Gateway Dr., Columbia, MD 21046 USA

Condition assessment refers to inspection of equipment for conditions such as corrosion, metal wear, and cracking. Effective methods for condition assessment of process equipment are critical to the efficient, safe operation of plants. The processes in these industries are operated at high temperatures and pressures, and in some cases in corrosive environments. Over 5 quadrillion Btu are consumed yearly in chemicals and petroleum refining for process heating and cooling. The potential for reducing energy use in two energy-intensive process applications through improved condition assessment technology is examined. The first is the ethylene cracking furnace and the second is preheat trains, used to preheat crude oil before downstream refining into products.

10:00 AM Break

10:20 AM
Energy Impacts of Corrosion: Industrial Case Studies: *Ross Brindle*[1]; [1]Energetics, Incorporated, 7164 Gateway Dr., Columbia, MD 21046 USA

Corrosion has been estimated to cost the U.S. economy over $276 billion each year. In the industrial sector alone, corrosion costs manufacturers an estimated $84 billion in the form of increased capital and operating costs, lost productivity, and lower efficiencies. Materials corrosion also leads to significant energy losses in industrial processes due to increased frequency of required shutdowns and startups, missed opportunities to capture waste heat, efficiency limitations imposed by materials performance at higher temperatures, and efficiency degradation over time. An analysis of several industrial applications indicates that the magnitude of potential energy savings associated with developing corrosion-resistant materials can be significant. Three industrial case studies have been analyzed: stress-assisted corrosion of Kraft recovery boiler tubes in pulp and paper operations, the use of recuperators in aluminum melting furnaces, and carburization of tubes in ethylene pyrolysis furnaces.

10:50 AM
Industrial Waste Heat Recovery and Reuse: *Vilayanur V. Viswanathan*[1]; James D. Holbery[1]; Richard W. Davies[1]; [1]Pacific Northwest National Laboratory, Energy Sci. & Tech. Dept., 902 Battelle Blvd., PO Box 999, MS K2-44, Richland, WA 99352 USA

This work discusses and defines the opportunities to recover energy from industrial waste heat and emissions. To evaluate these opportunities, industrial waste heat and emissions data was obtained from public, technical and business literature and publications; and analysis was performed to identify major sources of waste heat, and emissions with fuel value that might be converted into usable energy. The study was focused on the highest energy consuming industries in the U.S. - including petroleum, chemicals, forest products and primary metals industries. The amount and quality of energy available in industrial waste heat for potential recovery was characterized and quantified. The electrochemical potential and residual fuel value of industrial emissions were evaluated, and the associated energy savings, emissions reductions, and economic benefits were assessed. Major technologies, both commercial and emerging, that have high potential to recover energy from industrial waste heat and emissions will be discussed.

11:20 AM
Energy Reductions Estimated for Microchannel Technology: *Landis D. Kannberg*[1]; [1]Pacific Northwest National Laboratory, Energy Scis. Dept., PO Box 999, K9-09, Richland, WA 99354 USA

Energy reduction is one of the many benefits from utilization of microchannel architectures in systems involving heat and mass transport. Such architectures can be applied to components, systems and devices involving chemical and fuel processing, heat exchange and recuperation, and cooling and heating processes The benefits come from both fundamental improvement of process performance, as well as displacement of conventional technology with substantially different technology (enabled through process intensification achievable in microchannel architectures) that is more systemically efficient. Projected savings from such benefits, through reasonable deployment, will be provided. Finally, information on foreign investment in development of one principal area (chemical processing) will be briefly explored as an indicator of worldwide consideration of microtechnology development.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Interfacial Reactions and Phase Stability in Lead Free Solder Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Monday AM Room: 3014
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504 USA; Martin W. Weiser, Honeywell Electronic Materials, Spokane Valley, WA 99216 USA

8:30 AM Invited
Solder Thermal Interface Materials in Microprocessor Cooling: *Martin W. Weiser*[1]; Brian D. Ruchert[1]; Colin F. Edie[1]; [1]Honeywell Electronic Materials, 15128 E. Euclid Ave., Spokane Valley, WA 99216 USA

As the computational power of microprocessors grows amount of heat generated grows so that current devices generate 100W or more. This has severely strained the ability to remove the heat in a cost effective maner. One part of the solution to this problem is to reduce the thermal resistance at the interfaces between the die and the heat spreader since newer chip designs often concentrate a large portion of the heat in a small number of hot spots on the chip. Solders have traditionally been used to solve this problem in power die, but the die used in microprocessors are not as mechanically and thermally stable as power die. As a result it is necessary to to develop new solders for this application. This paper will discuss some of the issues that have arisen and been solved for indium based solders for this application.

9:00 AM
Effects of Surface Finishes on the IMC Formation and Solder Joint Reliability of Memory Module Assembly Using BGA Sn-Ag-Cu Solders: Chang Yong Park[1]; *Byung Man Kim*[1]; Dong Chun Lee[1]; Si Don Choi[1]; Joo Youl Huh[2]; [1]Samsung Electronics Co, LTD, Module Engrg. Part, Device Packing Ctr., Semiconductor Business, Asan, Chungcheongnam-do 336-711 S. Korea; [2]Korea University, Matls. Sci. & Engrg., Anam-Dong 5-1 Sungbuk-Ku, Seoul 136-701 S. Korea

Memory module involves the solder ball attachment on electronic components and the surface mount technology(SMT) of the ball grid array (BGA) components on a printed circuit board (PCB). The most widely used surface finish of the component pads is electrolytic Ni/Au, whereas organic solderability preservative (OSP) or electroless nickel with immersion gold (ENIG) is currently employed for the surface finish of the board side pads. Our previous study showed that the mechanical reliability of the component-side solder joints is largely affected by the surface finishes on the board side pads. In this work, we carried out a systematic study to identify the cause for the mechanical weakness occurring at the component-side solder joints when the OSP finish was used for the board side pads. Employing two, different solder alloys, eutectic Sn-3.5%Ag and Sn-3.0%Ag-0.5%Cu, intermetallic compounds (IMC) formed at the component-side joints during reflow, isothermal aging at 175°C, and thermal cycling were investigated for the OSP and ENIG finishes of the board-side pads. Solder joint reliability was examined by pull-off testing and bending impact testing. This talk will also discuss some remedies to improve the solder joint reliability for the case using the OSP board finish.

9:20 AM
Reliability of In-48Sn Solder/Au/Ni/Cu BGA Packages During Reflow Process: *Ja-Myeong Koo*[1]; Dae-Gon Kim[1]; Seung-Boo Jung[1];

[1]Sungkyunkwan University, Dept. of Advd. Matls., 300 Cheoncheon-dong, Jangan-gu, Suwon, Gyeonggi-do 440-746 Korea

In-48Sn solders offer low melting point and long fatigue life, so are particularly attractive for use in systems requiring low-temperature processing, such as optoelectronic devices. Interfacial reaction and ball shear properties of In-48Sn solder/Au/Ni/Cu BGA packages were investigated, to evaluate the reliability of the solder joints, during reflow process; the reflow process was performed in the reflow temperature range of 403 to 483K for 10 to 7200s in a nitrogen atmosphere. The thickness of $AuIn_2$ IMC at the interface increased during reflow process. The depletion of thin Au layer resulted the formation of Ni_3 $(Sn,In)_4$ IMC layer between the solder and Ni layer. $AuIn_2$ layer spalled away, due to the weak adhesion between $AuIn_2$ and Ni_3 $(Sn,In)_4$ layers. Shear properties decreased due to the growth and spalling of brittle $AuIn_2$ IMCs with reflow time, but increased, after 300s, due to the spalling of $AuIn_2$ IMCs and the formation of $Ni_3(Sn,In)_4$ IMC layer.

9:40 AM

Effect of Additives on Electroplating of SnAg Solder: *Hsiao-Yun Chen*[1]; Chih Chen[1]; Jia-Min Shieh[2]; Bau-Tong Dai[3]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Engrg., 1001 Ta Hsueh Rd., Hsin-Chu 30050 Taiwan ; [2]National Nano Device Laboratories, 1001-1 Ta-Hsueh Rd., Hsin-Chu 30050 Taiwan

Eutectic Sn-Ag alloy solder is one of the most promising lead-free materials for low temperature processing of solder bumps on wafer. Smooth, fine-grained, and near eutectic composition (Sn-3.8 atom% Ag) of Sn-Ag alloy films could be obtained in pyrophosphate-iodide baths containing organic compounds such as PEG600 and other efficient additives by electrodeposition under both galvanostatic and potentiostatic condition. In other words, the composition of electroplated film could be controlled by optimizing with appropriate ratio of additives in main electrolytes and current density on the plating surfaces. All of the plated Sn-Ag films consisted of Ag3Sn phase, Sn, and Ag elements on a Cu foil. Electrochemical analysis such as potentiodyamic (PD) polarization was also performed to investigate the roles of these additives in electrolytes during plating. The results and mechanism will be presented in details in the conference.

10:00 AM

Wetting Properties of Lead-Free Solders: Natalie Sobczak[2]; Rafal Nowak[2]; Artur Kudyba[2]; Boris Mikulowski[3]; *Herbert Ipser*[1]; Adolf Mikula[1]; Hans Flandorfer[1]; [1]University of Vienna, Dept. of Inorganic Chmst., Waehringerstrasse 42, A-1090 Wien A-1090 Austria; [2]Foundry Research Institute, 73 Zakopianska St., Krakow PL-30418 Poland; [3]AGH University of Science and Technology, 30 Mickiewicza Ave., PL-30059 Krakow Pl-30059 Poland

The wetting and spreading behavior of different lead-free solder materials on Cu substrates was determined by a sessile drop method under different atmospheres. After these measurements, the solidified samples were cut into two halves. One part was used for push-off tests in order to determine the shear strength of the solder/substrate couple whereas the other part could be used for various metallographic investigations. Up to now, lead-free solders of the Sn-Ag-Cu, the Sn-Ag-In, and the Sn-Ag-Bi type have been investigated on Cu substrates, and the push-off tests were performed both immediately after solidification and after thermal cycling. The effect of alloying elements and testing conditions on the relationship between wetting and mechanical properties of solder/Cu model joints are discussed. It can be shown that thermal cycling changes the shear strength considerably in some instances. All the results are compared with those obtained for standard Sn-Pb solders.

10:20 AM Break

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Effect of Interfacial Reaction on the Tensile Strength of Sn-3.5Ag/Ni-P and Sn-37Pb/Ni-P Solder Joints: *Zhong Chen*[1]; Min He[1]; Aditya Kumar[1]; Guojun Qi[2]; [1]Nanyang Technological University, Sch. of Matls. Engrg. 639798 Singapore; [2]Singapore Institute of Manufacturing Technology, 71 Nanyang Dr. 638075 Singapore

This work investigates effect of interfacial reaction on the joint strength of two types of solder joints, Sn-3.5Ag/Ni-P and Sn-37Pb/Ni-P. Both tensile strength and fracture behavior of the joints under different thermal aging conditions have been studied. It is found that the tensile strength decreases with increasing aging temperature and duration. The decrease is due to the change in fracture behavior. Several typical failure modes have been classified. In general the failure mode shifts from within the bulk solder in the as-soldered condition toward interfacial failure modes, giving rise to the decrease of joint strength. This work also finds that for the same aging treatment, the strength of Sn-3.5Ag/Ni-P joint degrades faster than that of Sn-37Pb/Ni-P. The main cause of degradation of joint strength is found to be the microstructure change during interfacial reaction. The difference between the two types of joints can be explained by the difference in their interfacial reaction products and their growth kinetics. Current study finds there is an empirical relation between the solder joint strength and the Ni3Sn4 intermetallic compound (IMC) thickness. Therefore the IMC thickness could be used as an indication of the joint strength degradation. Kirkendall voids have been observed in the Sn-3.5Ag/Ni-P joint; however they do not appear to affect the joint strength. The volume change in Ni-P phase transformation during thermal aging generates high tensile stress inside the Ni-P layer. This stress causes mudflat cracking and delamination of the Ni-P coating from its underlying substrate after long time aging at high temperatures.

10:50 AM

Reliability of Adhesion Strength of the Sn-9Zn-1.5Ag-xBi/Cu During Isothermal Aging: *Chih-Yao Liu*[1]; Moo-Chin Wang[2]; Min-Hsiung Hon[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., 1 Ta-Hsueh Rd., Tainan 70101 Taiwan; [2]National Kaohsiung University of Applied Science, Dept. of Mechl. Engrg., 415, Chien-Kung Rd., Kaohsiung 80872 Taiwan

The reliability of adhesion strength of the Sn-9Zn-1.5Ag-xBi/Cu during isothermal aging have been investigated. Due to the growth and decomposition of the intermetallic compounds (IMCs), the adhesion strength vary with aging 150°C from 100 to 1000 hour as wetted at 250°C for 60 s. The Cu6Sn5,Cu5Zn8 and Ag3Sn IMCs are identified at the Sn-9Zn-1.5Ag-xBi/Cu interface as aging. Fracture morphology are revealed that the fracture occured in solder matrix by sanning electron microscopy (SEM) and transmission electron microscopy (TEM) after aging. The diffusion cofficients of Sn and Zn in IMCs layer are determined at the Sn-9Zn-1.5Ag-xBi/Cu interface for aging, respectively.

11:10 AM

Effect of Flux on the Wetting Characteristics of SnAg, SnCu, SnAgBi, and SnAgCu Lead-Free Solders on Copper Substrates: Mario F. Arenas[1]; *Viola L. Acoff*[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487 USA

The effect of flux on the wetting characteristics of four lead-free solders namely, Sn-3.5Ag, Sn-3.5Ag-4.8Bi, Sn-3.8Ag-0.7Cu and Sn-0.7Cu, (all in wt%) on copper substrates have been studied. The fluxes investigated were rosin-nonactivated (R), rosin mildly activated (RMA), and rosin activated (RA). The wetting tests were conducted using the sessile-drop method in combination with a photographic technique that allowed a precise determination of the wetting angle. Experiments were performed at temperatures ranging from 240°C to 280°C. Results showed that fluxes significantly affect the wetting properties of the solder alloys. Contact angles ranging from 10° to 30° for RMA and between 20°-30° for RA were obtained. The use of flux R yielded larger contact angles ranging from 35° to 60°, revealing that it is not suitable as a flux for lead-free solders. The effect of temperature on contact angle depended on the type of flux used. When the temperature was increased, the contact angle decreased for fluxes RA and R. However, the use of RMA flux produced a slight increase in contact angle and in many cases the contact angle was independent of temperature. From the lead-free solders studied, Sn-3.5Ag-4.8Bi exhibited the lowest contact angles indicating improved wettability with addition of bismuth. The microstructure of the solder/copper interface was analyzed by scanning electron microscopy with energy dispersive X-ray spectroscopy. The wetting reaction of molten solder on Cu led to the formation of Cu-Sn intermetallic compounds at the solder-Cu interface. These were identified as Cu_6Sn_5 adjacent to the solder and Cu_3Sn adjacent to the substrate.

11:30 AM

Fundamental Characteristics at Sn-9Zn-1.5Ag-xBi/Cu with Precoating Sn: *Chih-Yao Liu*[1]; Moo-Chin Wang[2]; Min-Hsiung Hon[1]; [1]National Cheng Kung University, Dept. of Matl. Sci. & Engrg., 1 Ta-Hsueh Rd., Tainan 70101 Taiwan; [2]National Kaohsiung University of Applied Science, Dept. of Mechl. Engrg., 415, Chien-Kung Rd., Kaohsiung 80872 Taiwan

The fundamental Characteristics at Sn-9Zn-1.5Ag-xBi/ Cu with precoating Sn have been investigated. The intermetallic compounds (IMCs) are identified by X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) as wetted at 250°C for 60 s. The wetting time and wetting force are determinded by wetting force balance. The corrosion behavior of solder alloys in 3.5 percent NaCl solution are determined by electrochemical measurements. Damage test are revealed the maximun force of Sn-9Zn-1.5Ag-xBi/ Cu with precoating Sn by dynamic mechanical analysis (DMA).

11:50 AM
The Influence of Ni3Sn4 Nanoparticles and Ni Concentration on Morphology of Sn-Ag-Ni Solders by Mechanical Alloying: *Hsiang-Yi Lee*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

In the practice of package system, solder plays a crucial role in the interconnection of silicon die. In this study, mechanical alloying (MA) process was considered as an alternative method to produce the lead free solder pastes of Sn-3.5Ag-xNi (x=0.1, 0.5, 1.5, and 2.0). The particle size of pure-Ni doped solder was above 100μm. With increasing Ni concentration, the particle size was reduced. To reduce the particle size of SnAgNi alloys, Ni_3Sn_4 nanoparticles were doped into Sn and Ag powders to form SnAgNi composite solder. For Ni_3Sn_4-composite solder, the particle size was smaller than the pure-Ni doped solder. When the Ni_3Sn_4 concentration was low (x=0.1, 0.5), MA particles aggregated to a flat ingot with larger particle size. For higher Ni_3Sn_4 concentration (x= 1.5, and 2.0), MA particles turned to be nearly spherical with smaller particle size. The distinction of milling mechanism of Ni_3Sn_4-composite solder and the pure-Ni doped solder was discussed. Besides, the DSC results ensured the compatibility to apply the solder material for the reflow process. Wettability test also revealed that the wetting angle of Ni_3Sn_4-composite solder was smaller than the pure-Ni doped solder.

Magnesium Technology 2005: Magnesium, Primary Production and Environmental

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Monday AM Room: 2004
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA

8:30 AM
Use of CO2-Snow for Protecting Molten Magnesium from Oxidation: Friedrich-Wilhelm Bach[1]; *Alexander Karger*[1]; Christoph Pelz[2]; Mirko Schaper[1]; [1]University Hanover, Inst. for Matls. Sci., Schoenebecker Allee 2, 30823 Garbsen Germany; [2]Linde Gas AG, Unterschleissheim Germany

When processing magnesium alloys, prevention of oxidation of the liquid metal is of prime importance, because of the high oxygen affinity of molten magnesium. A special danger of environmental pollution occurs from the usage of protective gases for molten magnesium. The protective gas SF6, which is widely used nowadays, increases the greenhouse effect due to it's GWP of approximately 23900 relative to CO2. At the Kyoto summit in 1997, this gas was detected as one of six gases to be restricted in use. The environmentally friendly alternatives are being examined by authors of this paper. The research goals of this group are to develop and to evaluate new methods for protecting the surface of magnesium melts. One possible alternative is covering the magnesium melt with CO2-snow. The current results will be presented in the following paper.

8:50 AM
Solid Oxide Membrane (SOM) for Cost Effective and Environmentally Sound Production of Magnesium Directly from Magnesium Oxide: *Ajay Krishnan*[1]; X. Lu[1]; U. B. Pal[1]; [1]Boston University, Dept. of Mfg. Engrg., 15 St. Marys St., Brookline, MA 02446 USA

The Solid Oxide Membrane (SOM) process is being investigated for the direct reduction of magnesium from magnesium oxide. The proof of concept for magnesium extraction by the SOM process was demonstrated earlier using a Magnesium Fluoride based flux system at 1300°C. Since the YSZ anode is the most expensive part of the system, its long-term stability is critical to the success and eventual commercialization of the SOM process. Efforts to increase membrane stability have resulted in the development of a new lower-temperature flux (1150°C). Minor additions of Yttrium-containing compounds to the flux was seen to further improve the YSZ anode stability. The results of the SOM experiments that were conducted at 1300°C and 1150°C using the old and the new flux systems, respectively, are analyzed in terms of their Faradaic efficiency, power consumption, mass-transfer characteristics and purity of magnesium produced. A preliminary cost comparison of the SOM process with the existing state of the art technology is also discussed.

9:10 AM
Wear Testing of Inert Graphite Anodes for Magnesium Production: *Boyd R. Davis*[1]; Joshua Rubenstein[2]; [1]Kingston Process Metallurgy Inc., 1102 Lancaster Dr., Kingston, Ontario K7P 1S6 Canada; [2]Queen's University, Mining Engrg., 25 Union St., Kingston, Ontario K7L 3N6 Canada

The longevity of cells requiring dimensional stability of the electrodes is limited in part because of gradual wearing of the graphite inert anodes. This wear causes increased cell resistance and hinders hydrodynamics. Preliminary scoping tests in a laboratory electrolysis cell were performed to determine whether confocal surface profilometry could be used to study anode wear. This approach provided good reproducibility, limited variation, and both quantitative and qualitative results. Oxide concentration and speciation (MgO vs. MgOHCl) in the electrolyte, as well as graphite properties, were varied to see if they could impact anode wear. Oxide speciation and concentration were found to have a direct impact on anode wear. As well, the graphite's grain size, density, and mechanical properties considerably modified the anode wear.

9:30 AM
Pilot Experiments of Magnesia Direct Electrolysis in a 10kA Magnesium Reduction Cell: *Huimin Lu*[1]; *Chunfa Liao*[1]; *Ruixin Ma*[1]; *Wenhui Yuan*[1]; [1]University of Science and Technology, Metallurgl. Engrg. Sch., No. 30 Xueyuan Rd., Beijing 100083 China

A new technique of magnesia direct electrolysis to produce magnesium with the LaCl3-MgCl2 system as support electrolyte published in Magnesium Technology 2004 has low energy consumption, high current efficiency and less pollution for the environment. In this paper, pilot experiments of the new technique of magnesia direct electrolysis in a 10kA magnesium electrolysis cell are conducted. The electrolyte is also the LaCl3-MgCl2 system. Some experiment phenomena are described. The experiment results indicate that the LaCl3-MgCl2 system is promising. These pilot experiments lay a good foundation for industrial comprehensive utilization of bischofite from Qinghai Lakes in China. In the meantime, the thermo-chemistry, electrolyte ionic species and electrode reactions of the magnesia direct electrolysis in the LaCl3-MgCl2 system are studied. The operational parameters of the new process are also compared with those of the common aluminum production process and MgCl2 electrolysis process.

9:50 AM
Electrochemical Co-Deposition of Magnesium Alloy from Alkali Chloride Melts: Xi Zhang[1]; Shuqiang Jiao[1]; *Hongmin Zhu*[1]; [1]University of Science and Technology, Dept. Physl. Chmst., 30 Xueyuan Rd., Haidian, Beijing 100083 China

A novel process is proposed for direct preparation of magnesium alloys through electrochemical co-deposition from alkali chloride molten salts with relatively lower density (i.e. LiCl~NaCl) than that of magnesium metal. According the theoretical calculation magnesium - aluminium alloy could be produced galvanosatic electrolysis at the conditions of appropriate concentration ratio of Mg2+ and Al3+ in melt. A series of electroanalytical measurements was performed for the system of MgCl2-AlCl3-NaCl-LiCl molten salts, to investigate the electrochemical behavior and basic parameters for the electrode process. A laboratory test of the direct production for the Mg-Al alloy by step-current co-deposition was also performed.

10:10 AM Break

10:25 AM
Thermal Properties of Electrolytic Magnesium Production Feed: *Muhammad R. Tawalbeh*[1]; K. W. Ng[1]; R. Harris[1]; [1]McGill University, Mining, Metals & Matls. Engrg. Dept., 3610 Univ. St., M.H. Wong Bldg., Montreal, Quebec H3A 2B2 Canada

The thermal conductivity of commercial magnesium chloride dihydrate (Prills and powdered prills) was measured at temperatures up to 200°C and of MgOHCl manufactured in house at temperatures up to 500°C as a function of porosity. It was found that the thermal conductivity of the Prills powder as a function of temperature could be represented as: k/k (298.15) =1 + 0.0015(T - 298.15) And as a function of porosity: k = 5.77*exp(- 4.47e) Furthermore, similar correlations were obtained for the MgOHCl powder in terms of the thermal conductivity, which could be represented as a function of temperature: k/k (298.15) =1 + 0.0124(T - 298.15) And as a function of porosity: k = 9.76*exp(- 2.63e) These results will facilitate the study of the thermal history of the prills in Magnola Metallurgie Inc's 'Super-chlorinator'.

10:45 AM
Protecting Liquid Mg by Solid CO2: New Ways to Avoid SF6 and SO2: *Peter Biedenkopf*[1]; Alexander Karger[2]; Michael Laukötter[4]; Willi Schneider[3]; [1]Linde AG - Linde Gas Division, Mkt. Dvlp., Carl-von-Linde-Str. 27, Unterschleissheim (Munich), Bayern 85716 Germany; [2]Universitaet Hannover, Fachbereich Technologie der Werkstoffe, Schoenebeckerallee 2, Hannover, Niedersachsen 30823 Germany; [3]Audi Ag, I/PG-63, Ingoltstadt, Bayern 85045 Germany; [4]Laukötter Gusstechnik GmbH, Krummer Weg 27-29, Wadersloh 59329 Germany

Lots of research had been done to find a new gas solution for replacing SF6 or SO2 in Mg production and processing caused by their extremely high global warming potential or toxicity. Linde Gas in cooperation with industrial and academic partners had developed a new tool for protecting liquid magnesium by covering the melt with solid CO2. Cold dry ice hinders material losses caused by oxidation and reduces also the evaporation of Mg at higher temperatures. The cooling effect of the dry ice also extinguishes fires into a furnace by cooling the surface and replacing leaking nitrogen and oxygen. Lot of work was done to reduce the CO2 consumption to make the process competitive to SF6 or SO2 operation and will be presented.

11:05 AM
Lifecycle Environmental Impact of Magnesium Automotive Components: *Paul Koltun*[1]; Ambalavanar Tharumarajah[1]; Subramania Ramakrishnan[1]; [1]CSIRO, Mfg. & Infrstuct. Tech., PO Box 56, Highett, Victoria 3190 Australia

The development of magnesium applications for automotive industries is receiving significant attention. One aspect is the assessment and reduction of the cradle-to-grave environmental impact of components. The study investigates magnesium converter housing starting from the production of magnesium ingots to the manufacture and assembly, use and recycling. Sensitivity analysis examines the impact of key parameters such as cover gases other than SF6, product yield and use of secondary magnesium, that can improve the environmental performance. Several environmental performance scenarios are proposed and used to compare the impact of similar functional components made of magnesium produced in China, aluminium and iron. The investigations shows significant reductions in the greenhouse gas impact may be achieved from the lighter magnesium components. Also, process improvements to reduce the impact improve the break-even distances in the use of automobiles at which magnesium becomes comparable with other competing metals.

11:25 AM
Mg2(OH)3Cl.2H2O Chlorination: Sina Kashani-Nejad[1]; K.-W. Ng[1]; *Ralph Harris*[1]; [1]McGill University, Mining, Metals & Matl. Engrg., 3610 Univ., Wong Bldg., Montreal, Quebec H3A 2B2 Canada

The kinetics of the hydrochlorination of Mg(OH)2Cl3 isolated form the feed of a commercial electrolytic magnesium production process, with HCl gas was investigated. It was found that particles of the isolated Mg2(OH)3Cl had an average diameter of 150 micron. Over 90% conversion to MgCl2 was observed within 30 minutes by contacting the Mg2(OH)3Cl particles with HCl gas at 350°C. Analysis of the kinetic data suggested that the chlorination process was divided into two regimes. As the chlorination process proceeds, the rate of the chlorination became controlled by the rate of HCl transfer through theMgCl2 layer formed on the Mg2(OH)3Cl core surface.

Magnesium Technology 2005: Wrought Magnesium Alloys I

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Monday AM Room: 2006
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Ravi Verma, General Motors Research & Development, Warren, MI 48090-9055 USA

8:30 AM
A Comparison of the Mechanical Response High Purity Magnesium and Az31 Magnesium Alloy: *Carl M. Cady*[1]; Richard C. Korzekwa[1]; Ellen K. Cerreta[1]; Micheal F. Lopez[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, MST-8, MS G-755, Los Alamos, NM 87544 USA

Constitutive property studies have been conducted on an AZ31 magnesium alloy and high purity magnesium as a function of temperature (-75°C to 200°C), strain rate (0.001 s-1 to 2500 s-1), orientation, and stress state. Minimal influence of strain rate on the yield and flow stress of this material was seen over the range of strain rates from 0.001 to 2500 s-1. Differences in the mechanical response, as a function of orientation with respect to rolling direction and stress-state, will be shown. The yield stress in the through-thickness orientation is higher than that of the in-plane direction for all strain rates. The flow stress for the in-plane orientation is initially lower than that seen in the through-thickness direction but it intersects at ~12% strain. Varying the temperature also influences the through-thickness flow stress behavior significantly more than the in-plane behavior. These differences can be attributed to the dominant deformation mechanism activated for each orientation.

8:50 AM
Magnesium Wrought Alloy Properties of the AZ - Series: *D. Letzig*[1]; J. Swiostek; J. Bohlen; J. Göken; K.U. Kainer; [1]GKSS Forschungszentrum Geesthacht GmbH, Max-Planck-Str. 1, D - 21502 Geesthacht, Germany

The alloy composition in combination with the kind of thermomechanical treatment determines the properties of magnesium semi-finished products like sheets or profiles. The development of the microstructure dependent on the process and on the chemical composition is seen as a fundamental basis for the understanding of the obtained mechanical properties. This also includes the texture development and its influence on the mechanical anisotropy of wrought alloys. The mechanical properties of different wrought alloys were determined by extrusion processes such as indirect and hydrostatic extrusion. A relationship between profile microstructure and resulting mechanical properties was investigated by also using additional methods like damping and acoustic emission measurements. In this paper an overview on the results obtained from the magnesium AZ-series will be given. They will be discussed with respect to the special needs for future developments of magnesium wrought alloys.

9:10 AM
Large Profile Magnesium Alloys Extrusions for Automotive Applications: *Gady Isaac Rosen*[1]; [1]Alubin Ltd., R&D, 36 Yosef Levi St., PO Box 1188, Kiryat Bialik, Israel 27112 Israel

The aim of this project is to develop magnesium alloys extrusion technology for large profiles for application in the automotive industry. In particular it is expected to significantly reduce Chassis weight, thus leading to fuel consumption reduction and enhancement of performance of the cars produced by the end user. The entire process required for magnesium alloys extrusion of automobile Chassis will be addressed as a first attempt of large magnesium profile extrusion. The challenge of large magnesium profile extrusion (outer diameter up to 10") has never been addressed due to technological barriers that relate to limited capability of magnesium extrusion technology. However, these barriers can be overcome if one has control over the starting material. Since the final performance of the finished product depends on the original microstructure formed over Direct Chill (DC) casting, homogenization, extrusion and heat treatment, no processing step can be isolated from the other. The R&D activities will focus on alloy selection, production of large magnesium billets, die design, heat treatment and extrusion parameters. These will be accompanied by an economic assessment of the new technology and additional applications.

9:30 AM
Microstructure Refinement of Magnesium Alloy (AZ31) Through a Combination of Extrusion and Room Temperature Equal Channel Angular Process: *Jeong-Whan Han*[1]; [1]INHA University, Sch. of Matl. Sci. & Engrg., Inchon 402-751 Korea

In order to obtain ultra fine microstructures of magnesium alloy (AZ31), a new processing procedure was applied to Mg-3wt.%Al-1wt%Zn alloy. This procedure involves the sequential application of hot extrusion and equal-channel angular pressing at room temperature. The AZ31 alloy was extruded to a billet with a thickness of 5mm, and then ECA Pressing was carried out at room temperature on the as-extruded material through a die with an internal angle(¥Õ) of 135 between the vertical and horizontal channels and a curvature angle(¥×) of 45. For the ease of room temperature ECAP, heat treatment of extruded slab was carried out at a temperature of 623K. Experiments show that the magnesium alloy(AZ31) has an initial grain size of about 90§- after casting and it is further reduced to about 2§- when the extruded alloy is subjected to ECAP at room temperature(298K). A

combination processing of extrusion and room temperature ECA Pressing using magnesium alloy suggested a application as structural material because microstructure was refined by severe plastic deformation just like extrusion under high extrusion ratio followed by a shear deformation by means of room temperature ECA Pressing without grain growth.

9:50 AM
Measuring the Temperature Dependence of the Flow Surface of Magnesium Alloy Sheet: *Ashutosh Jain*[1]; Sean R. Agnew[1]; [1]University of Virginia, Matls. Sci & Engrg., 116 Engineer's Way, Charlottesville, VA 22904 USA

The present study attempts at characterizing the anisotropic behavior of the Mg AZ31B sheet alloy and constructing a flow surface using minimum possible experimental data. Uni-axial tests in compression and tension were conducted at room temperature and at elevated temperatures to measure the strain anisotropy at various strain levels. The true-stress true-strain curves at room temperature show that the material is soft during in-plane compression and then strain hardens rapidly at å >0.05. Compression in the normal direction is observed to be the typical parabolic hardening curve. With an increase in temperature, the steep hardening in the RD and TD compression decreases and finally vanishes at T ~ 150°C even though the strain anisotropy is significant at this temperature. The flow stresses at different strains were used to construct flow surfaces, at different temperatures. Finally, a visco-plastic self-consistent approach was used to model the deformation behavior and simulate a poly-crystal yield sur face at room temperature. The model was fit using the RD tensile flow stress and strain anisotropy.

10:10 AM Break

10:25 AM
High Strength Wrought Magnesium Alloys in Employing Grain-Refined Powder: *Katsuyoshi Kondoh*[1]; [1]University of Tokyo, Rsch. Ctr. for Advd. Sci. & Tech., 4-6-1, Komaba, Muguro-ku, Tokyo 153-8904 Japan

Magnesium alloy powder with fine grains less than 5 ìm was produced via continuously direct plastic working on coarse raw powder with 1~4mm length. Dynamic recrystallization of the matrix grains during the warm plastic working causes the refinement of grains and the drastic hardening. When optimizing the process parameters of the plastic working, AM60 wrought alloy after hot extrusion shows UTS of 380MPa and 6% elongation at room temperature. It reveals uniformly refined microstructure, in particular the mean grain size of 1.4 ìm.

10:45 AM
Microstructure Control of AZ31 Alloys by Different-Speeds-Rolling and the Formability: *Tsunemichi Imai*[1]; Naobumi Saito[1]; Ichinori Shigematsu[1]; Kazutaka Suzuki[1]; Shangli Dong[2]; [1]National Institute of Advanced Industrial Science & Technology, Matls. Rsch. Inst. for Sustainable Dvlp., 2266-98 Shimoshidami, Moriyama-ku, Nagoya 463-8560 Japan; [2]Harbin Institute of Technology, Sch. of Matls. Sci. & Engrg., PO Box 432, Harbin 150001 China

AZ31 alloys processed by different-speeds-rolling (DSR) have relatively uniformly fine grain size of 10~20mm and strong anisotropy of mechanical properties by texture built during DSR processing. And, the AZ31 rolled with the billet temperature of 623K and roller of room temperature produces anisotropy in superplastic elongation at the strain rate of 10-2 s-1 and 723K. Conical Cup Testing and Blow Forming of the AZ31 processed by DSR are performed to make clear the Formability.

11:05 AM
Effect of Strontium on the Microstructure and Mechanical Properties of AZ31 Magnesium Alloy: *Yingxin Wang*[1]; Xiaoqin Zeng[1]; Wenjiang Ding[1]; *Alan Luo*[2]; Anil K. Sachdev[2]; [1]Shanghai Jiao Tong University, Natl. Engrg. Rsch. Ctr. of Light Alloys Net Forming, Huashan Rd. 1954, Shanghai, Shanghai 200030 China; [2]General Motors Research and Development Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA

The effect of strontium on the microstructure and mechanical properties of AZ31 magnesium alloy, and the sensitivity to section thickness of the grain refinement are investigated. Three phases, block-shaped Mg17Al12, acicular Mg20Al20Mn5Sr and insular Mg16(Al,Zn)2Sr are identified in the Sr-containing AZ31 alloys. The results show that, with reducing section thickness, the amount of block-shaped Mg17Al12 phases increases, but acicular Mg20Al20Mn5Sr phases diminish and insular Mg16(Al,Zn)2Sr phases are refined and granulated. The study suggests that the grain size decreases with the reducing section thickness at a given composition, while the grain size decreases first, then increases and finally decreases again with increasing Sr content at a given section thickness. The mechanical properties of AZ31 magnesium alloy are improved by the Sr grain refinement, and the yield strength (sy) can be expressed as a function of the grain size (13d-1/2) according to the Hall-Patch relationship sy=35.88+279.13d-1/2.

11:25 AM
Grain Refinement of Wrought AZ61 Magnesium Alloy: Zhou Haitao[1]; Liu Chuming[2]; Ding Wenjiang[1]; [1]Shanghai Jiaotong University, Sch. of Matls. Sci. & Engrg., 1954 Huashan Rd., Shanghai 200030 China; [2]Central South University, Sch. of Matl. Sci. & Engrg., Hunan Changsha, Changsha 410083 China

Deformation behaviors of AZ61magnesium alloy during hot compression at temperature range from 523 K to 673 K and strain rate from 0.001s-1 to 1 s-1 were investigated. Dynamic recrystallization took place, and grains could be greatly refined through dynamic recrystallization. The mean size of the recrystallized grains decreased with the decrease of temperature or the increase of Z (Zener-Hollomon parameter), while the reciprocal of the recrystallized grains had a good linear relationship with the natural logarithm of Z value. The fine grain microstructure of extruded AZ61 magnesium alloy between 10-25µm were observed at the lower deformation temperature of 523-623K by means of the relationship between d and Z parameter.

11:45 AM
Investigation of Low Temperature Superplasticity on Mg-AZ31B Sheet: *Ozgur Duygulu*[1]; Sean R. Agnew[1]; [1]University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA

Mechanical properties and low temperature superplasticity of commercial magnesium alloy AZ31B sheet is investigated by tensile tests at temperatures between room temperature and 200°C. In order to examine the low temperature superplasticity of magnesium alloy, tensile tests are performed at elevated temperatures at initial strain rates between $1x10^{-4}$ and $1x10^{-1}$ s^{-1}. Strain rate jump tests are also performed at true strain rates between $5x10^{-7}$ and $5x10^{-2}$ s^{-1}. Strain rate sensitivity is found to be less than 0.2 at 200°C for strain rates higher than $5x10^{-5}$ s^{-1}. Even though strain rate sensitivity is low, respectable ductility is observed with elongations up to 250%.

Materials Processing Fundamentals: Solidification & Casting

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Monday AM Room: 3001
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

8:30 AM
Modeling the Effects of Internal Convection on Dendritic Evolution in Stainless Steel Alloys: *Alaina B. Hanlon*[1]; Robert W. Hyers[1]; Douglas M. Matson[2]; [1]University of Massachusetts, Dept. of Mechl. & Industl. Engrg., Engrg. Lab Bldg., 160 Governors Dr., Amherst, MA 01003 USA; [2]Tufts University, Dept. of Mechl. Engrg., Rm. 025 Anderson Hall, 200 College Ave., Medford, MA 02155 USA

Certain Fe-Cr-Ni stainless steel alloys solidify from an undercooled melt by a 2-step process in which the metastable ferrite phase transforms to a stable austenite phase. Recent experiments have shown that the lifetime of the metastable phase is strongly influenced by flow within the molten sample. The current research will provide insight to why flow affects the metastable phase. This will lead to the use of convection to control microstructural evolution and advancements in spray-forming of stainless steels. Simulations using a commercial computational fluid dynamics package, FIDAP, yield a range of convective flow velocities that cause mechanical damage to the dendrites and are compared to experimental results. If the convective velocities are great enough such that the dendrites bend then low angle boundaries

form at the points of collision. These result in high energy sites that could serve as nuclei for the stable phase.

8:55 AM
Predictions of Segregation Related Defects in Vacuum Arc Remelting of Titanium Alloys: *Kent J. VanEvery*[1]; Matthew John M. Krane[1]; [1]Purdue University, Matls. Engrg., 501 Northwestern Ave., W. Lafayette, IN 47907-2044 USA

Macrosegregation during vacuum arc remelting (VAR) of a wrought titanium alloy (Ti-10-2-3) has been modeled. Features of the macroscopic model include species transport and DC electromagnetic effects coupled with fluid flow, heat transfer, and solidification. The effects of different melt histories and hot top procedures on predicted macrosegregation patterns, local solidification times, and liquid pool profiles are demonstrated. Changes in the ingot radius and height, as well as the current distribution into the melt pool, are evaluated. The triple melt of an ingot is also modeled, with the segregation in a given stage affecting the input of metal into the downstream stages. The triple melt results are compared to the segregation levels for a single melt with a homogeneous electrode.

9:20 AM
Inverse Algorithm for Parameter Identification and Process Design in Materials Processing: *Kei Okamoto*[1]; *Ben Q. Li*[1]; [1]Washington State University, Sch. of Mechl. & Matls. Engrg., PO Box 642920, Pullman, WA 99163 USA

This paper discusses the parameter identification and process design for solidification processing systems using the inverse methods. The inverse algorithm entails the use of the Tikhonov regularization method, along with the L-curve method used to select optimal regularization parameters for inverse calculations. The algorithm development and its application to determine material property parameters and to the design of materials processing systems are discussed. The inverse identification of diffusivity data from experimental measurements is discussed. The design algorithm also is applied to determine the appropriate boundary heat flux distribution in order to obtain a unidirectional solidification front in a 2-D cavity by eliminating the effect of natural convection. Inverse calculation is also performed for the case in which the solid-liquid interface is prescribed by a process designer.

9:45 AM
Effect of Dendrite Coarsening and Back Diffusion on Macrosegregation in a Solidifying Alloy: *Deep Samanta*[2]; Nicholas Zabaras[1]; [1]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 188 Frank H.T. Rhodes Hall, Ithaca, NY 14853 USA; [2]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 169 Frank H.T. Rhodes Hall, Ithaca, NY 14853-3801 USA

Solidification of an alloy is invariably accompanied by redistribution of solute on both macro and micro length- scales. Microsegregation, which refers to distribution of solute on a length scale of secondary dendrite arms spacings, influences the underlying microstructure of a cast alloy. This in turn affects final mechanical properties of the alloy. Two limits of microsegregation are the Lever rule (infinitely fast back diffusion) and Scheil rule (no back diffusion). Under the assumption of equilibrium at the solid-liquid interface, solute redistribution with a finite amount of back diffusion occurs between these two limits. Dendrite coarsening also affects solute redistribution. It has been shown to bridge differences in microsegregation predicted by the two limiting models even under equilibrium conditions. Our main aim is to incorporate dendrite coarsening, back diffusion and interfacial transport mechanisms into an alloy solidification model where the governing equations of fluid flow, heat and solute transport are based on volume averaging. This composite model will then be used to study effects of both these mechanisms on macrosegregation in a solidifying alloy. Macrosegregation predictions from here will be compared with those obtained using Scheil and Lever rule models in the presence of convection. Simulations will be carried out using a stabilized finite element model for solidification developed recently and both two and three dimensional examples will be considered.

10:10 AM Break

10:25 AM
Numerical Simulation and Experimental Characterization of a Binary Aluminum Alloy Spray - Application to the Spray Rolling Process: *Samuel B. Johnson*[1]; Jean-Pierre Delplanque[1]; Yaojun Lin[2]; Yizhang Zhou[2]; Enrique J. Lavernia[2]; Kevin M. McHugh[3]; [1]Colorado School of Mines, Div. of Engrg., 1610 Illinois St., Golden, CO 80401 USA; [2]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Coll. of Engrg., One Shields Ave., Davis, CA 95616 USA; [3]Idaho National Engineering and Environmental Laboratoty, Industl. & Matl. Techs. Dept., PO Box 1625, Idaho Falls, ID 83415 USA

A stochastic, droplet-resolved model has been developed to describe the behavior of a binary aluminum alloy spray during the spray rolling process. In this process, a molten aluminum alloy is atomized and the resulting spray is deposited on the rolls of a twin-roll caster to produce aluminum strip. The one-way coupled spray model allows the prediction of spray characteristics such as enthalpy and solid fraction and their distribution between the nozzle and the deposition surface. This paper outlines the model development and compares the predicted spray dynamics to PDI measurements performed in a controlled configuration. Qualitative comparisons are also made with the observed behavior of the spray rolling process.

10:50 AM
The Recrystallisation and Texture in Strip Cast Low Carbon Steel: *Wanqiang Xu*[1]; Michael Ferry[1]; [1]University of New South Wales, Sch. of Matls. Sci. & Engrg., Sydney, NSW 2052 Australia

Direct strip cast steel is being commercialised because of its low cost. The formability of strip cast low carbon steel, mainly depended on the texture components, needs to be improved through thermal mechanical processing. Four microstructures -acicular ferrite, bainite, fine polyganol ferrite and coarse polyganol ferrite, can be formed in strip casting low carbon steel. Fours kinds of specimens with the above imitated microstructures and one kind of direct strip cast specimen with acicular ferrite, were cold rolled at a reduction rate 50%, 70% and 90% respectively. All of the cold rolled specimens were annealed at 580, 600, 620 and 640°C in lead bath. The recrystalisation behaviour and texture in the annealled specimen were analysed.

11:15 AM
Microstructural Evolution and Deformation Behavior of Austenitic Stainless Steel in Semi-Solid State: *Jingyuan Li*[1]; [1]University of Tokyo, Inst. of Industl. Sci., Komaba 4-6-1, Meguro-Ku, Tokyo 153-8505 Japan

Thixoforming or Semi-Solid Metal Forming offers many advantages in comparison with casting and conventional forging. The purpose of the present study is to provide the basic microstructure and deformation data for austenitic stainless steel under mushy state. As well known, the alloys in Fe-Cr-Ni ternary system solidify in different modes according to the different position in the phase diagram. In this paper, microstructural evolutions of three kinds of austenitic stainless steel, which solidify in different modes, are investigated during partial remelting by way of SIMA (Strain Induced Melted Activation). Also, hot compression tests of these alloys for varied combination of deformation rate and deformation temperature in semi-solid state are conducted. Flow stress curves exhibit abrupt change according to the difference in inner microstructure. Phase segregation, fracture and deformation of solid particles are observed after compression tests. Last, various deformation mechanisms are proposed for various microstructures.

11:40 AM
Investigation of the Effects of As-Cast Microstructure and Temperature on the Response of Aluminum Alloy 3004 Ingots to Homogenization: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Sch. of Engrg., L. V. Eberhard Ctr., Ste. 718, Grand Rapids, MI 49504-6495 USA

The effects of the fineness of as-solidified cast microstructure and temperature on the response of aluminum alloy 3004 to homogenization heat treatment have been investigated. The study was conducted on directionally solidified laboratory-size ingots with a range of solidification rate of 0.10 K/s to 30 K/s, at 811-868 K. Optical and electron microscopy, quantitative image analysis, electrical conductivity, and electron probe microanalysis were used to quantify the effects of homogenization on the microstructure of aluminum alloy 3004. In general, increase in fineness of as-cast microstructure and homogenization temperature enhanced the alpha-transformation reaction, dissolution of Mg2Si, elimination of dendritic microsegregation, and dispersoid formation. Homogenization reduced the average size of second phase particles in ingots solidified at <1 K/s. However, in ingots solidified at above 1 K/s, homogenization caused the coarsening of the second phase particles. Mn in solid solution was independent of homogenization heat treatment temperature.

Mechanical Behavior of Thin Films and Small Structures: Strengthening Mechanisms at Small Length Scale

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Monday AM Room: 2024
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Xinghang Zhang, Texas A&M University, Dept. Mechl. Engrg., College Sta., TX 77843-3123 USA; Richard G. Hoagland, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

8:30 AM Opening Remarks: Welcome and Overview of the Symposium

8:35 AM Invited
Shear of Weak Interfaces as a Strengthening Mechanism in Nanoscale Layered Composites: *Richard G. Hoagland*[1]; John P. Hirth[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

In layered composites containing incoherent interfaces, such as those formed from metals with dissimilar crystal structure or large lattice parameter mismatch, coherency stresses are unlikely as a source of resistance to slip. However, when the layer thicknesses are reduced to a few nanometers, these materials, e.g., Cu/Nb, achieve very high strength levels. Atomistic simulations of an fcc/bcc system containing incoherent interfaces, reveals that such interfaces will shear in response to the stress fields of nearby dislocations. We present these results and show their implications that: 1) dislocations near such interfaces are attracted to them, 2) available glide dislocation densities are reduced or eliminated as the layer thickness is reduced and 3) once they enter an interface the cores of the dislocatios spread. Both consequences make it difficult for slip to tranfer across the interface and we suggest that this behavior can explain the origins of strength in systems with incoherent interfaces. We also de scribe a linear elastic model of a dislocation interacting with shear cracks created on a weak interface that helps to generalize the results to enable predictions of the relation of interfacial shear strength to the overall strength of this type of composite. This work was supported by the Office of Basic Energy Sciences, U. S. Dept. of Energy.

9:00 AM
Mechanical Behavior of Nanolayered Laminated Composites: X. Deng[1]; C. Cleveland[1]; M. Koopman[2]; T. N. Lin[3]; Y. Y. Hsieh[3]; N. Chawla[1]; *K. K. Chawla*[2]; J. P. Chu[3]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA; [2]University of Alabama, Dept. of Matls. Sci. & Engrg., Birmingham, AL 35209 USA; [3]National Taiwan Ocean University, Inst. of Matls. Engrg., Keelung 202 Taiwan

Small-length scale multilayered structures are attractive materials because of their extremely high strength and flexibility, relative to conventional laminated composites. In this talk we present results on nanolayered laminated composites of Al and SiC. The laminated composites were fabricated by physical vapor deposition (magnetron sputtering) of alternate layers of Al and SiC. The microstructure of the multilayered structures was characterized by transmission electron microscopy (TEM). The mechanical properties of the layered materials were characterized by nanoindentation and tensile testing. Tensile testing was conducted on a high-resolution micromechanical testing system. The influence of layer thickness on hardness and Young's modulus of individual layers was quantified. The Young's modulus and tensile strength of the composites were also measured. Comparison of experimentally-determined Young's modulus with predictions from analytical models will be presented. Finally, a comparison of tensile strength of the composite with that of monolithic materials, as well as differences in fracture behavior will be discussed.

9:15 AM
Strength of Metallic Multilayers at All Length Scales Via a Dislocation-Based Model: *Lei Fang*[1]; Lawrence H. Friedman[1]; [1]Pennsylvania State University, Dept. of Engrg. Sci. & Mech., 212 Earth & Engrg. Sci. Bldg., Univ. Park, PA 16802 USA

Metallic multilayers can be used as ultra-high strength coatings. They exhibit a very strong Hall-Petch-like size-effect where the mechanical strength depends on layer thickness. At large length scale, the behavior of multilayers can be described by scaling laws (Friedman and Fang, TMS Letters, 1:3, 2004). At small length scales, discreteness of dislocations becomes important, and large deviation from the scaling law occurs. A complete analytic model should apply at all length scales. A dislocation-based model is constructed analytically for the very large and very small length scales. Combining these solutions, an analytic formula that spans the entire length scale range is obtained. The consequences to the multilayer deformation map (Misra, et al., Script Mat., 41(9): 973, 1999) are also discussed. The model is applied to Cu/Ni multilayers.

9:30 AM Invited
Nano-Structured Ultra-Hard Al-Si Films Synthesized by High Rate Deposition: Velimir Radmilovic[1]; *David Mitlin*[2]; Ulrich Dahmen[1]; [1]Lawrence Berkeley National Laboratory, University of California, NCEM, MS-72, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [2]University of Alberta, Cheml. & Matls. Engrg., Edmonton, Alberta T6G 2G6 Canada

We used high rate electron-beam co-evaporation to synthesize Al-Si thin films displaying remarkable mechanical properties. The composition of these films was varied from 2 to 23at.%Si. These films were compared with pure Al films grown using identical deposition conditions to be studied as a baseline. With increasing Si content the nano-indentation hardness increased from 1 GPa for the pure Al film to 4 GPa for the film containing 30at.% Si. Analysis of the indents indicated that all samples exhibited plasticity during deformation. We analyzed the films' microstructure using conventional, atomic resolution and analytical transmission electron microscopy. The microstructure of the alloy films was an order of magnitude finer than that of pure Al, with grain sizes in the range from tens to several hundreds of nanometers. The Si grains were multiply twinned, whereas as expected the Al grains were twin-free. In addition, the Si concentration in the Al grains was consistently measured to be well above the equilibri um room temperature composition. This work was supported by the Director, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

9:55 AM
Mechanical Characterization of Nanoporous Zeolite Low-k Thin Films: *Junlan Wang*[1]; Lili Hu[1]; Yushan Yan[2]; [1]University of California, Dept. of Mechl. Engrg., Riverside, CA 92521 USA; [2]University of California, Dept. of Cheml. & Environml. Engrg., Riverside, CA 92521 USA

Zeolites are a class of inorganic crystalline oxides with uniform molecular-sized pores (0.3 - 2nm). Nanoporous zeolite thin films have been demonstrated to be a promising candidate for low-k applications. A key advantage of zeolite-based low-k materials is the ability to lower k while maintaining higher mechanical strength than other amorphous porous low-k materials. With the rapid development in the synthesis process of different zeolite thin films and the evaluation of their electrical and chemical functions, characterizations of the mechanical and interfacial properties have also been brought to demand. During the integration process of low-k films with other semiconducting materials, mechanical and interfacial strength of the film plays an important role in controlling the later performance and lifetime of the integrated devices. In this work, depth sensing nanoindentation and laser induced thin film spallation techniques are used to evaluate the mechanical and interfacial properties of zeolite thin film s on Si substrates synthesized with different hydrothermal methods. The results provide meaningful guidelines for the optimization of zeolite thin film synthesis processes.

10:10 AM Break

10:25 AM Invited
Mechanical Properties of Copper with Nano-Scale Twins: L. Lu[1]; Y. F. Shen[1]; *K. Lu*[1]; [1]Chinese Academy of Sciences, Inst. of Metal Rsch., Shenyang Natl. Lab. for Matls. Sci., Shenyang 110016 China

Significantly increasing the number of grain boundaries (GBs) by grain refinement in polycrystalline metals into the nanometer scale leads to a substantial change in mechanical properties. Similarly, it is interesting to learn the mechanical behaviors of polycrystalline metals in which a high density of twin boundaries (TBs) are introduced, esp. when the twin lamella thickness is the nanometer regime. By using the pulsed electro-deposition technique, a pure Cu sheet was synthesized with a unique microstructure: a high density of twins with nano-scale thickness confined in submicron-sized grains. Mechanical properties of the the as-deposited sample were investigated by means of tensile tests and nanoindentation measurements. An extremely high tensile strength up to 1 GPa with an elongation-to-failure of 13.5% was observed. The plastic deformation mechanism and the effect of a high density of TBs on mechanical properties will be discussed in terms of microstructural observations and measurement results of the strain rate sensitivity.

10:50 AM
Grain-Boundary Relaxation in Nanocrystalline Fe and its Effect on the Mechanical Behavior: Dongchan Jang[2]; *Michael Atzmon*[1]; [1]University of Michigan, NERS & MSE, Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Dept. of MSE, 3062 H. H. Dow, 2300 Hayward, Ann Arbor, MI 48109-2136 USA

Extensive plastic deformation, e.g., by mechanical attrition, can result in grain diameters as small as 10 nm. The dependence of the mechanical behavior on the grain size in this regime has been studied extensively. However, the mean grain diameter is not the only parameter that determines the mechanical behavior. In the present study, we have investigated the structure and mechanical behavior of nanocrystalline Fe formed by ball milling. The mean grain size has been characterized by the Warren-Averbach method and confirmed by transmission electron microscopy (TEM). Following low-temperature thermal treatment that does not affect the grain size or the hardness, we observe significant changes in the strain-rate sensitivity. The evolution is similar at different annealing temperatures, but its rate is temperature dependent. A likely explanation of this behavior is based on nonequilibrium structure of or impurity redistribution at the grain-boundaries. In this paper, both possibilities are addressed using high-resolution TEM.

11:05 AM
The Influence of Grain Size on Mechanical Behaviors in Nanostructured Metals: *Hongqi Li*[1]; Fereshteh Ebrahimi[2]; [1]University of Tennessee, Matls. Sci. & Engrg. Dept., Knoxville, TN 37996 USA; [2]University of Florida, Matls. Sci. & Engrg. Dept., Gainesville, FL 32611 USA

Based on the hardness measurement and computer simulation results, there generally is a strongest grain size. That is, the strength of nanocrystalline materials starts to decrease with further decreasing the grain size when the grain size is below a critical value. Furthermore, computer simulation reveals that the deformation mechanisms are different below and beyond this critical value. In current study, the tensile behaviors of nanocrystalline nickel and nickel-iron alloys were investigated to characterize how they change with the grain size.

11:20 AM Invited
Yield of Gold Nanowires: *Ken Gall*[1]; Jiankuai Diao[1]; Martin L. Dunn[1]; [1]University of Colorado, Dept. of Mechl. Engrg., Boulder, CO 80309 USA

Strength is one of the most fundamental and significant mechanical properties of a material; it measures the basic capability to bear load. The yield, or fracture, of a metal results in a decrease, or complete loss, of load bearing capacity and, in most cases, loss of functionality. We use atomistic simulations to systematically investigate the strength of experimentally observed gold wires that span atomic and nanometer size scales. The atomistic predictions of strength are quantitatively consistent with discrete experimental observations at extreme size scales and they reveal, for the first time, the mechanisms for increasing nanowire strength with decreasing dimensional scale. At nanometer scales, the mechanism for strengthening involves the scarcity and low mobility of dislocations coupled with constraint from tensile surface stresses. As the wires approach the atomic, or sub-nanometer scale, a sharp increase in strength occurs commensurate with a change in the stable structure of the nanowires and a disappearance of dislocation-mediated yield. The results constitute a new fundamental understanding of the yield behavior of low-dimension metallic wires.

11:45 AM
Mechanical Properties of GaN and ZnO Nanowires Using Nanoindentation: *Gang Feng*[1]; Youngki Yong[2]; Cheol Jin Lee[3]; Kyeongjae Cho[2]; William D. Nix[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305 USA; [2]Stanford University, Mechl. Engrg., Durand Bldg., 496 Lomita Mall, Stanford, CA 94305 USA; [3]Hanyang University, Dept. of Nanotech., 17 Haengdang-dong, Seongdong-Gu, Seoul 133-791 Korea

Interest in the mechanical properties of nanowires is related to the large strains that can exist in these materials and the electronic effects of such strains. Because of the small dimensions, nanoindentation is a useful technique for the study of their mechanical properties. In this work, the mechanical properties of GaN and ZnO nanowires have been studied using the Nanoindenter XP™ with image scanning capability. To minimize the uncertainty in determining the contact area, the true hardness (H_t) is used, HM^2/E^2. Here, H and E are the Oliver-Pharr hardness and Oliver-Pharr reduced modulus, respectively; M is the calculated reduced modulus based on the bulk properties of the materials under study. We found that H_t remains constant while H scatters significantly. Since the geometry of the indentation of nanowires differs from that for the indentation of a half space, a new model is provided to analyze for this effect.

12:00 PM
Anomalies in Stiffness and Damping of a 2D Discrete Viscoelastic System Due to Negative Stiffness Components: *Yun-Che Wang*[1]; John G. Swadener[1]; Roderic S. Lakes[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]University of Wisconsin, Dept. of Engrg. Physics, 1500 Engrg. Dr., 146 Engrg. Rsch. Bldg., Madison, WI 53706 USA

The recent development of using negative stiffness inclusions to achieve extreme overall stiffness and mechanical damping of composite materials reveals a new avenue for constructing high performance materials. One of the negative stiffness sources can be obtained from phase transforming materials in the vicinity of their phase transition, as suggested by the Landau theory. To understand the underlying mechanism from a microscopic viewpoint, we theoretically analyze a 2D, nested triangular lattice with pre-chosen elements containing negative stiffness to demonstrate anomalies in overall stiffness and damping. Combining with current knowledge from continuum models, based on the composite theory, such as the Voigt, Reuss, and Hashin-Shtrikman model, we further explore the stability of the system with Lyapunov's indirect stability theorem. The evolution of the microstructure in terms of the discrete system will be discussed. A potential application of the results presented here is to develop special thin films with unusual in-plane mechanical properties.

12:15 PM
Investigation of Size Effects in the Mechanical Behavior of FCC Bicrystals by Quasicontinuum Method: *Frederic Sansoz*[1]; Jean-Francois Molinari[2]; [1]University of Vermont, Dept. of Mechl. Engrg., 33 Colchester Ave., Burlington, VT 05405 USA; [2]Johns Hopkins University, Dept. of Mechl. Engrg., Latrobe Hall, Baltimore, MD 21218 USA

Grain boundary deformation mechanisms play an important role on the onset of crystal lattice plasticity and cooperative grain behavior in nanocrystalline films and coatings at room temperature. Only the relevant information obtained from atomic scale processes at the boundaries, however, should be taken into account in order to address the challenge of scales involved in the hierarchical modeling of deformation of nanocrystalline materials. This task is made complicated by the fact that the constitutive response of a grain boundary, which is usually obtained by testing a bicrystal, accounts for both interface behavior (atom shuffling, sliding, GB dislocations) and grain bulk behavior (lattice dislocations, mechanical twinning). This investigation provides new insights into the influence of grain size on the mechanical properties of Σ tilt bicrystals in Cu and Al. Molecular static simulations incorporating the quasicontinuum theory were used to model the shear and separation of a bicrystal containing a boundary at its center. The grain size was varied from few atomic planes up to 100 nm. Both symmetric and asymmetric tilt grain boundaries were investigated. It is observed that the grain size has a strong effect on the maximum grain boundary strength, which is related to the nature of mechanisms triggered in the GB vicinity. Atom shuffling process is shown to have the most significant contribution in the decrease of boundary strength. This process is found related to the grain boundary structure through migration of GB defects.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Dislocation Mechanics of Plasticity

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

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Monday AM Room: 3000
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: F. R.N. Nabarro, University of the Witwatersrand, Sch. of Physics, Johannesburg S. Africa; M. A. Meyers, University of California, Matls. Sci. & Engrg., La Jolla, CA 92093 USA

8:30 AM Opening Remarks: Dr. William D. Nix

8:35 AM Invited
Chemical Theory of Dislocation Mobility: *John Joseph Gilman*[1]; [1]University of California, Matls. Sci. Dept., 6532 Boelter Hall, Los Angeles, CA 90095 USA

The conventional theory of dislocation mobility (Peierls-Nabarro theory) considers the geometry of dislocation cores, but not their chemistries. As a result it does not agree with the experimental facts. An adequate theory should take into account differing bonding types: covalent, metallic, ionic, or dispersion. Dislocation lines move when kinks along their lengths move. These kinks may be either sharp or diffuse depending on the bonding type. Diffuse kinks, as in simple metals, offer no resistance to kink motion, so electron and phonon viscosity determine mobility. At sharp kinks in covalent crystals, embedded chemical exchanges determine the mobility. Walsh correlation diagrams can be used to calculate mobilities in this case.

8:55 AM Invited
A Dislocation Multiplication Length Scale for Plasticity: Strain Hardening in Small Crystals by Dislocation Starvation: *William D. Nix*[1]; Julia R. Greer[1]; Gang Feng[1]; [1]Stanford University, Dept. Matls. Sci. & Engrg., 416 Escondido Mall, Stanford, CA 94305-2205 USA

For more than 50 years strain hardening of soft metal crystals has been understood as a process of dislocation multiplication and elastic interaction. According to this classical picture one might expect small crystals with free surfaces to be soft and to exhibit little strain hardening. Dislocations created by multiplication might be free to leave the crystal before interacting with other dislocations. But if dislocations multiply through the glide motion of other dislocations, as J.J. Gilman and others argued nearly forty years ago, then there is a length scale for the multiplication process, related to the average distance a dislocation glides before creating another. When the crystal size is less than this length scale, dislocations would leave the crystal faster than they are created by multiplication. As a consequence, the crystal would be expected to initially soften before hardening by a process of dislocation starvation. As the dislocation density approaches zero, nucleation of new dislocations either at free surfaces or within the bulk of the crystal would be required to maintain plastic flow. The flow stress needed for these processes would approach the theoretical strength of the crystal. We have recently conducted deformation experiments in crystals on a small scale to shed light on these processes. In particular, we have conducted uniaxial compression experiments on tiny samples of single crystal gold made by focused ion beam machining and integrated circuit fabrication methods. These experiments involve small deformation volumes and minimal strain gradients. They show that sub-micron sized crystals become remarkably stronger as they are plastically deformed and approach the theoretical strength after 10-20% strain. The results are consistent with picture of strain hardening by dislocation starvation, which is expected when the crystal size is less that the length scale for dislocation multiplication.

9:15 AM Invited
Length Scales for Volumes Small in 2 and 3 Dimensions: *William W. Gerberich*[1]; [1]University of Minnesota, Chem. Engrg. & Matls. Sci., Minneapolis, MN 55455-0132 USA

Mechanically, very thin films, nanospheres, nanotowers, nanoboxes and other small geometric features are principally characterized by nanoindentation. Such structures have the additional commonality of small volume (V) to surface area (S) associated with the required small indentation depth. Since it has been shown that V/S as a length scale can be related to both surface energy effects at small penetration and dislocation hardening at deeper depths, this is used as a predictor of flow strength. In terms of V/S there are several ways to define a length scale. One could take the entire volume and divide by the entire surface area or alternatively find the ratios of just the deformed volume to the contact surface area. If you assess the former, you find that a solid cube of height, h, or a solid sphere of diameter,d, gives a length scale of h/6 or d/6. This compares to a hollow cube or sphere with length scale of t/2 with t the wall thickness. This gives a reasonable representation of yield strength for the initial deformation using an Orowan criterion. Thus thin-walled structures may have length scales much smaller than their solid counterparts, the implication being that these would support higher load without permanent deformation. At larger strains where constraint is supplied by a diamond contact on the top and a hard substrate on the bottom, a linear hardening model based on dislocation spacing is more appropriate. This gives a length scale of 2b(h-Ä)/Ä where b is the burgers vector and Ä is displacement. One can also compare this to h/6 for a solid cube which implies a greater strength for a thin-walled box undergoing displacements of Ä > b/12. To first order these give predictions of the flow strength as a function of displacement. Results on titanium, silicon and aluminum will be presented. The impact of such length scales on both the analysis of constitutive relations for small volumes and the use of these in computational materials science is discussed.

9:35 AM
Correlating Changes in Dislocation Substructures to Plasticity Size-Effects in FCC-Derivative Metals: *S. J. Polasik*[1]; D. M. Dimiduk[2]; M. D. Uchic[2]; H. L. Fraser[1]; M. J. Mills[1]; [1]Ohio State University, Matls. Sci. & Engrg. Dept., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, 2230 10th St., Wright Patterson AFB, OH 45433 USA

Using current micromechanical fabrication and testing methods, it is now possible to systematically explore size effects related to mechanical properties, i.e., changes in mechanical response due simply to shrinking the physical size of the deforming volume. Interesting new size effects have been discovered in the uniaxial compression testing of micron-size single-crystal samples for both pure Ni and Ni_3Al alloys, where for the latter the flow stress has been observed to increase in proportion to the inverse-square-root of the sample diameter. The size effects in Ni are not as dramatic, but there is a distinct transition in mechanical properties as the sample size shrinks below 10 microns in diameter. However, to date there is still not a connection between these size effects and observable changes to the dislocation substructure, namely because of the challenges in preparing transmission electron microscope (TEM) foils from miniature test specimens. In this study, we present the results of TEM analysis of deformed microsamples in the aforementioned materials, in order to gain a better understanding of the effect that artificially truncating the sample volume has on the fundamental micromechanisms of dislocation glide, storage, and multiplication.

9:55 AM Invited
Grain Boundary Contributions to Deformation and Solid-State Flow in Severe Plastic Deformation: *Erika V. Esquivel*[1]; L. E. Murr[1]; [1]University of Texas, Metallurgl. & Matls. Engrg., 500 W. Univ. Ave., El Paso, TX 79968 USA

More than 40 years ago, Professor Jim Li put forth the concept of grain boundary ledges as sources for dislocations. Since then, there have been numerous confirmations of grain boundary dislocation sources, consistent with the fundamental features of interfaces, and we will demonstrate these observations in deformed metals and alloys observed in the TEM after specific levels of plastic strain. More recently, Professor Li has proposed a new mechanism for severe plastic deformation, or superplastic flow beyond the normal realm of plasticity which involves a so called "interfacial fluid" that allows for continuous, solid-state flow accommodating large plastic strains, and even very high strain rates. Some interesting examples of severe plastic

deformation illustrating these issues, and the connectedness to the incipient and normal plastic regime (the stress-stain diagram) will be presented by examining high velocity and hypervelocity impact craters in polycrystalline metals and alloys. These exhibit varying degr ees of plastic strain and associated microstructures evolving from dislocation emission and interaction, and the actual plastic flow and jetting of solid-state material from the crater rims, which are shown to be arrays of adiabatic shear bands composed of dynamically recrystallized grains. The concept of an "interfacial fluid" will be examined considering dense dislocation arrays in the grain boundary phase regime utilizing the amorphous dislocation lattice concept also originated by Professor Li several decades ago.

10:15 AM Break

10:20 AM Invited
Dislocation Pile-Ups: From {110} Cracking in MgO to Modern Theoretical Strength Considerations: *Ronald W. Armstrong*[1]; [1]University of Maryland, Mechl. Engrg., College Park, MD 20742 USA

Early researches of Professor J.C.M. Li and colleagues dealt with modeling a secondary role of mutually-blocked dislocation pile-ups in producing otherwise unexpected {110} cracks at diamond pyramid hardness indentations put into {001} MgO crystal surfaces. Continuing research has shown that the surface displacements at such indentations are uniquely tied to the primary dislocation motions. But it is the cumulative dislocation interactions in the pile-ups, say, as revealed in x-ray diffraction topographs, that establish the local build-up of stress concentrations magnifying the applied loading forces. In polycrystals, there are analogous grain boundary blockages of the egress of slip or twinning deformation system displacements leading to quantitative description of an inverse square root of grain diameter dependence of the applied stress for cleavage fracturing and, also, a similar dependence for the yield and plastic flow stress; that is, the so-called Hall-Petch (H-P) dependencies for these respective stresses. In this case, Li and Chou provided a classic paper assessing the theoretical role of dislocation pile-ups in the flow stress/grain size relationships, covering analytical and numerical evaluations of dislocation pile-up properties, whether modeled as the real discrete entities or in terms of continuous distributions of dislocations with infinitesimal dislocation Burgers vectors. The latter consideration is critical in establishing the essential equivalence of a dislocation pile-up and a shear crack, as described on a continuum mechanics basis. Thus, there is connection between the Griffith-Irwin-Orowan fracture mechanics (FM) description of precracked polycrystal properties and the H-P dependence, at least, for cleavage, or reasonably brittle, fracturing. Added complication occurs, therefore, when both FM and H-P dependencies are coupled in determining the fracturing properties of real materials. One beneficial consideration is that effective grain size refinement can be understood to provide both an increase in yield strength and greater fracture toughness of a polycrystalline material. Lastly, there is the rather direct consideration of how far one can go in strengthening a material by reducing its grain size. Here, Li and Liu have modeled the behavior of circular dislocation pile-ups involving small numbers of dislocations that transition, in the limit, to the stress requirement for one dislocation loop expanding against the grain boundary resistance. And, in this case, near theoretical limiting strength levels are predicted for crack-free polycrystals unless other grain boundary weakening mechanisms come into play.

10:40 AM Invited
Dislocation Density Model for the Effect of Grain Size on the Flow Stress of a Ti-15.2 at.% Mo β-Alloy at 4.2-650 K: *Hans Conrad*[1]; Kai Wang[1]; [1]North Carolina State University, Matls. Sci. & Engrg., Raleigh, NC 27695-7907 USA

The effect of grain size d (4.6-22 μm) on flow stress σ and dislocation density ρ of a Ti-15.2 at.% Mo β-alloy was determined at 4.2-650 K. The grain size dependence of the flow stress was given by the Hall-Petch equation. Moreover, the dislocation density increased with increase in strain and decrease in grain size. The effect of grain size on the flow stress in terms of the dislocation density was given by $\sigma=\sigma_0(T)+C\rho^{1/2}$ where C was relatively independent of strain, grain size and temperature. The results are discussed in terms of the mechanisms for the effects of grain size and temperature on the flow stress.

11:00 AM Invited
Boundary Strengthening in Undeformed and Deformed Polycrystals: *Niels Hansen*[1]; [1]Risoe National Laboratory, Matls. Rsch. Dept., Ctr. for Fundamental Rsch.: Metal Struct. in 4-D, Roskilde DK-4000 Denmark

The Hall-Petch relation between the stress and the grain size is discussed separately for the yield stress of undeformed polycrystalline metals and for the flow stress of deformed metals. In the former case the grain size is the key structural parameter whereas in the latter structural parameters characterizing the dislocation structure must also be taken into account. An analysis of experimental data supports the Hall-Petch relation for undeformd metals over a grain size range from about 20 nanometers to hundreds of micrometers. For deformed metals where the microstructure evolves with increasing strain the strength of boundaries is not a constant and the Hall-Petch relation must be modified. To rationalize the observed behaviour different mechanisms for strengthening by high angle boundaries and dislocation boundaries will be discussed.

11:20 AM Invited
Flow Processes in Superplastic Yttria Stabilized Zirconia - A Deformation Limit Diagram: *N. Balasubramanian*[1]; Terence G. Langdon[2]; [1]R V College of Engineering, All India Council of Techl. Educ., Mysore Rd., Bangalore 560 059 India; [2]University of Southern California, Aeros. & Mechl. Engrg. & Matls. Sci., Olin Hall of Engrg., Rm. 430G, 3650 McClintock Ave., Los Angeles, CA 90089-1453 USA

The possible rate controlling mechanisms in the deformation of superplastic 3Y-TZP are considered. Several attempts have shown that intragranular dislocations have limited role at stresses below 100 MPa at temperatures 1573 to 1873 K, as the stress is too low to generate them. A single rate controlling mechanism is proposed consistent with a single activation energy observed. This is the interface-reaction controlled grain boundary diffusion creep. An equation derived on the basis of the above mechanism fits the data obtained by different investigators on the stress dependence of strain rate within a factor of 10. A deformation limit diagram is constructed to show that different regions with specific combinations of stress and grain size exponents are limiting cases of one and the same mechanism discussed above.

11:40 AM Invited
Influence of Microstructures on Deformation Behavior of Silicon Single Crystals: *Subhash Mahajan*[1]; [1]Arizona State University, Cheml. & Matls. Engrg., PO Box 876006, Tempe, AZ 85287-6006 USA

We have investigated the deformation behavior of as-grown float zone (FZ) and Czochralski (CZ) silicon single crystals. The CZ crystals contain oxygen, and this is above the solubility limit. Thus, by annealing microstructures of CZ crystals can be modified by precipitating oxygen. The as-grown FZ and CZ crystals exhibit pronounced yield drops and Lüders bands at 700°C. The magnitude of the yield drop decreases as the deformation temperature increases. The yield drops seen in the CZ crystals at 700°C can also be eliminated by annealing them prior to testing. In addition, Lüders front has well defined crystallography and its propagation can be likened to stress-induced migration of small angle tilt boundaries. Arguments will be developed to rationalize these observations.

12:00 PM Invited
Extended Planar Dislocation Boundaries in Metals Subjected to Plane Strain Deformation: *Xiaoxu Huang*[1]; John A. Wert[1]; [1]Risoe National Laboratory, Matls. Rsch. Dept., Ctr. for Fundamental Rsch.: Metal Struct. in 4-D, Roskilde DK-4000 Denmark

During plastic deformation of single crystals, various kinds of deformation bands are developed due to the localization of deformation on different length scales. In the case of plane strain deformation, the concept of equivalent slip systems has been proposed to simplify the mechanical analysis of the macroscopic crystal rotation associated with the deformation banding. A physical manifestation of equivalent slip systems has been found in the coincidence between the equivalent slip plane and the planar dislocation boundary planes experimentally observed in single crystals with high-symmetry crystal orientation subjected to plan strain deformation. In this work similar mechanical analysis is made for warm rolled interstitial steel to understand the correlation between the extended planar dislocation boundary planes and the active slip systems.

12:20 PM
Plastic Deformation Mechanisms of Pure Metals at Low Homologous Temperatures: *Chen-Ming Kuo*[1]; [1]I-Shou University, Dept. of Mechl. Engrg., 1, Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Plastic deformation mechanisms of pure metals at low homologous temperatures are attributed to the motion of dislocations and their interactions with each other or other kinds of obstacles. Physically based modeling of these mechanisms is generally considered as thermally activated motion of dislocations past obstacles and structural evolution of the obstacles. In this study, stress rate change experiments, which give rapid stress changes, are conducted using pure copper at room temperature. Incremental method is employed in the

numerical simulation of above modeling. Excellent agreement is observed in the comparison between experimental data and numerical calculations. Sensitivity analyses are also performed to better understand both flow kinetics and structural evolution law. Simple kinetic model such as the regular distributed rectangular obstacle is adequate enough. Obstacle structure is strongly influenced by the dislocation annihilation processes.

Microstructural Processes in Irradiated Materials: Modelling Defect Evolution

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Monday AM Room: 3011
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Maria Jose Caturla, Universidad de Alicante, Fisica Aplicada, Alicante E-03690 Spain; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, LMPGM, Villeneuve d'Ascq Cedex 59700 France

8:30 AM Invited
Atomistic Modelling of Point Defects and Impurities in Steels: *Graeme J. Ackland*[1]; [1]University of Edinburgh, Sch. of Physics, 5411 JCMB, Kings Bldgs., Edinburgh, Scotland EH9 2LZ UK

Results are presented for computer simulations of the behaviour of defects and impurities in alpha-iron under irradiation. Ab initio simulations are used to provide data for parameterisation of new interactomic potentials for iron with dilute impurities of Cu, P, Al and C. Molecular dynamics calculation of point defects and their interaction with impurities are presented. From these we attempt to determine coarse-grained objects (defect-impurity clusters) suitable for use as the fundamental objects in defect-dynamics calculations (e.g. kinetic Monte Carlo, dislocation dynamics). The stability/mobility of these emergent objects is checked by subsequent ab initio calculation such that the molecular dynamics/ab initio step of multiscale modelling is done self-consistently.

9:10 AM
Multiscale Modeling of Defect Kinetics in Electron Irradiated Iron: *Chu Chun Fu*[1]; Jacque Dalla Torre[1]; Francois Willaime[1]; Jean-Louis Bocquet[1]; Alain Barbu[1]; [1]CEA/Saclay, SRMP, Gif sur Yvette 91191 France

Changes in microstructure of nuclear materials are governed by the kinetics of defects produced by irradiation. The population of vacancies, interstitials, and their clusters can however be followed only indirectly, e.g. by macroscopic resistivity measurements. The information on the mobility, recombination, clustering or dissociation of defects provided by such experiments is both extremely rich and difficult to interpret. By combining ab initio and kinetic Monte Carlo methods, we successfully reproduce the abrupt resistivity changes — so called recovery stages — observed upon annealing after electron irradiation in pure iron. New features in the mechanisms responsible for these stages are revealed. We show that small vacancy clusters and tri-interstitials contribute to the stages attributed to mono-vacancy and di-interstitial migration respectively, predict the effect of the unexpected low migration barriers found for tri- and quadri-vacancies. The influence of carbon atoms on the recovery stages are also discussed.

9:30 AM
Weak-Beam Imaging of Small Point-Defect Clusters Using the Howie-Basinski Equations and the Multi-Slice Method: Z. Zhou[1]; S. L. Dudarev[2]; *M. L. Jenkins*[1]; A. P. Sutton[1]; M. A. Kirk[3]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK; [2]EURATOM/UKAEA Fusion Association, Theory & Modlg. Dept., Culham Sci. Ctr., Oxfordshire OX14 3DB UK; [3]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439 USA

Nanometer-sized dislocation loops and other small point-defect clusters are commonly found in irradiated materials. Complete characterisation of such defects often is very difficult. They are usually investigated by using diffraction contrast images produced by conventional transmission electron microscopy (TEM), particularly under weak-beam conditions which offer improved contrast and resolution compared with other imaging conditions. Image simulations obtained under the same conditions are necessary for a full analysis of such images. Such simulations may be carried out by solving the equations of dynamical diffraction theory. In the present work we have employed the Howie-Basinski equations rather than the usual Howie-Whelan equations, since weak-beam images of very small clusters are better simulated without the use of the column approximation. In simulations of this kind it is usual to obtain the displacement fields or strain fields by elasticity theory. It is well known that this method has limitations in the core region of defects, due to the inaccuracy of elasticity theory in this region. This disadvantage of the Howie-Basinski approach can be overcome by using strain fields calculated from atomistic configurations of small defects. The atomistic structures were obtained by conjugate-gradient (CG) energy minimisation. Simulations were carried out for dislocation loops and for stacking-fault tetrahedra. Weak-beam images were calculated using the Howie-Basinski approach and the multi-slice method for the same atomic structures to compare the capabilities of the two methods in TEM weak-beam imaging. The advantages and disadvantages of the two approaches will be discussed and comparisons made with experimental images.

9:50 AM
Effect of the Interatomic Potentials on Point-Defect Clustered Fraction in Molecular Dynamics Displacement Cascades Simulated in bcc-Fe: *Dmitry A. Terentyev*[1]; Christina Lagerstedt[2]; Pär Olsson[3]; Kai Nordlund[4]; Janne Wallenius[2]; Lorenzo Malerba[1]; [1]SCK-CEN, RMO, Boeretang 200, Mol 2400 Belgium; [2]Royal Institute of Technology, Dept. of Nucl. & Reactor Physics, Roslagstullsbacken 21, Stockholm SE-106 91 Sweden; [3]Uppsala University, Dept. of Neutron Rsch., Ångstrom Lab., Box 525, Uppsala SE-751 20 Sweden; [4]University of Helsinki, Accelerator Lab., POB 43 (Pietari Kalmin katu 2), Helsinki 00014 Finland

Depending on the interatomic potential used for molecular dynamics simulation of displacement cascades in bcc-Fe, primary states of damage characterised by different spatial distributions of point-defects (average clustered fractions) are obtained. These differences may influence the long-term microstructural evolution predicted by, e.g., kinetic Monte Carlo codes. In this work, a number of displacement cascades initiated by recoils of 5, 10 and 20 keV have been simulated using four different interatomic potentials for á-Fe, each providing, among other things, different descriptions of self-interstitial behaviour in this metal. The corresponding defect clustered fractions (for both self-interstitial atoms and vacancies) have been studied using the same criteria for cluster definition and compared. The outcome is discussed in terms of how the point-defect description provided by the potential used may influence the predicted primary state of damage.

10:10 AM Break

10:40 AM Invited
Ab Initio Modeling of Defect Properties With Substitutional and Interstitial Elements in Steels and Zr Alloys: *Christophe Domain*[1]; [1]EDF R&D, MMC, Les Renardières, Moret sur Loing F-77250 France

In steels, the constituting elements interact with each other as well as with point defects. In Zr alloys, in addition to these interactions, hydrogen pick up modifies the mechanical properties. The microstructure properties are directly linked to the chemical interactions between the different constituting elements, and the point defects or the extended defects (stacking faults, dislocation). All these atomic scale interactions govern the elementary mechanisms ruling the kinetics of the system, and are thus among the key parameters needed to model the evolution of the microstructure under ageing or irradiation. Ab initio methods allow to determine important quantities such as defect formation, binding or migration energies. Data on interstitial point defects (C, N, P) in Fe and H in Zr as well as on substitutional transition metal elements (Cu, Ni, Mn, Si, Cr, ...) interaction in bcc Fe will be presented and discussed. When available comparison with experimental data will be made in order to check the resul ts and in fine to predict unknown quantities. The link between the obtained atomic quantities and the related consequences on the macroscopic properties will be discussed.

11:20 AM
Point Defect Dynamics in Irradiated bcc Metals: *Joerg Rottler*[1]; David J. Srolovitz[1]; Roberto Car[1]; [1]Princeton University, PRISM, 70 Prospect Ave., Princeton, NJ 08544 USA

The macroscopic mechanical properties of irradiated metals are intimately related to their microstructural features and their spatiotemporal evolution. We present a statistical model for the dynamics of point defects in bcc metals that is solved through kinetic Monte Carlo (kMC) and rate equations. Self-interstitial atoms and vacancies can be produced in abundance upon irradiation with energetic particles, but they subsequently anneal due to recombination and absorption at sinks such as dislocations and grain boundaries. The model reveals a sequence of kinetic regimes that lead to a final steady state. We also determine the size distribution of voids that form when vacancies aggregate into cluster. The model is then extended to include long ranged elastic interactions between point defects and dislocations, which have so far mostly been ignored in kMC simulations of microstructural evolution. We also consider metal alloys, where interstitial and vacancy transport must be described through effective diffusivities and correlation effects become important.

11:40 AM
Kinetic Monte Carlo Simulation of Irradiation Effects in bcc Fe-Cu Alloys: Experimental Validation: *Lorenzo Malerba*[1]; Christophe Domain[2]; Charlotte S. Becquart[3]; Abderrahim Almazouzi[1]; [1]SCK CEN Nuclear Research Centre, Reactor Matls. Rsch. Unit, Boeretang 200, Mol B-2400 Belgium; [2]EDF R&D, Dept. Materiaux et Mecanique des Composants, Les Renardieres, Moret sur Loing F-77250 France; [3]USTL, Lab. de Métallurgie Physique et Génie des Matériaux, UMR 8517, Bat C6, Villeneuve d'Ascq F-59655 France

Iron-copper alloys are model materials for reactor pressure vessel (RPV) steels and are typically used in modelling-oriented experiments to gain insight into the basic mechanisms of steel embrittlement via copper precipitation and matrix damage formation. In this work a selection of positron annihilation and atom probe irradiation experiments on low-concentration Fe-Cu alloys available from the literature, complemented by dedicated thermal annealing experiments on Fe-1%Cu, is used as a reference to test the validity of a mesoscopic kinetic Monte Carlo model. In this model, the binding energies of copper-vacancy complexes (CuVC), which are among the main parameters governing the nucleation and growth of copper precipitates, are described using laws obtained from a detailed study of a large number of CuVC. In addition, a state-of-the-art description of SIA and SIA cluster mobility in Fe is applied. The validity of the model is assessed and possible further improvements of the model are discussed, based on the feedback from the experimental validation.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Analysis of Interdiffusion Mircrostructures: Session I

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Monday AM — Room: 3007
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Carelyn E. Campbell, National Institute of Standards and Technology, Metall., Gaithersburg, MD 20899-8555 USA; Samuel M. Allen, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Diffusion Paths and Interdiffusion Microstructures: Applications and Remaining Challenges: *J. E. Morral*[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

Interdiffusion can influence the properties and cost of products that experience high temperature during processing or service (e.g. coated turbine blades, solid oxide fuel cells, carburized sun gears, etc.). Accordingly the ability to predict interdiffusion microstructures can be a useful alloy design tool. Software such as DICTRA can predict interdiffusion microstructures via diffusion paths and phase diagrams. Although useful for certain applications, both DICTRA and the theory of diffusion paths have serious limitations. Software for the Phase Field Method (PFM) requires more computer time, but has fewer limitations and can predict microstructures directly. Also the PFM takes into account both precipitate morphology and diffusion in precipitates, which yields a more accurate result. However without adequate databases and a better understanding of interdiffusion fundamentals, the value of these programs to alloy design will be limited.

9:15 AM Invited
Canonical Phase Diagrams: *John W. Cahn*[1]; [1]National Institute of Standards and Technology, MSEL, 100 Bureau Dr., Gaithersburg, MD 20899-8555 USA

The molar free energy of a solution F and its various derivatives with respect to concentrations can be used instead of the concentrations as the axes of phase diagrams. Such axes were explored by John Morral, in his paper "On Characterizing Stability Limits for Ternary Systems" (Acta Metall, 1972, 20, 1061). He found that when phase diagrams are plotted with second derivatives of F as axes instead of with the usual temperature and concentration axes, all regular solutions phase diagrams are distorted into triangular pyramids on a universal canonical phase diagram, in which stability limits are depicted as a double cone. One cone marks the stability limit with respect to continuous ordering and the other cone the stability limit with respect to spinodal decomposition. A solution's regular solution interaction parameters determines placement of each triangular pyramid phase diagram with respect to the double cone. This talk will explore some other choices of thermodynamics functions as axes. Phase diagram plots using axes of chemical potentials instead of concentration, as suggested by Scatchard and Pelton, are useful. Using derivatives of F w.r. to order parameters as axes gives canonical phase diagrams and insight into complex ordering, such as for Cu-Au, involving first-order phase transitions and more than one ordered phase.

9:45 AM Invited
Application of Diffusion Path Analysis in Determination of Phase Formation and Phase Diagrams: *J.-C. Zhao*[1]; [1]GE Global Research, One Rsch. Cir., K1-MB239, Niskayuna, NY 12309 USA

This presentation will give several examples to illustrate the applicability of diffusion path analysis in determination of phase formation in multicomponent diffusion couples. Very often regular plots of composition vs distance are not the most useful ones in understanding the formation of phases. The composition against composition plots are far more informative, especially when coupled with calculated multicomponent isothermal sections. In the latter case, it is important to select right elements to plot against one another to give the most valuable information. The composition against composition plots are also extremely useful for determination of phase diagrams using diffusion couples and diffusion multiples. Examples will be given for several ternary systems where the diffusion path analysis is essential for correct interpretation of the phase equilibrium data.

10:15 AM Break

10:30 AM Invited
Observations and Analysis of Interdiffusion Phenomena in Selected Multicomponent Systems: *Mysore A. Dayananda*[1]; [1]Purdue University, Sch. of Matls. Engrg., 501 Northwestern Ave., W. Lafayette, IN 47907 USA

Interdiffusion in multicomponent systems is reviewed with emphasis on the determination of interdiffusion fluxes and assessment of diffusional interactions among the components. The phenomena of uphill diffusion, zero-flux planes, diffusional instability at interfaces and microstructural evolution are illustrated with single phase and multiphase diffusion couples investigated in selected multicomponent systems. Uncommon diffusion paths and diffusion structures experimentally observed for single phase and multiphase diffusion couples in Cu-based and Fe-based ternary systems and ternary aluminides and silicides are presented and discussed in the light of diffusion paths, variation of diffusion coefficients with composition and grain boundary contributions to diffusion. The determination of interdiffusion coefficients from individual multicomponent diffusion couples and the generation of their concentration profiles from the calculated interdiffusion coefficients are also illustrated with the MultiDiflux program currently being developed. The research is supported by the National Science Foundation.

11:00 AM
Assessment of Analytical Methods for the Determination of Composition-Dependent Interdiffusion Coefficients in Ternary

Alloys: *Narayana Garimella*[1]; Abby Puccio[1]; Yongho Sohn[1]; [1]University of Central Florida, AMPAC & MMAE, Box 162455, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA

Understanding of concentration profiles and/or interdiffusion fluxes that develop in a ternary diffusion couple during an isothermal anneal generally entails the determination of interdiffusion coefficients based on extended Fick's law based on Onsager's formalism. The description of ternary diffusion requires four concentration-dependent interdiffusion coefficients, two main and two cross-coefficients. Several techniques including those based on Boltzmann-Matano analysis, and average interdiffusion coeffficients are available to analyze concentration profiles for the determination of ternary interdiffusion coefficients. Governing principles on these analytical methods are presented with concentration profiles obtained from selected isothermal diffusion studies in the alpha-phase (fcc) of Cu-Ni-Zn system at 775°C. Ternary interdiffusion coefficients determined based on these techniques are compared and complimented for the assessment of diffusional behavior and interactions in the Cu-Ni-Zn system at 775°C.

11:25 AM Invited
Microstructural Evolution in Interdiffusion Zones and its Effect on Diffusion Paths: Kaisheng Wu[2]; John E. Morral[1]; *Yunzhi Wang*[1]; [1]Ohio State University, Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA; [2]University of Wisconsin, Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

The phase field method is being used to study explicitly the microstructural evolution that occurs in interdiffusion zones and its effect on diffusion paths. In this presentation we will discuss simulation results obtained from single-phase/two-phase and two-phase/two-phase diffusion couples of Ni-Al-Cr alloys. Free energy and mobility data of the system were obtained from CALPHAD databases. The simulated microstructural features and kinetics of boundary migration were compared with experimental observations. Also, diffusion paths calculated from the phase field simulations were compared with analytical and DICTRA predictions. The Kirkendall effect, Gibbs-Thompson effect, and the effect of nucleation on microstructural evolution and diffusion path shape will be addressed. This work is supported by the National Science Foundation.

11:55 AM
Trans-Interface Diffusion-Controlled Coarsening: *A. J. Ardell*[1]; V. Ozolins[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095-1595 USA

We present a model of coarsening controlled by diffusion through the coherent interface between an ordered precipitate and a disordered matrix. Since chemical diffusion in ordered phases is much slower than in disordered phases, the interface is a diffusion bottleneck. The new theory predicts the following: The average radius squared increases linearly with time; Solute is depleted linearly with the inverse square-root of time; A scaled particle size distribution (PSD) that is much broader than the PSD of the LSW theory; The coarsening rates are completely independent of volume fraction. Published data on the coarsening of γ' (Ni_3Al) precipitates agree quite well with the new theory, which also predicts that coarsening of disordered γ (Ni-Al solid solution) precipitates in a γ' matrix should obey traditional LSW kinetics and be strongly volume-fraction dependent, as observed experimentally. No other theory is consistent with the totality of these experimental results.

Neutron Diffraction Characterization of Mechanical Behavior: Facilities, Techniques, and Capabilities

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Monday AM Room: 3004
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Hahn Choo, University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA; Peter K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA

8:30 AM Invited
An Overview of the Spallation Neutron Source: *Ian S. Anderson*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, PO Box 2008, Oak Ridge, TN 37831-6474 USA

The Spallation Neutron Source is an accelerator-based neutron source being built in Oak Ridge, Tennessee. When completed in 2006 SNS will provide the most intense pulsed neutron beams in the world for scientific research and industrial development. The SNS is being built by a partnership of six DOE laboratories, and like all DOE facilities will be operated as a user facility, open to scientists and engineers from universities, industry, and government laboratories in the United States and abroad. Three cryogenic and one ambient water moderator will serve up to 24 instruments, 16 of which have been approved and are in various stages of construction. All instruments at SNS have been designed to be best in class and will provide unprecedented opportunities to explore the structure and dynamics of novel materials.

8:50 AM Invited
Compression Loading and Neutron Diffraction at Cryogenic Temperatures: *Mark A.M. Bourke*[1]; Don W. Brown[1]; Hahn Choo[2]; Bjorn Clausen[1]; Veronica Livescu[1]; Thomas A. Sisneros[1]; Raj Vaidyanathan[3]; [1]Los Alamos National Laboratory, LANSCE-12, Los Alamos, NM 87545 USA; [2]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [3]University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA

A recent addition to the SMARTS instrument at the Los Alamos Neutron Science Center is an in situ cryogenic deformation capability. The capability allows neutron diffraction spectra to be recorded under uniaxial compression. This complements existing high (up to 1800K) temperature loading capability and has opened new areas of study. Current measurements have been performed at 200K but measurements at 150K are planned before the TMS '05 meeting. The design and modifications to the SMARTS load frame will be described. Preliminary results on transformations in R phase NiTi, twinning in Zirconium and strain induced martensite in steel will be presented to illustrate the potential of the new capability.

9:10 AM
In Situ Measurement of Texture and Elastic Strains with HIPPO-CRATES: *Sven C. Vogel*[1]; Christian Hartig[2]; Heinrich Mecking[2]; [1]Los Alamos National Laboratory, LANSCE-12, MS H805, PO Box 1663, Los Alamos, NM 87545 USA; [2]Technische Universität Hamburg-Harburg, Werkstoffphysik & Werkstofftechnologie, Eißendorfer Straße 42, 21073 Hamburg Germany

Neutron diffraction has been used to determine the elastic strains and texture evolution during plastic deformation of two materials: A two phase, powder-metallurgically (PM) produced 33vol.%Fe-67vol.%Cu alloy, and the Magnesium alloy AZ31. These were the first experiments with the new load-frame CRATES for the neutron diffractometer HIPPO at LANSCE. The unique HIPPO-CRATES combination allows deformation studies in-situ by following both lattice strain and texture changes in 20 sample directions per sample orientation. Both materials were tested in uni-axial compression and tension loading cycles up to a maximum plastic strain of 6.4 % (Fe-Cu) and 2.1 % (Mg-AZ31). Several neutron time-of-flight diffraction patterns were collected during the deformation, yielding information from many crystal lattice planes simultaneously. In the case of the Fe-Cu alloy

results were obtained elucidating the stress distribution during the plastic co-deformation of two ductile phases with very different yield stresses. Yield stresses of ~100 MPa and ~400 MPa were measured for the pure PM produced copper and iron materials, respectively. The evolution of the elastic strains in Fe and Cu is compared with FEM calculations for both phases based on input parameters obtained from macroscopic stress-strain curves of the individual materials. The variation of elastic strains and texture in the Magnesium alloy gives clear evidence about the activation of tensile twins at an early stage of plastic deformation.

9:30 AM
In-Situ Deformation of Cu Mosaic Crystals by TOF Neutron Transmission: *Javier Roberto Santisteban*[1]; [1]Open University, Matls. Engrg., Walton Hall, Milton Keynes MK2 2UT UK

We introduce the basic features of the neutron transmission by mosaic crystals through demonstration experiments comprising in-situ loading tests on plastically deformed Cu crystals, originally used as neutron monochromators. We show that these experiments can be used to monitor the variation in orientation, mosaic spread and interplanar distances, that occur as a result of elastic or plastic deformation. Distinctive features of this technique are the ability to study many crystal reflections simultaneously, together with the possibility of 2D spatial resolution. In its present state, it is possible to study the spatial distribution of a specimen's deformation in-situ over an area of 25x25mm2 with 2mm spatial resolution. We report the anisotropic evolution of the mosaic spread of a crystal as a result of plastic deformation. We present the dependence of the mosaic spread on the macroscopic shear strain and applied stress, as well as its spatial variation across the specimen.

9:50 AM
The TOF Strain Scanner POLDI with Multiple Frame Overlap - Concept and Performance: Uwe Stuhr[1]; Mirco Grosse[1]; *Werner Wagner*[1]; [1]Paul-Scherrer-Institute, Spallation Neutron Source Div., Villigen PSI 5232 Switzerland

POLDI is a new neutron diffraction instrument at the Swiss continuous spallation neutron source SINQ at PSI, primarily dedicated to residual stress investigations. The instrument has realized a novel type of time-of-flight (TOF) diffractometer allowing multiple frame overlap. Besides for the benefit of higher intensity (as compared to a conventional single-pulse mode), this concept allows an independent tuning of intensity and resolution, an immense advantage for a flexible adjustment to dedicated experiments. The concept makes use of the mutual dependence of the neutron's arrival time at the detector and the related scattering angle to assign the neutrons to the originating pulse at the chopper. The principle concept of the instrument, some special components, and the performance in terms of intensity, resolution and flexibility for strain mapping and structure analysis will be presented.

10:10 AM Break

10:30 AM Invited
Industrial Application of Residual Strain Measurement by Neutron in Japan: *Yukio Morii*[1]; [1]Japan Atomic Energy Research Institute, Neutron Sci. Rsch. Ctr., Tokai, Ibaraki 319-1195 Japan

The instrumental specifications and performance of the neutron diffractometer for Residual Stress Analysis, RESA, installed at JRR-3 of JAERI will be briefly introduced. New method of residual stress analysis, which does not require measurements of stress-free lattice space d0(hkl), was proposed and applied to evaluate the residual stress distribution in a welded sample. Preliminary results of the analysis will be discussed. Number of industrial application of neutron residual strain measurement is increasing in Japan. A few of the recent measurements will be introduced. The status of constructions of Japan Proton Accelerator Research Complex, J-PARC, and a Pulsed neutron diffractometer for Residual Stress Analysis, RESA-P, is briefly reported, with the activities supported by the local government to promote industrial research using neutron beam.

10:50 AM Invited
Neutrons at Work for Industry: *Ronald B. Rogge*[1]; [1]National Research Council, Canada, Neutron Prog. for Matls. Rsch., Chalk River Labs., Bldg. 459, Chalk River, Ontario K0J 1J0 Canada

In general diffraction is a powerful technique for gaining an understanding of the behaviour of materials. When combined with the high penetration of neutrons one has access to a uniquely powerful and non-destructive probe. Other presentations in this session show, for example, that in-situ loading experiments have revealed much about deformation in materials. It is also true that neutrons provide an excellent tool for evaluating and verifying real industrial processes and/or fabrication routes. This presentation will focus on examples in which neutrons have been put to work for industry, simulating realistic conditions, contributing to failure analysis, verifying the effectiveness of a processing steps, and dealing with regulatory bodies.

11:10 AM Invited
Neutron Strain Scanning: Going Beyond Present Limitations: *T. Pirling*[1]; [1]Institut Laue-Langevin, Diffraction Grp., 6 rue Jules Horowitz, F-38042 Grenoble France

Neutron strain scanning is nowadays a well-established method for the nondestructive determination of residual stresses in "real" engineering components. A big advantage of neutron strain-scanning are the high penetration power and the 90 degrees measuring geometry which allows, in almost all types of samples, measurements in all orientations necessary to determine the strain tensor. ILL has undertaken developments to improve resolution and utility of strain scanning instrumentation. These ideas are now part of the new instrument SALSA (Strain Analyser for Large and Small Applications) on which first tests are foreseen for August 2004. The construction of the instrument is a collaboration with the University of Manchester and partially founded by the EPSRC. Many instruments use slit apertures for defining the gauge volume. They have high transmission, but the inconvenience that they have to be placed as close as possible to the gauge volume. Measurements in big samples therefore show low lateral resolution be cause the gauge size increases with distance from the gauge volume due to the unavoidable beam divergence. This effect increases when focusing monochromators are used. However double focussing bent crystal monochromators seems to be most efficient for monochromatic strain scanning instrumentation. They take advantage of the divergence of the incident neutron beam and focus it to the gauge volume dimensions, without reducing the angular resolution of the instrument thanks to phase space focusing. The concept at ILL is to use radial focusing collimators for the beam definition, which takes advantage of the beam divergence and at the same time defines a precise gauge volume. Compared to using a slit, the distance to the gauge volume is several times higher. This enables measurements in big components with good precision. Another positive effect of the collimators is the reduction of the surface effect, which leads to huge errors in near surface or interface measurements. Using the collimator set-up, precise meas urements within twenty to fifty micrometres to an interface are possible, as well as measurements on samples that are thinner than the dimension of the gauge volume. Another limiting factor of instrumentation can be the sample alignment and positioning. The various, often complicated shapes of samples need a sample stage that is adaptable to all the different requirements. ILL has, in further collaboration with INRIA (France) and the manufacturer OHE (Switzerland), developed a design for a Stewart Platform (or hexapod) as sample stage. It allows tilt, rotation, oscillation and translation of the sample and scans along any trajectory in space. Its load capacity is more than 500 kg and samples up to 1.4 m in length can be mounted. The positioning accuracy lies in the range of 10 micrometres. The paper presents high resolution measurements mostly on aerospace applications and a short presentation of the new instrument SALSA.

11:30 AM Invited
Positron Annihilation as an Addidional Source of Information About Plastic Deformation in Structural Materials: *Hans Georg Priesmeyer*[1]; [1]Kiel University, Lab. for Applied Neutron Physics, c/o GKSS Rsch. Ctr., Max-Planck-Str.1, Geesthacht D-21502 Germany

During strain measurements by neutron diffraction additional information is available through simultaneous gamma-ray spectroscopy. Neutron absorption is always a competing process to neutron scattering. It leads to the emission of high-energy prompt gamma radiation, which in turn produces positrons within the bulk of the specimen. The subsequent decay of these so called antiparticles and the resulting annihilation radiation are influenced by material properties like defects. The interaction of positrons with electrons has in recent years become a successful technique to measure the momenta of electrons in solids. These are influenced by crystalline defects, such as dislocations created by plastic deformation. Experimentally, this leads to an increase in the mosaic spread of the BRAGG reflections for elastically scattered neutrons as well as changes of the shape of the 511 keV line of the positron annihilation radiation. Suitable collimation can assure that the information gathered by neutron diffraction and from positron annihilation comes from the same gauge volume. Correlation between the width of the BRAGG reflections and the S-parameter (describing the shape of the annihilation line) as a function of the degree of plastic deformation has been established. Experimental work of simultaneous on-line investigation of the plastic deformation behaviour of copper and aluminium as model substances is performed at the FRG-I research

reactor at GKSS and will be reviewed. Work funded by DFG Pr 267/13-1 and GKSS Research Centre Geesthacht GmbH.

11:50 AM
Design and Performance of the Second Generation Neutron Residual Stress Mapping Facility (NRSF2) at ORNL: *Camden Richards Hubbard*[1]; Alexandru D. Stoica[1]; Michael C. Wright[1]; Hahn Choo[1]; Stewart Craig[1]; William Bailey[1]; Fei Tang[1]; Ke An[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., MS 6064, Bldg. 4515, Oak Ridge, TN 37831-6064 USA

The NRSF2 design aims to improve the prior system's capabilities. Upgrades include utilizing: (a) the larger beam at HB-2; (b) two multiwafer Si, doubly focusing monochromators (wavelengths from 0.145 nm to 0.227 nm); (c) two high precision goniometers, one is capable of supporting large specimen mapping while the other enables strain tensor studies; and (d) and seven 40 x 100 mm active area 1-D position sensitive detectors. The flux on specimen is predicted to be close to 10**8 neutrons/cm**2/sec. The combined gains indicate approximately a 10-fold improvement in capability. A new load frame for tension/compression studies in the elastic, plastic and failure regimes of most materials has been developed. Environmental cells for use with the load frame for studies of environmental assisted corrosion studies using neutron strain mapping are also available. Results of monochromator, calibration, precision and performance will be reported.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Magnetic and Semiconducting Materials

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Monday AM — Room: 3016
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Douglas J. Swenson, Michigan Technological University, Dept. of Matl. Sci. & Engrg., Houghton, MI 49931 USA; Hyuck Mo Lee, Korea Advanced Institute of Science and Technology, Dept. of Matl. Sci. & Engrg., Taejon 305-701 S. Korea

8:30 AM Invited
Selective Oxidation of Tunnel Barrier Metals in Magnetic Tunnel Junctions: *Y. Austin Chang*[1]; [1]University of Wisconsin, Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

Oxidation of an ultra-thin metal layer (less than 1 nm) to form a quality oxide tunnel barrier is of critical importance in fabricating Magnetic Tunnel Junctions (MTJs) with low product of resistance and area (RA). The ultimate goal in this research is to utilize a scheme to completely oxidize the metal to form its oxide without oxidizing the adjacent ferromagnetic layers in such junctions. Indeed it is extremely difficult if not impossible to achieve this objective using the conventional plasma or air oxidation methods. However, it is possible to use a gas mixture with fixed chemical potential of oxygen in such way that only the tunnel barrier metal is oxidized but not the ferromagnetic layers. In this presentation, I will show experimental results obtained by these students in my group at the University of Wisconsin at Madison in oxidizing metals such as Al, Y etc. These results show that indeed the thin metal layers are completely oxidized but not the adjacent ferromagnetic layers such as CoFe.

9:00 AM Invited
In Situ Ultrahigh Vacuum Transmission Electron Microscope Investigations of Dynamical Changes of Nanostructures on Silicon: *L. J. Chen*[1]; [1]National Tsing Hua University, Matls. Sci. & Engrg., 101, sec. 2, Kuang Fu Rd., Hsinchu 300 Taiwan

In situ ultrahigh vacuum transmission electron microscope is a powerful tool to investigate the dynamic changes of nanostructures on silicon. By observing growth and phase transitions in situ, understanding of their mechanisms can be used to model relevant processes. With the precise knowledge of the changes occurred on an atomic level, accurate control of the growth process can be achieved. The dynamical changes occurred on the nano scale are often unexpected, which also underscores the importance of the approach. In this presentation, we will highlight several examples to demonstrate the unique capability of in situ TEM to study the dynamical changes. The examples include the formation and disintegration of TiSi2 nanowires in Ti/Si system, stacking and vibration of NiSi2 nanodots on silicon, migration of Au "block" on Si bi-crystal and epitaxial growth of beta-FeSi2 on silicon.

9:30 AM
Characterization of the Pd/GaSb Thin Film Reaction for Pd-Based Metallizations to GaSb: Suzanne Mohney[1]; *Joshua Alexander Robinson*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & Matls. Rsch. Inst., 101 Steidle Bldg., Univ. Park, PA 16802 USA

GaSb plays a vital role in thermophotovoltaic, microwave, and optoelectronic devices. Knowledge based on the interaction of a metal and semiconductor, both in the bulk and thin film regime, is often used to engineer contacts to compound semiconductors. To date, information about the behavior of a Pd thin film on GaSb has been limited. In this study, we explore the interaction between a 50 nm layer of Pd and GaSb substrate annealed in the temperature range 100 - 350°C for 10 - 360 min. We report on the formation of Pd-rich nanocrystalline and polycrystalline ternary phases at temperatures below 200°C, followed by Pd-Ga and Pd-Sb binary phases above 300°C. Finally, we describe the development of low resistance ohmic contacts to n-GaSb using Pd as one of the contact metals.

9:50 AM
Self-Assembled Si-Ge Nanostructures on Si(100) and Si(110): *Douglas J. Swenson*[1]; Xurui Deng[1]; [1]Michigan Technological University, Dept. of Matls. Sci. & Engrg., 1400 Townsend Dr., Houghton, MI 49931 USA

Self-assembled, Group IV (Si, Ge, C, Sn) nanostructures exhibit an array of unique electronic and optoelectronic properties. The microstructural evolution of these structures results from the interplay of thermodynamic factors (bulk, interfacial and strain energy) and kinetic factors (e.g., surface diffusivities, material deposition rate). Here, the similarities and differences of microstructural evolution are presented for Si_xGe_{1-x} films grown under similar conditions by molecular beam epitaxy (MBE) on (100) and (110)-oriented Si substrates. In both cases, it is possible to create self-organized pits, decorated by islands of various shapes, under certain growth conditions. However, 2-D film growth is much more persistent on (110) surfaces than it is on (100) surfaces, and upon exceeding a critical film thickness, Si_xGe_{1-x} nanowires, aligned with the single in-plane <110> azimuth are observed. Rationale for these differences in morphology are given primarily in terms of strain energy and surface energy phenomena.

10:10 AM Break

10:30 AM Invited
The Application of the Solid State Electrochemical Method to the Investigation of Electronic Materials: *Krzysztof Fitzner*[1]; [1]AGH University of Science and Technology, Lab. of Physl. Chmst. & Electrochmst., Faculty of Non-Ferrous Metals, 30 Mickiewicza Ave., 30-059 Krakow Poland

Systematic electromotive force (emf) studies were carried out to determine thermodynamic properties of several semiconducting, magneto-optic and superconducting phases. First, electrochemical cells were used in coulometric titration experiments to determine the first order interaction parameters in the liquid III-V-O systems: In-As-O, Ga-As-O, In-P-O and Ga-P-O systems. Second, the stability of rare-earth manganates ($LnMnO_3$, where Ln = Nd, Pr, Sm, Gd, Ho and Yb) was determined using cells with a zirconia solid electrolyte. Finally, by combining the cells with zirconia and CaF_2 electrolytes, Gibbs energy changes of respective reactions of formation of $LnBa_2Cu_3O_7$ and Ln_2BaCuO_5 (where Ln = Yb, Tm, Er, Ho, Dy, Gd) were measured. New thermodynamic data are presented and discussed.

11:00 AM
A Thermodynamic Database for III-N Semiconductors: *Ursula R. Kattner*[1]; Albert V. Davydov[1]; Benjamin P. Burton[2]; [1]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., Stop 8555, Gaithersburg, MD 20899-8555 USA; [2]National Institute of Standards and Technology, Ceram. Div., 100 Bureau Dr., Stop 8520, Gaithersburg, MD 20899-8520 USA

GaN and related nitrides are important materials for photonic and microelectronic devices. Development of optimum growth and thermal processing conditions for the device structures still relies mostly on trial-and-error methods. Knowledge of the thermodynamics of Group III-V systems is not only important for optimizing semiconductor growth/processing conditions but also key for understanding reactions with metal contact materials. Although binary phase diagrams of III-V materials appear to be simple with only one compound at 1:1 stoichiometry, development of thermodynamic descriptions is plagued by a lack of data and large uncertainties in the available data. Available thermochemical and phase diagram experimental data and ab-initio data will be discussed for some constituent binary and ternary systems, and a preliminary thermodynamic database will be presented. This database can be used to define growth and processing conditions of III-N compounds and for description of semiconductor-metal contacts.

11:20 AM
Designing Experimental Determination of Sheet Resistance of a Submicron Titanium Self-Aligned Silicide Formation: *Jau Shiung Fang*[1]; Chen Siang Hsu[1]; [1]National Huwei University of Science and Technology, Dept. of Matls. Sci. & Engrg., 64 Wunhua Rd., Huwei 632, Yunlin 632 Taiwan

The sheet resistance of titanium self-aligned silicide was shown to determine the optimum process of a 0.25 mm polysilicon gate technology. The influence of arsenic doping dosage, the thickness of titanium, the temperature of rapid thermal annealing on the sheet resistance of a polysilicon gate was experimentally analyzed. Experimental results revealed that thickness of titanium, the temperature of RTP-2, and the interaction between the thickness of titanium and the temperature of RTP-2 dominated the sheet resistance of TiSi2. Statistical analysis showed that sheet resistance decreased as titanium thickness increased or RTP-1 temperature increased. An optimum RTP-2 temperature was also required for reducing sheet resistance of TiSi2. A low sheet resistance was yielded for titanium thickness = 32-35 nm, RTP-1 = 720-750°C for 75 sec, and RTP-2 = 860°C for 20 sec.

Phase Transformations Within Small-Size Systems: Thermodynamics, Phase Equilibria and Kinetics

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee
Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Monday AM Room: 3002
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: George Spanos, Naval Research Laboratory, Washington, DC 20375-5000 USA; Robert D. Shull, National Institute of Standards and Testing, Metall. Div., Gaithersburg, MD 20899-8552 USA

8:30 AM Opening Remarks: Vijay Vasudevan, George Spanos, Robert Shull and Xinghang Zhang

8:40 AM Invited
Two-Phase Equilibrium in Individual Nanoparticles of Bi-Sn: *William A. Jesser*[1]; C. Thomas Schamp[1]; [1]University of Virginia, Matls. Sci. & Engrg., 116 Engrs. Way, PO Box 400745, Charlottesville, VA 22904-0745 USA

When evaluating the path of phase transformations in systems with nanoscopic dimensions one often relies on bulk phase diagrams for guidance because of the lack of phase diagrams that show the effect of size. In order to provide insight into how the phase diagrams vary when a high surface curvature exists, binary alloys of Bi and Sn were prepared as a collection of individual crystalline particles vapor deposited onto amorphous carbon substrates in ultra-high vacuum. These crystallites were annealed after deposition to equilibrate the phases and structures. After annealing they were transferred to the transmission electron microscope for analysis of the phase state as a function of composition and surface curvature, i.e., particle radius. Individual crystallites were analyzed with respect to crystallinity, two-phase or one phase coexistence, and composition. The data show that there is a critical size below which there is no limit to the solubility, in strong contrast to that found in the bulk system, which is a simple eutectic alloy with only a few percent solubility on the bismuth-rich solid solution side of the phase diagram and about 15% on the tin-rich side. The change in solubility limit with size was found to be very strong in the bismuth-rich terminal solid solution and very-weak in the tin-rich terminal solid solution. A thermodynamic approach to using free-energy expressions modified to account for surface curvature can be successful in showing the shift in solubility with size. It is shown that the appropriate thermodynamic potential to minimize is a modified Helmholtz free energy.

9:15 AM Invited
In Situ TEM Observation of Alloy Phase Formation in Isolated Nanometer-Sized Particles: *Hirotaro Mori*[1]; JungGoo Lee[1]; [1]Osaka University, Rsch. Ctr. for Ultra High Voltage Electron Microscopy, Yamadaoka 2-1, Suita, Osaka 565-0871 Japan

Alloy phase formation in nanometer-sized particles has been studied by in-situ TEM using particles in the Au-Sn, Bi-Sn, and Pb-Sn systems. It is revealed that not only the surface energy but also the interface energy of an interface between two different phases (solid-solid or solid-liquid) significantly changes the phase equilibrium of nanometer-sized particles. For example, due to the interface energy of an interface between two different solid phases, the eutectic point in the three alloy systems decreases much faster than the melting point in pure substance with decreasing size of particles. Furthermore, it is revealed that the solid solubility is greatly enhanced in nanometer-sized alloy particles. In approximately 16-nm-sized lead solid solution, the tin solubility was higher than 56 atomic percent at 110 degrees, which is almost five times higher than that in the corresponding bulk material. Factors governing the phase equilibrium of nanometer-sized alloy particles will be discussed.

9:50 AM Invited
Interface-Controlled Phase Equilibria in Nanoscale Systems: *Gerhard Wilde*[1]; [1]Research Center Karlsruhe, Inst. of Nanotech., PO Box 3640, Karlsruhe D-76021 Germany

Phase equilibria in nanoscale systems, e.g. of matrix-embedded nanoparticles, can deviate strongly from the phase diagrams valid for the respective macroscopic system. Specifically, the observation of size-dependent melting transformations has stimulated controversial discussions concerning the underlying mechanism. Yet, recent experimental results indicate that the shift of the melting temperature at small system size is thermodynamic in nature, as opposed to explanations based on nucleation kinetics, and that the topology of the particle/matrix interface controls the phase transformation. In multicomponent and nanoscaled alloy systems, interface segregation and interface-induced stresses are known to affect the phase boundary lines of the phase diagrams. Yet, already the presence of internal heterophase interfaces contributing an excess free energy is sufficient to severely modify the phase equilibrium and the associated phase transformations in nano-size alloy systems. Here, new results on the constitutive behavior of binary nanoscaled model alloys, obtained by experiment and calculation, will be highlighted.

10:25 AM Break

10:40 AM Invited
Structural and Compositional Transformations in Nanocrystals: *Paul Alivisatos*[1]; [1]University of California, Dept. of Chmst, D-43A Hildebrand Hall, Berkeley, CA 94720 USA

The kinetics of structural and compositional transformations in nanocrystals is markedly different and more homogeneous than in bulk solids. This talk will review several such changes including pressure induced isomerizations, cation exchanges, and reactions involving interdiffusion.

11:15 AM Invited
Surface-Stress Effects on the Phase Equilibrium in Nanoscale Metal Hydrides: *Jörg Weissmüller*[1]; Christian Lemier[1]; [1]Forschungszentrum Karlsruhe, Inst. of Nanotech., PO Box 3640, Karlsuhe D-76021 Germany

Size-dependent shifts in the chemical equilibrium can arise from elastic interaction between interfaces and the bulk, e.g. in solid solutions equilibrated with a reservoir of solute at controlled chemical potential. Changes in the local composition give rise to forces at surfaces, which must be balanced by stress in the bulk. The pressure can reach values of several GPa; it affects the chemical potential as well as the apparent solute-solute interaction energy. Metal hydrides provide model systems for related experimental studies, since hydrogen can be ex-changed reversibly. By combining the analysis of the simultaneous chemical and mechanical equilibrium with experimental data for nanoscale metal hydrogen systems, one can measure the interface stress as a function of the chemical potential, and one can measure the 'stretch', a deformation normal to the interface. At nanometer system size, the equilibrium phase diagrams of solid solutions with a miscibility gap are strongly affected by elastic interactions.

Precious Metals: Au, Ag, Pt, Pd, Os, Rh, Ir, Ru

Sponsored by: Extraction & Processing Division, Light Metals Division, EPD-Aqueous Processing Committee, EPD-Precious Metals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizer: Richard S. Kunter, Behre Dolbear Company, Golden, CO 80401-9420 USA

Monday AM Room: 2022
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Richard S. Kuech, Behre Dolbear Company, Golden, CO 80401-9420 USA

8:30 AM
Effect of Magnetic Field on the Permeation of Hydrogen in Ni-Pd Alloys: *Sivaraman Guruswamy*[1]; Purushottam Kumar[1]; Robert Pennington Corson[1]; Pinai Mungsantisuk[1]; Michael L. Free[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

This paper examines the effect of magnetic field on the diffusivity of hydrogen isotopes in Ni-Pd alloys. Ni-Pd is an interesting alloy system for this study since both Ni and Pd absorb hydrogen, NiPd alloys shows significant magnetostrictive strain, and Ni-Pd system exists as a continuous single-phase region over the entire composition range. Electropermeation method as devised by Devanathan and Stachurski was used in this study. In this method, the coverage of hydrogen on one side of the NiPd alloy membrane is maintained at a constant level by cathodically charging hydrogen while it is anodically maintained at zero level on the other (discharge)side. The current on the discharge cell circuit gives the permeation rate. Alloys examined include Ni-70 at.% Pd alloy that shows a large magnetostrictive strain. Diffusivity of hydrogen calculated using lag times shows significant changes under a magnetic field. Support by University of Utah gratefully acknowledged.

8:55 AM
Magnetostriction and Magnetic Properties of PdX (X=Fe, Ni, Co) Single Crystals: *Purushottam Kumar*[1]; Robert Pennington Corson[1]; Pinai Mungsantisuk[1]; Sivaraman Guruswamy[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

Fe-Pd, Co-Pd, and Ni-Pd alloy systems show appreciable magnetostrictive strains that are of interest for sensor applications. These systems exhibit congruent melting minima. In this work, we have examined the magnetic and magnetostrictive behavior of congruent melting Fe-Pd, Co-Pd, and Ni-Pd compositions. The alloys examined include Fe- 54.9 at.% Pd, Ni-45 at.% Pd, and Co-45 at.% Pd. The paper describes the growth of bulk single crystals of these alloys using Czochralski technique. X-ray diffractometer was used to obtain rocking curves that were used to determine the [001] crystal direction. Magnetostriction in [001] direction was measured using strain gages. Magnetic properties were measured using a vibrating sample magnetometer. NiPd showed the highest magnetostriction values among the congruent melting compositions. Magnetic and Magnetostrictive behavior of these alloy single crystals are presented. Work partially supported by NSF Grant # DMR 0241603.

9:20 AM
Magnetostriction and Magnetic Properties of Polycrystalline PdX (X=Ni, Co, Fe) Alloys: *Pinai Mungsantisuk*[1]; Kevin Nguyen[1]; Robert Pennington Corson[1]; Purushottam Kumar[1]; *Sivaraman Guruswamy*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The presence of congruent melting minima, the occurrence of ferromagnetic transition over a large composition range, and FCC type crystal structure make the Pd-Ni, Pd-Co, and Pd-Fe based systems attractive candidates in our search for materials with no rare-earths, large low-field magnetostriction, and good mechanical properties. The presence of paramagnetic-ferromagnetic transition over a broad range of composition also provides opportunity for fundamental studies. In this work, we have examined the microstructure, hardness, elastic modulus, magnetic and magnetostrictive behavior of Pd-Ni, Pd-Co, and Pd-Fe alloys over the entire composition range. Alloy sample rods were prepared by arc melting and casting. X-ray diffractometer was used to obtain lattice parameter and texture data. Magnetostriction was measured along the rod axis using strain gages. Magnetic properties were measured using a vibrating sample magnetometer. Elastic modulus measurements were made using RUS technique. Work supported by University of Utah and NSF-DMR Grant #0241603.

9:45 AM
An Electrochemical Investigation on Intensification of Gold Cyanidation by Heavy Metal Ions: *Guanghui Li*[1]; Yongbin Yang[1]; Qian Li[1]; Yufeng Guo[1]; Huang Zhucheng[1]; Tao Jiang[1]; [1]Central South University, Sch. of Resources Procg. & Bioengrg., Changsha 410083 China

The effects of heavy metal ions on gold cyanidation have been studied by using electrochemical techniques. Results show that addition of thallium, bismuth, silver, mercury and lead ions greatly widen potential zone of activation dissolution of gold and significantly improve the anodic current density. The suitable concentration of heavy metal ions for the activation is found to be 10-6M for lead, 10-5M for bismuth, 5×10-5M for mercury and 8×10-5M for silver. Gold dissolution rate increases continuously with thallium concentration within the range of 10-6-10-4M. Based on research results on anodic and cathodic processes, mixed-potential models of gold dissolution in the presence of heavy metal ions have been constructed. Investigation shows that the enhancing effect of the heavy metal ions on gold leaching is clearly improved if there are cathodic assistants such as hydrogen peroxide.

10:10 AM
Controlling Mercury Emissions with Spent Catalytic Converter Cores: *Zhiyong Xu*[1]; Jinjing Luo[1]; Robert Greenlund[1]; Bowen Li[1]; Jim Hwang[1]; [1]Michigan Technological University, Inst. of Matls. Procg., 1400 Townsend Dr., Houghton, MI 49931 USA

In this study the spent automobile catalytic converter cores were examined to evaluate the effectiveness of using treated spent automobile catalytic converter core materials as low cost, viable sorbents in the capture of mercury chemical forms associated with coal-fired power plant emissions. This study was conducted by comparing the mercury adsorption effect between spent catalytic converter core materials and activated carbons, common used mercury sorbents. The results showed that spent catalytic converter cores were significantly more effective than activated carbon.

Shape Casting — The John Campbell Symposium: Liquid Metal Quality

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday AM Room: 2008
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Diran Apelian, MPI - WPI, Matls. Sci. & Engrg., Worcester, MA 01609 USA

8:30 AM **Welcome:** Murat Tiryakioglu and Paul N. Crepeau

8:40 AM
Bifilms - The Most Exciting Discovery of the Century: *John Campbell*[1]; [1]University of Birmingham, Metall. & Matls., Elms Rd., N. Campus, Edgbaston, Birmingham, W. Midlands B15 2TT UK

A short overview is presented of the key role that bifilms play in the initiation of porosity, hot tears, and the control of ductility of cast metals. In addition, their transient nature in cast irons is indicated by

the recent studies of carbon films lining bubble trails, constituting leakage defects in grey iron castings. The role of bifilms in the control of cast structures in Al alloys appears extensive: (i) grain size is influenced by convection that can be suppressed by sufficiently large or numerous bifilms; (ii) DAS is sometimes controlled if the local region of the casting is separated from the bulk by bifilms, and undercools, giving a fine local DAS; and (iii) the mechanism of the modification of Al-Si alloys by Na and Sr is explained for the first time by the deactivation of bifilms as substrates for Si. It seems likely that the majority of all metal failures (whether of cast or wrought material) may be attributable to bifilms. Bifilms appear to be an important feature that we need to add to our metallurgical knowledge.

9:10 AM
A Method to Study the Life-Cycle of a Double Oxide Film Defect in Liquid Aluminum Alloys: *Ramin Raeiszadeh*[1]; W. D. Griffiths[1]; [1]University of Birmingham, Interdisciplinary Rsch. Ctr. in Matls. Procg., Dept. of Metall. & Matls. Sci., Elms Rd., Edgbaston, Birmingham, W. Midlands B15 2TT UK

Entrained double oxide films have been held responsible for a reduction in mechanical properties in aluminum casting alloys. However, to date, their life cycle in the liquid metal has not been studied directly. A silicon nitride rod with a 13.3 mm diameter hole in one end was plunged into liquid aluminum to hold a known volume of air in contact with the liquid metal at a controlled temperature. The change in the air volume with time was recorded by real time x-ray radiography to determine the reaction rates of the bubble atmosphere with the liquid aluminum as a model for the behaviour of an entrained double oxide film defect. The results showed that first oxygen and then nitrogen were consumed by the aluminum alloy to form aluminum oxide and aluminum nitride. The effect of adding different elements to the aluminum and different hydrogen contents has also been studied.

9:35 AM
The Use of Bifilm Index as an Assessment of Liquid Metal Quality: *Derya Dispinar*[1]; John Campbell[1]; [1]University of Birmingham, Metall. Matls., Elms Rd., N. Campus, Edgbaston, Birmingham, W. Midlands B15 2TT UK

There is growing evidence that, in general, aluminium castings under-perform by a large margin. This is not only because of shrinkage or gas porosity, but particularly because of existence of extremely thin but detrimental defects called bifilms. Bifilms are the initiators of porosity. The opening of a bifilm to make a pore involves negligible energy, being so easy that it can be assumed that this process will be overwhelmingly favoured. The discriminating use of the Reduced Pressure Test clearly reveals the existence of bifilms. Until now the unpredictable behaviour of bifilms has made the RPT hard to quantify. A new fundamental parameter is proposed here to assess melt quality called the Bifilm Index. Typical total bifilm lengths vary from 3 to 300 mm. The effect of the bifilm index on the mechanical properties was studied using several common Al-Si alloys cast under different conditions.

10:00 AM Break

10:10 AM
Preventive Metal Treatment Through Advanced Melting: *C. Edward Eckert*[1]; [1]Apogee Technology, 1600 Hulton Rd., Verona, PA 15147 USA

Conventional aluminum melting results in a deterioration of metal quality due to in-situ melt oxidation, exposure of molten metal to products of combustion, and from separation of the oxide envelope that surrounds the charge media. Peak melt surface temperatures in reverberatory melting can exceed 2000OF. This results in an increase in oxidation rate by a factor of 64, compared to a bulk temperature of 1350OF. Additionally, the dew point of typical products of combustion is equivalent to saturated air at 75OF, further exacerbating oxidation and establishing a high partial pressure of monatomic hydrogen. Such melting processes depend on downstream remedial metal treatment for removal of consequential inclusions and dissolved hydrogen. An advanced melting process has been developed under the support of the US Department of Energy Office of Industrial Technology, with the original objectives of minimizing specific melting energy and melts loss. This has been accomplished. In addition, it has been found that dissolved hydrogen levels well under 0.2 cm3 H2/100g Al, with a freedom from visible supernatant oxides, typify aluminum melted by this process. Importantly, the peak temperature attained is approximately 40OF lower than bulk temperature, and no products of combustion are produced. The melting process also includes an integral flotation device to separate surface oxides introduced by the charge media. Metal treatment has now become implicitly preventative through the use of advanced melting. This paper will describe the development, performance, and impact on metal quality that characterizes the advanced melting process. Preventative metal treatment represents a paradigm shift in aluminum melt preparation methodology.

10:35 AM
The Relation Between Al_3Ti Particle Formation and Impurity Removal During In-Situ Precipitation Treatment of Al-Ti-X Alloys: *Sompong Srimanosaowapak*[1]; Keyna O'Reilly[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

Precipitation-sedimentation of Al_3Ti particles has been found recently by the authors[1] to increase the cleanliness of an Al-Ti melt. However, so far, little has been presented regarding quantitative evaluation of impurity removal by using this precipitation technique. In order to improve the understanding of the incorporation of impurities in precipitating particles in molten alloys, doping with different alloying elements and controlling the cooling rate during particle precipitation in Al-0.4, 0.6%Ti have been studied. Results indicated that elements, which have trialuminide structure, substantially substituted in the Ti sublattice of Al_3Ti. The reduction of impurity removal of solid inclusions by in-situ Al_3Ti precipitation with increasing cooling rate has been discussed by relating to the possibility of the reduction in incorporation by engulfment caused by relatively low Al_3Ti particle growth rates and the increase in convective flow within the melt at high cooling rates. [1]Srimanosaowapak, S. and O'Reilly, K.A.Q., Light Metals (Metaux Legers), 2004, 283-297.

11:00 AM
Panel Discussion: Inclusions in Aluminum Melting, Treatment and Casting: *David Neff*[1]; Paul N. Crepeau[2]; [1]Metaullics Systems, Solon, OH 44139 USA; [2]General Motors, 895 Joslyn Rd., MC 483-710-251, Pontiac, MI 48340 USA

A panel of 4 well-known figures in the aluminum casting industry will discuss a series of slides illustrating inclusions encountered in aluminum melting, treating and casting. This presentation is sponsored by the American Foundry Society and is an update of material shown at the most recent AFS Casting Congress. The material is will not be included in the conference transactions.

Superalloys and Coatings for High Temperature Applications: Bond-Coat Technologies - I

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Monday AM Room: Nob Hill A/B
February 14, 2005 Location: San Francisco Marriott

Session Chairs: Roger C. Reed, University of British Columbia, Dept. of Metals & Matls. Engrg., Vancouver, BC V6T 1Z4 Canada; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

8:30 AM Invited
Environmental and Thermal Barrier Coatings for Gas Turbine Engine Components: Status and Challenges Ahead: *Ramgopal Darolia*[1]; [1]GE Transportation, Matls. & Process Engrg., 1 Neumann Way, MD M-89, Cincinnati, OH 45215 USA

The nickel-base superalloys used for high-pressure turbine (HPT) airfoils are subjected to very high temperatures, stresses and oxidizing and corrosive environments. Significant progress has been made in developing high strength superalloys as well as in advanced protective coatings. To reduce surface temperatures and durability of the airfoils, thermal barrier coatings (TBC) have been used for the past decade. An understanding of the behavior of two essential components of TBC (zirconia topcoat and the alumina forming bond coat) is essential for reliable utilization of TBC. The bond coat use temperature limits the maximum allowable TBC surface temperature partly due to potential for erosion and impact damage of TBC. Higher temperature bond

coats, and improvement in erosion and impact resistance of the topcoat are two key development needs. Coating/superalloy interactions are also concerns in higher refractory containing superalloys. As the TBC surface temperature is increased, phase and microstructural stability are other limiting considerations.

9:00 AM Invited
The Influence of Bond Coat Properties and Phase Transformations in the Degradation of Thermal Barrier Coatings: *Kevin J. Hemker*[1]; [1]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA

Thermal barrier coatings (TBCs) used in gas turbine engines are comprised of multilayers with multiple functions. A ceramic top coat is the insulator, the formation of a thermally grown oxide (TGO) slows oxidation, the intermetallic bond coat provides a reservoir for TGO formation and improved adherence, and the superalloy substrate carries the loads. During thermal cycling, the interaction of these chemically and mechanically diverse layers determines the lifetime of the coating. The occurrence of a martensitic transformation in platinum modified nickel aluminide bond coats has been shown to promote TGO rumpling, which results in topcoat spallation. The interplay between transformation temperatures and elevated temperature creep strength of the bond coat holds particular importance, and both of these quantities have been shown to be extremely sensitive to alloy composition. This presentation will outline recent efforts to characterize the effect of alloying on the martensitic transformation temperatures of NiAl-based alloys and to measure the creep strength of various bond coat compositions with elevated temperature microsample tensile testing.

9:30 AM
Ru-Modified Platinum Aluminide Bond Coatings for TBC Systems: *Brian Tryon*[1]; Ken S. Murphy[2]; Carlos G. Levi[3]; Jingyu Yang[3]; Tresa M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 3062 H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49461 USA; [3]University of California, Matls. Dept., Santa Barbara, CA 93106 USA

Diffusional processes for the fabrication of multi-layered Ru-modified bond coats for thermal barrier coatings (TBCs) have recently been of interest for potentially prolonging the useful life of TBC systems. Ru-modifications have been made to a conventional Pt-modified nickel aluminide diffusion bond coating for thermal barrier coating systems. Interdiffusion and phase equilibria issues involved in the fabrication of the coating have been investigated. Structure evolution at various stages of the coating process has been studied with scanning electron microscopy and electron microprobe analysis. A comparison between the oxidation characteristics of the Ru-modified Pt-aluminide coating and conventional Pt-aluminide coatings is presented.

10:00 AM
Oxidation Behavior of RuAl Intermetallics: Paul Bellina[1]; Amalia Catanoiu[1]; Francisco M. Morales[1]; *Manfred Rühle*[1]; [1]MPI für Metallforschung, Heisenbergstr. 3, Stuttgart 70569 Germany

Bond coats (BC) play a crucial role in the performance of thermal barrier coatings (TBC) systems. Ru alloys and Ru-containing intermetallics were identified as promising candidates. Systematic studies were performed on the oxidation behavior of near stoichiometric â-RuAl alloys. Thermal gravimetric analyses (TGA) were performed in oxygen or air at 1100°C for different times ranging from 0.1 h to 500 h. Microstructural studies were performed by SEM and TEM. Results of the oxidation showed a á-Al2O3 layer lying on top of a ä-Ru layer. Specific microstructural instabilities can lead to a sequence of alternating á-Al2O3/ä-Ru layers or to the formation of large elongated cavities parallel to the surface. Selected area diffraction (SAD) and high-angle annular dark field (HAADF) studies of the interfaces were performed. The results and implications will be discussed on the basis of an oxidation model. Work supported jointly by the NSF (DMR-0099695) and the EC (G5RD-CT2000-00573).

10:30 AM Break

10:50 AM
Analyses of Failure Mechanisms and Microstructural Characterization of a Thermal Barrier System with a Two-Phase Bond Coat Based on NiCoCrAlY: *Christopher Mercer*[1]; Sabine Faulhaber[1]; Tau Xu[1]; Anthony G. Evans[1]; [1]University of California, Matls. Dept., Engrg. II, Rm. 1355, Santa Barbara, CA 93110 USA

The mechanisms of failure of an EB-PVD thermal barrier coating (TBC) with a NiCoCrAlY bond coat have been ascertained on test specimens subjected to thermal cycling in a burner rig. In regions where the TBC is still intact, the morphology and microstructure of the thermally-grown oxide (TGO) has been characterized using a combination of scanning and transmission electron microscopy. Compositional analysis was carried out in the TEM using an energy-dispersive spectroscopy system with element mapping capabilities. In addition, sub-surface features were explored using a focused ion beam instrument with imaging capabilities. The TGO was found to exhibit a very non-uniform morphology and a complex microstructure and composition. A dominant delamination has been identified extending primarily along the interface between the TGO and the bond coat, but traversing the TGO at imperfections (pegs) leaving TGO islands embedded in the bond coat. Analytical modeling has revealed that the principal stresses induced around the pegs and energy release rates for interface cracks remain invariant on a cycle-by-cycle basis, and are too small to nucleate an interface crack. The implication is that occasional large imperfections nucleate interface delaminations. Once formed, these delaminations become unstable and propagate along the interface.

11:15 AM
Oxidation Resistance of Ir-Pt Coated Ni-Base Superalloys Prepared by Electorodeposition Method: *Aya Suzuki*[1]; Yingna Wu[2]; Yoshinobu Yamamoto[3]; Hideyuki Murakami[4]; [1]National Institute for Materials Science, High Temp. Matls. Grp., Matl. Engrg. Lab., 1-2-1 Sengen, Tsukuba Sci. City, Ibaraki 305-0047 Japan; [2]National Institute for Materials Science, Thermal Spray Grp., Matl.. Engrg. Lab., 1-2-1 Sengen, Tsukuba Sci. City, Ibaraki 305-0047 Japan; [3]National Institute for Materials Science, Non-Oxide Ceram. Grp., Advd. Matl. Lab., 1-1 Namiki, Tsukuba Sci. City, Ibaraki 305-0044 Japan; [4]University of Tokyo, Dept. of Matls. Engrg., Sch. of Engrg., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

Pt-modified aluminide coatings, as well as MCrAlY overlay coatings have widely been used as bond coat materials for thermal barrier coating systems. Recent investigation also proposed that Pt coated and simply annealed samples demonstrated better oxidation resistance than simply aluminized specimens. In this study, we propose electrodeposited Ir-Pt coatings because Ir may play a role as a solid-solution hardener of Pt-base coatings with maintaining oxidation resistance. The effects of composition and post electrodeposition treatment, on the characteristics of the coated materials were investigated. aim of this study is to investigate the high-temperature characteristics of Ir-Pt alloy coatings prepared by the electrodeposition process. Ir-Pt alloys with various compositions were coated on Ni-base sigle crystal superalloy TMS-82+ substrates. They were then treated by the conventional Al-pack-cementation process (typically 1273K for 5h). Cyclic oxidation tests, hot-corrosion tests and high-temperature mechanical test were conducted to characterize the high-temperature mechanical properties.

11:40 AM
A Platinum-Enriched γ+γ' Bond Coat with Different Hf Contents: *Y. Zhang*[1]; B. A. Pint[2]; J. A. Haynes[2]; L. D. Chitwood[2]; [1]Tennessee Technological University, Dept. of Mechl. Engrg., 115 W. 10th St., Box 5014, Cookeville, TN 38505-0001 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6156 USA

The constant demand for increased operating temperatures in gas turbine engines has been the driving force for development of more reliable thermal barrier coating (TBC) systems. Recently, it has been recognized that compared to the commercial single-phase β-(Ni,Pt)Al bond coat, a two-phase γ+γ' coating could offer some potential advantages such as higher creep strength, better compatibility between coating and superalloy substrate, improved metallurgical stability, and reduced manufacturing cost. In this study, a Pt-enriched γ+γ'-type coating has been applied to a directionally-solidified René 142 superalloy with three different Hf contents (0.02, 0.76, and 1.37 wt.%). The γ+γ' coating was prepared by electroplating a thin layer of Pt (~7μm) on the superalloy substrate followed by a diffusion treatment in vacuum at 1150°C. Isothermal and cyclic oxidation tests were conducted on the coated specimens at 1100 and 1150°C. It was found that the oxidation performance of the γ+γ' coating was comparable to the β-(Ni,Pt)Al coating and much better than the simple β-NiAl coating at the same testing temperature. The beneficial effect of Hf was clearly demonstrated by the fact that the oxidation behavior of the γ+γ' coating was proportional to the Hf content of the substrate, with higher Hf levels resulting in improved performance.

12:05 PM
Degradation of Protective Coatings Under Thermal and Mechanical Loading: *Bernd Baufeld*[1]; Marion Bartsch[1]; Serdar Dalkiliç[2]; Michael Heinzelmann[3]; [1]German Aerospace Center, Inst. of Matls. Rsch., Porz-Wahnheide, Linder Höhe, Köln, Nordrhein-Westfalen 51147 Germany; [2]Anadolu University, Coll. of Civil Aviation, Eskisehir 26470 Turkey; [3]Fachhochschule Bonn-Rhein-Sieg, Technische

Mechanik & Festigkeitslehre, Von-Liebig-Straße 20, Rheinbach, Nordrhein-Westfalen 53359 Germany

Different high temperature testing schemes like cyclic thermal, cyclic mechanical at constant high temperatures, and cyclic thermal mechanical, the latter either with or without additional temperature gradient, were performed on specimens with a NiCoCrAlY coating and in some cases with an additional thermal barrier coating (TBC). While the testing under thermal mechanical loading with thermal gradient is the most complex, it simulates most closely in-service conditions for internally cooled components. The resultant defects and the degradation evolution proved to depend critically on the testing scheme and on the presence of the TBC. With the help of finite element analysis the observed defects can be related with the testing condition. The results suggest the possibility to deduce the prevailing conditions from the observed defects. Furthermore, they show the necessity to design laboratory tests as realistic as possible, especially if the test data are used for lifetime assessment.

Texture and Microstructure in Thin Films and Coatings: Copper Metallization

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Monday AM Room: 3010
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: John E. Sanchez, Unity Semiconductor, Sunnyvale, CA USA; Kris Kozaczek, Hypernex Inc., State College, PA 16801 USA

8:30 AM Invited
Texture-Related Reliability Problems of Cu Thin Films For Semicondutor Application: *Junichi Koike*[1]; Makoto Wada[1]; [1]Tohoku University, Dept. of Matls. Sci., 02 Aoba, Aramaki, Aoba-ku, Sendai, Miyagi 980-8579 Japan

Cu thin films have been extensively studied in the past decade for interconnect application in advanced semiconductor devices. Texture and microstructure of the Cu film have been known to influence the interconnect reliability. The presentation will address three important topics for the reliability of the Cu thin films with regard to the dependence of initial film texture on (1) room-temperature self-annealing, (2) the texture after heat treatment and (3) the formation of voids under thermal stress conditions. The observed results can be consistently explained by considering a large elastic anisotropy of Cu, elastic energy density and stress distribution.

9:00 AM
Textures of Copper Interconnects with Different Geometries: *Dong-Nyung Lee*[1]; Hyo-Jong Lee[1]; Ui-Hyung Lee[1]; Sook-Hoon Kang[1]; Kyu-Hwan Oh[1]; [1]Seoul National University, Rsch. Inst. of Advd. Matls. & Sch. of Matls. Sci. & Engrg., Seoul Korea

The properties of deposited metal films and interconnect structures at submicrometer scale are sensitive functions of microstructural features. Therefore, understanding of the factors which control microstructural evolution is necessary for the development and design of reliable, manufacturable interconnect structures, especially in copper damascene interconnects. In this study, the trench width of copper interconnects was increased from 0.2 to 6 microns at a given space of 0.2 microns and the space was increased from 0.2 microns to 1 microns at a given width of 0.5 microns. The interconnects were annealed in various ways, UV heating at 200°C immediately after electrodeposition, UV heating at 200°C after self-annealing for 3 h, and self-annealing for a week. The annealed interconnects were subjected to the texture-measurement by EBSD. The texture changed from {111}<110> to {111}<112> and eventually to <111> fiber with increasing trench width, and tended to change from {111}<112> to {111}<110> with increasing space regardless annealing conditions. The results were discussed based on the strain-energy-release-maximization, in which the annealing texture is determined such that the minimum Young's modulus direction of annealed grains is parallel to the maximum stress direction of deposits.

9:20 AM
Investigating the Texture and Grain Boundary Character of ECD Copper Films Using Orientation Imaging: *Matthew Nowell*[1]; Aaron Frank[2]; [1]TSL/EDAX, 392 E. 12300 S., Ste. H, Draper, UT 84020 USA; [2]Texas Instruments, TX USA

The use of copper as a device interconnection material in integrated circuit devices has developed through the use of the damascene manufacturing process due to the need to improve electromigration failure resistance. In this process the copper metal is electrochemically deposited onto the oxide substrate and fills patterned interconnect trenches. Chemical-Mechanical polishing (CMP) then removes the overburden film, and leaves only the copper interconnections. The diffusion barrier layers between the oxide and the deposited film and the current densities used in the deposition process can influence the texture and grain boundary character of the resulting copper film. Additionally, the thermal annealing prior to CMP will also affect the microstructure. In this work, orientation imaging microscopy (OIM) is used to investigate the effects of barrier layer material and deposition current density on texture and grain boundary development during copper deposition. The effects of subsequent thermal annealing are also addressed.

9:40 AM
Texture Evolution During Annealing of Electroplated Cu Films: *Nojin Park*[1]; David P. Field[2]; Matthew M. Nowell[3]; [1]Kumoh National Institute of Technology, Sch. of Advd. Matls. & Sys. Engrg., 188 Shin Pyung Dong, Kumi, Kyung Buk 730-701 Korea; [2]Washington State University, Mechl. & Matls. Engrg., Box 642920, Pullman, WA 99164-2920 USA; [3]TexSEM Laboratories/EDAX, 392E 12300S, Ste. H, Draper, UT 84020 USA

Structural evolution during annealing of electroplated copper films is a strong function of film processing parameters, including sublayer material and thickness, bath chemistry, film thickness, and annealing temperature, among others. Because electroplating of copper into narrow trench structures is employed in the fabrication of modern integrated circuits, understanding structural evolution becomes important in controlling properties and maintaining uniformity of the circuit. A {111} fiber texture is generally assumed to be the preferred structure in such films. The strength of this texture weakens dramatically by the accumulation of twin boundaries that are prevalent in relatively low stacking fault metals such as copper. Evolution of both texture and grain boundary structure are examined by use of in-situ investigations using electron backscatter diffraction.

10:00 AM Break

10:30 AM
Role of Substrate in Influencing High Temperature Behavior of Cu Film Studied In-Situ by EBSD: *Kabirkumar Mirpuri*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Matls. Engrg., M. H. Wong Bldg., 3610 Univ. St., Montreal, QC H3A 2B2 Canada

High temperature behavior of Cu films is studied in-situ in SEM by electron back-scatter diffraction (EBSD). The microstructure of the films was already stabilized. Two different investigations were performed at RT, 200 and 400°C. The film showed the presence of (111) fiber texture together with {111}<110> and {111}<112> preferred orientations. In the first investigation the strength of {111}<110> became stronger compared to {111}<112> while in second it became weaker. Similar investigation for freestanding Cu film did not show any such texture transformation making it clear that substrate had a role to play. To ascertain the mechanism by which substrate was influencing texture transformation, the average misorientation between the neighboring scan points was computed in the range of 2 to 7°. The (111) grains with lower values of misorientation were termed as recrystallized and were supposed to be showing lower dislocation activity. The grains with larger values of misorientations were termed as substructured and were suppose to be showing higher dislocation activity. It was found that in the first investigation where the {111}<110> component became relatively stronger at higher temperature the increase in the percentage of substructured {111}<110> grains was lower compared to the second investigation where the increase in the percentage of the substructured {111}<110> grains was higher compared to first investigation. Interestingly, the {111}<110> component became relatively weaker during the second investigation with increasing temperature. Thus, clearly it seems that the dislocations have a role in deciding the strength of {111}<110> component. The inclination of the {111}<110> and {111}<112> grains to the specimen surface was computed as a function of temperature for both the investigations. The tilt of these grains as a means of stress relaxation in relation to dislocation activity is discussed. The information gained from these experiments could be used to explain similar texture transformation in

the Cu damascene lines where we observed a stronger tendency of the system to have preferred {111}<112> orientations for the larger linewidths in the as-deposited condition for the cases where the lines were closely spaced.

10:50 AM
Effects of Surface Condition and Clean Techniques on Cu Films for the Quality of Cu Wafer Bonding: *K. N. Chen*[1]; C. S. Tan[1]; A. Fan[1]; R. Reif[1]; [1]Massachusetts Institute of Technology, Microsys. Tech. Labs., 60 Vassar St., Rm. 39-623, Cambridge, MA 02139 USA

In order to achieve Cu wafer bonding with good quality, surface conditions of Cu films are important factors. In this work, the effects of surface conditions such as surface roughness and oxide formation on the bonding strength are investigated under different bonding conditions. Some Cu wafers were cleaned using HCl before bonding in order to remove the surface oxide. Surface roughness of Cu film with and without HCl clean was examined. Since surface clean before bonding removes oxides but creates surface roughness, it is important to study the corresponding bonding strength under different bonding conditions. These results offer the required information for the process design of Cu wafer bonding.

11:10 AM
Microstructure Cu-Cr Coatings: *Kuang-Tsan Kenneth Chiang*[1]; Ronghua Wei[1]; James Arps[2]; Vijay Jain[1]; [1]CNWRA-SwRI, CSPE, 6220 Culebra Rd., San Antonio, TX 78238 USA; [2]SwRI, 6220 Culebra Rd., San Antonio, TX 78238 USA

Copper alloys and composites are of interest for high strength and high thermal conductivity applications at elevated temperatures. However, their usage at high temperatures is usually limited by severe oxidation attack. In this investigation, Cu-Cr coatings with Cr contents of 10-50 weight percent were produced by vacuum-based ion beam deposition techniques. The coating microstructure was characterized by scanning electron microscopy, x-ray diffraction, and transmission electron microscopy. Oxidation kinetics of the coatings was evaluated using a continuous-recording microbalance. At low Cr contents, the oxidation rates were controlled by outward diffusion of copper ions to form external Cu-oxides. For high Cr contents, a fine-grained Cr-rich oxide was formed on the coating surface. The effect of coating microstructure on high temperature oxidation resistance will be discussed.

11:30 AM
Effect of Low-K Dielectric on Textural and Microstructural Evolution of Cu Damascene Interconnects: *Jae-Young Cho*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

As the features of integrated circuitry (IC) chips are scaled to deep submicron dimensions, the signal delay (the resistance-capacitance delay) caused by interconnect becomes a dominant factor limiting speed performance. To reduce this delay, it is recommended to replace Al by Cu and to introduce low-k dielectric. From the previous research, it was found that the distribution of stress can be a major factor affecting textural and microstructural evolution of Cu damascene interconnects in embedded the SiO2 dielectric during annealing. Since the low-k material has significantly higher thermal expansion coefficient (CTE) than Cu and SiO2, it can generate very different thermal stress distribution in the trench during annealing. In addition, the pitch width can affect the stress distribution during annealing. To analyze a relationship between the stress distribution and textural evolution in the samples investigated, changes of the micro stress were calculated from the different pitch width using FEM in both SiO2 and low-k dielectric materials. It was found that the inhomogeneity of stress distribution in Cu interconnects is an important factor required for understanding textural evolution after annealing. Textural evolution in damascene interconnects lines after annealing was analyzed depending on dielectric materials used and the pitch width.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Monday AM — Room: 3003
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: John William Morris, University of California, MSE, Berkeley, CA 94720 USA; Yunzhi Wang, Ohio State University, MSE, Columbus, OH 43210 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Multi-Scale Theory and Modeling of Complex Structures in Solids: *Armen G. Khachaturyan*[1]; [1]Rutgers University, Ceram. & Matls. Engrg., 607 Taylor Rd., Piscataway, NJ 08502 USA

The significance of a transformation-induced strain for the thermodynamics of the phase transformations and will be discussed. This strain can qualitatively change the thermodynamics of coherent systems. The density function kinetic equations provide an efficient way to study the kinetics of microstructure evolution. Depending on the research objectives, the density functions can be either atomic densities or densities of compositional or structural parameters in nano- and meso-scales. For atomic scale simulations, the phenomenological density kinetic equations are formulated by an extension of the existing microscopic kinetic equations that assume atomic redistribution over underlying host lattice sites to more general cases where atomic positions are arbitrary. Examples of the application of these methods to the modeling of complex microstructure transformations at atomic and nano- scales will be discussed. It will be shown that the theory and models are already mature enough to realistically reproduce complex microstructures of different nature and even to predict new mechanisms of the microstructure transformation.

9:10 AM Invited
Elastic Domains and Khachaturyan's Phase Field Microelasticity Theory: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. Sci. & Engrg., Coll. Park 20742 USA

Microstructural evolution in solids can be considered as combination of the coherent phase transformations and plastic deformations. The nucleation and movement of dislocations lead to relaxation of internal stresses and affect the thermodynamic and kinetic of phase transformations. The Khachaturyan's theory of microelasticity creates an effective and elegant tool for quantitative simulation of these interrelated microstructure mechanisms. On the other side, the concept of elastic domains with dislocation screening presents qualitative interpretation of simulation results. Both approaches will be discussed in the talk.

9:35 AM Invited
Self-Accommodation Mechanism of Thermoelastic Martensites: *Kazuhiro Otsuka*[1]; Takuya Ohba[2]; [1]National Institute for Materials Science, Advd. Matls. Lab., 1-1 Namiki, Tsukuba, Ibaraki 305-0044 Japan and Foundation for Advancement of International Science(FAIS), Tsukuba, Japan; [2]Shimane University, Dept. of Matls. Sci., Nishikawatsu, Matsue, Shimane 690-8504 Japan

All crystallogrphic characteristics, such as habit plane, orientation relationship etc., of martensitic transformations in most alloys can be described by the so-called "phenomenological crystallographic theory of martensitic transformations", in which the habit plane is treated as an invariant plane. Thus much of the strains arising from the martensitic transformation can be eliminated by the invariant plane strain condition. However, this mechanism cannot eliminate shear strains along the habit plane. Thus in order to eliminate strains further, two or four (in a special case may be three) habit plane variants are formed

side by side. This process is called self-accommodation. We will discuss the self-accommodation morphology of various martensites both experimentally and theoretically, which include B2-14M transformation in Ni-Al, B2-z2'(trigonal) transformation in Au-Cd, B2-R transformation in Ti-Ni-Fe and B2-B19 transformation in Ti-Ni-Cu. We also discuss a quite complicated case of B2-B19' (monoclinic) transformation in Ti-Ni.

10:00 AM
Crystallographic and Lattice Point Correlations of a New hcp-to-fco Martensitic Transformation in the Hf-Ni System: *J. H. Li*[1]; H. B. Guo[1]; *B. X. Liu*[1]; [1]Tsinghua University, Dept. Matl. Sci. & Engrg., Beijing 100084 China

Based on a newly constructed tight-binding Hf-Ni potential, molecular dynamics simulations reveal a new hcp-to-fco (face-centered orthorhombic) martensitic phase transformation in the Hf hcp-lattice, when the Ni solute atoms approach 19 at %, and the transformation speed is on the order of 100 m/s. The detailed mechanism of the martensitic phase transformation is figured out to involve the following four coinstantaneous movements: (1) sliding of (002) planes, (2) uniformly shearing on (010) planes along [010] directions, (3) stretching along [120] direction and (4) adjusting of the lattice constants in three principal crystalline axes of the resultant phase. After a series of analytical deduction, crystallographic constraints to the martensitic phase transformation and the corresponding matrix of lattice vectors between the initial hcp and final fco lattices are obtained, respectively, and they are also relevant for governing the possible hcp-to-bct (body-centered cubic) and hcp-to-fcc martensitic phase transformations.

10:15 AM Break

10:40 AM Invited
Incorporating Micromechanics into Phase Field Approach to Microstructure Evolutions in Complex Material Systems: *Yu U. Wang*[1]; [1]Virginia Tech, Matls. Sci. & Engrg., Blacksburg, VA 24061 USA

The problems of microstructure evolutions in complex material systems, such as multi-phase multi-domain microstructures, multi-dislocation and multi-crack ensembles, are discussed within the framework of Phase Field Microelasticity (PFM) theory. We consider different applications of PFM for modeling various processes by further developing the Khachaturyan's reciprocal space microelasticity theory. This is based on a new variational procedure that naturally incorporates micromechanics into the Phase Field approach. By using a virtual misfit strain field, the method efficiently solves the micromechanical problems involving arbitrary elastic modulus mismatch and crystal lattice misfit. Simulations of dislocation dynamics in bulk and thin films, crack propagations, surface roughening of heteroepitaxial films, phase transformations near free surface and in thin films are presented.

11:05 AM Invited
Phase Field Modelling of Transformation Strain Effect on Solid State Transformations: *Ingo Steinbach*[1]; Markus Apel[1]; [1]RWTH-Aachen, Access e.V., Intzestr. 5, Aachen D-52072 Germany

The ground breaking work of A. Khachaturyan and his co-workers on stress and strain effects on multiphase systems with coherent interfaces since more than one decade forms one pillar in Phase Field theory. Recently the principle model was generalized to treat elastically inhomogenous materials and it allows now to study effective elasticity in multigrain materials. In this presentation a multiphase field model is discussed, that follows the notion of the Khachaturyan model. Special emphasis is given to the elastic conditions in the diffuse interface between elastically different materials. It is demonstrated, how different interface conditions affect the elastic force on transformation in a multiphase system such as low carbon steel. An argument is derived for an 'optimal' model, that is applied to investigate nucleation and growth of ferrite into an austenite structure.

11:30 AM
Crystal Level Modeling of the Alpha to Epsilon Phase Transformation in Iron: *Nathan Barton*[1]; Richard Becker[1]; [1]Lawrence Livermore National Laboratory, PO Box 808, L-227, Livermore, CA 94551 USA

We present a crystal level model with phase transformation capabilities. The model is formulated to allow for large pressures (on the order of the elastic moduli) and makes use of a multiplicative decomposition of the deformation gradient in each constituent crystal. In this decomposition, elastic and thermal lattice distortions are combined into a single lattice stretch. This allows the model to be used in conjunction with general equation of state relationships. Within a given material element, phase transformations induce mass fraction rates between the constituents of the material. Deformation results from both these phase transformations and deformation of the constituents themselves. The driving force for phase transformations includes terms arising from mechanical work, from the temperature dependent chemical free energy change on transformation, and from interaction energy among the constituents. Simulation results are available for the alpha to epsilon phase transformation in iron. Results include simulations of shock induced transformation in single crystals and of compression of polycrystals. Results are compared to available experimental data. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

11:45 AM
"Burst-Like" Behavior During the Diffusionless Alpha Prime to Delta Transformation in a Pu-Ga Alloy: Kerri J.M. Blobaum[1]; *Christopher R. Krenn*[1]; Mark A. Wall[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, PO Box 808, L-353, Livermore, CA USA 94551

Phase transformations in plutonium alloys have many features that are difficult to model computationally. The transformation on cooling from δ to α' proceeds to less than 30% completion and has unusual double-c kinetics. A volume change of 20% during the transformation results in significant plastic deformation and a thermal hysteresis of 100K or more. Although 24 unique variants of α' phase are allowed, typically only 4 are seen to nucleate and grow in any given grain of δ. During the reversion back to δ at constant heating rates, unusual periodic "bursts" have been often observed, and the period appears to be independent of experimental technique and heating rate. We compare models of bursting caused by local elastic and thermal inhomogeneities, and conclude that plasticity induced residual stresses are the most likely explanation for the bursts. We also discuss general requirements for a complete computational model of this transformation.

12:00 PM
Severe Plastic Deformation and Deformation Twinning in NiTi Shape Memory Alloys: *Ibrahim Karaman*[1]; Ajay V. Kulkarni[1]; [1]Texas A&M University, Dept. of Mechl. Engrg., MS 3123, Coll. Sta., TX 77843 USA

In this study, thermomechanical properties of severely deformed Ti-50.8 at% Ni alloy using Equal Channel Angular Extrusion (ECAE) are investigated. Solutionized NiTi bars were deformed at different temperatures, i.e. room temperature which is above the austenite finish temperature (Af) and 450°C. The aim was to investigate the effects of ausforming (deformation above Af) on shape memory characteristics of NiTi such as superelasticity, transformation temperatures and fatigue properties. DSC was used to explore the effects of heat treatment temperature and time on the as-received, solutionized and as-deformed materials in terms of transformation temperatures, R-phase formation, and change in thermal hysteresis. TEM was utilized to reveal the changes in microstructure and formation of deformation twinning induced nanograins by in-situ heating and cooling experiment. Cyclic deformation tests are done on the as received, solutionized and as-deformed samples before and after some selected heat treatments. In this presentation improvement in thermal and mechanical properties with severe ausforming and subsequent annealing will be demonstrated. Stable cyclic response, pseudoelastic strain, change in transformation temperatures, formation of R-phase and nanograins, effects of precipitates will be rationalized with the observations on microstructures and possible deformation mechanisms. The unique microstructural findings were: 1) the observation of a mixture of heavily deformed B2 (austenite) and B19' (martensite) phases in the samples processed at room temperature although martensite stabilization was expected, 2) the observation of highly organized, twin-related nanograins in B2 phase of the samples deformed at room temperature, and 3) simultaneous observation of B2 austenite and strain induced B19' martensite in the samples deformed at 450°C. Strain-induced martensite in NiTi alloys was reported for the first time. The formation of well-organized twin-related nanograins via severe plastic deformation opens a new opportunity for twinning induced grain boundary engineering in NiTi alloys which is believed to improve cyclic stability and fatigue resistance of these alloys.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Creep

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Monday AM Room: 3024
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Michael E. Kassner, University of Southern California, Aeros. & Mechl. Engrg., Los Angeles, CA 90089-1453 USA; Atul H. Chokshi, Indian Institute of Science, Metall. Dept., Bangalore 560012 India; K. Linga Murty, North Carolina State University, Raleigh, NC 27695-7909 USA; Brian Wilshire, University of Wales Swansea, Matls. Rsch. Ctr., Sch. of Engrg., Singleton Park, Swansea SA2 8PP UK

8:30 AM
Contribution of Early Works by Terence Langdon in Modern Materials Science: *Ruslan Z. Valiev*[1]; Yuntian T. Zhu[2]; [1]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx str., Ufa 450000 Russia; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, STC, Los Alamos, NM 87545 USA

Prof. T.G. Langdon has been ranked as the second most cited author in materials science during the last 10 years by the international Institute for Scientific Information (ISI, Philadelphia, PA, USA). The analysis of his works shows that his most highly cited works are those dealing with recent studies of microstructural evolution and mechanical properties of ultrafine-grained (UFG) materials processed by severe plastic deformation, as well as earlier works on superplasticity carried out in the 1970-1980s. This paper traces how those early works have significantly affected the current investigations of the mechanical behaviour of UFG materials produced by severe plastic deformation.

8:45 AM
Rate-Controlling Processes in Creep of Subgrain-Containing Materials: *Oleg D. Sherby*[1]; Oscar A. Ruano[2]; [1]Stanford University, Matls. Sci. & Engrg., Stanford, CA 94305-2205 USA; [2]Centro Nacional de Investigaciones Metalurgicas, Dept. of Physl. Metall., CENIM, Av. Gregorio de Amo, 8, Madrid 28040 Spain

Polycrystalline materials that exhibit five-power-law creep behaviour are characterised by the presence of subgrains. In addition to the contribution of the subgrain-boundary as a barrier, it is also the source of the rate-controlling diffusion process in creep. It is proposed that the opposing stress fields, from the subgrain boundary and from the piled-up dislocations, will be relaxed by atom diffusion to allow climb of the lead dislocation in the pile-up. The atom diffusion process (solute or solvent) involves the combination of two edge dislocations in the subgrain boundary to form a double burgers vector (Smoluchowski) dislocation that releases the dislocation. The Smoluchowski dislocation reverts back to the prior two-dislocation structure during the climb of the dislocation(s) from the pile-up. It is a form of thermal-mechanical ratchetting. The model explains unusual creep behaviour in a number of metal systems.

9:00 AM
Deformation Mechanism Maps and Microstructural Influences: *Geoffrey Wilson Greenwood*[1]; [1]University of Sheffield, Dept. of Engrg. Matls., Sheffield UK

Areas on these maps define ranges, of homologous temperature and of applied stress divided by an elastic modulus, over which specific mechanisms of deformation are predominant. Langdon has illustrated their important features. The lines separating the mechanisms of overall sliding from those of diffusional drift are of particular significance but can be difficult to locate. It is shown how observations of microstructural features assist in this location and also provide extensive information on the way in which microstructure can influence the line positions. Notably, the flux patterns of diffusional drift are dependent upon the type of stress system superimposed as well as on microstructure. In many instances, the flux patterns can be determined to permit the evaluation of deformation rates.

9:15 AM
Grain and Grain Boundary Zone Contributions to Strain Accumulation During Creep of Polycrystalline Copper: *Brian Wilshire*[1]; Howard Burt[1]; Alun John Battenbough[1]; [1]University of Wales Swansea, Matls. Rsch. Ctr., Sch. of Engrg., Singleton Park, Swansea SA2 8PP UK

With pure metals, variations in the creep behaviour patterns displayed with decreasing applied stress are often interpreted in terms of a change from dislocation processes occurring entirely within the grains to boundary-dependent diffusional mechanisms which do not involve grain deformation. This transition is considered by reference to data obtained for pure polycrystalline copper, including the experimentally observed effects of grain size variations and of prestraining at room temperature. This evidence indicates the importance of distinguishing between the contributions made by the grain interiors and the grain boundary zones to the overall rates of strain accumulation at different stress levels.

9:30 AM
Effect of Temperature on Transitions in Creep Mechanisms in Class-A Alloys: *K. Linga Murty*[1]; Glenn T. Dentel[1]; Jeff C. Britt[1]; [1]North Carolina State University, PO Box 7909, Raleigh, NC 27695-7909 USA

Alloy class materials exhibit viscous glide controlled creep where dislocations are locked by solute atoms thereby decreasing their glide velocity while dislocation annihilation by climb still occurs. The creep-rate here varies as cube of the applied stress (n=3) while the activation energy for creep becomes equal to that for solute atom diffusion. At high enough stress, the dislocations get freed from solute atom locking and transition to climb controlled creep is noted with n~5 while at still higher stresses, power-law breakdown is observed. For relatively small grain-sizes, viscous creep mechanisms such as Nabarro-Herring or Coble creep usually occurs at low stresses. A close examination of the model equations reveals that at temperatures below a critical temperature, a transition from diffusional viscous creep to dislocation climb occurs without the intermediate viscous glide creep. Recent experimental results on a Zr-Nb sheet clearly follow these formulations exhibiting narrower intermediate region with decreasing temperature.

9:45 AM
Inhomogeneous Flow Characteristics in Plastically Accommodated Creep of a Composite: *Eiichi Sato*[1]; Kenshi Kawabata[2]; Kazuhiko Kuribayashi[1]; [1]Institute of Space and Astronautical Science/ Japan Aerospace Exploration Agency, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]Nagaoka University of Technology, Dept. of Mechl. Engrg., 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188 Japan

During high-temperature creep of a composite consisting of matrix and reinforcements, if diffusional accommodation is not sufficient, the strain mismatch between the matrix and inclusions must be accommodated by inhomogeneous flow of the matrix, called as "plastic accommodation". Unlike room temperature deformation showing work hardening, steady-state, plastically-accommodated creep might be inhomogeneous in space, but might not yield the rotation of the matrix lattice. This paper present, for the first time, the direct observation of inhomogeneous deformation without lattice rotation in a crept Ti/TiB(w) composite in plastic accommodation condition. Inhomogeneous flow was observed by the deposited AgPd and lattice rotation was observed by EBSP on the polished surface. The nature of plastically accommodated creep is then discussed.

10:00 AM
Compression and Tensile Creep of Binary NiAl: *S. V. Raj*[1]; [1]NASA Glenn Research Center, Matls. Div., MS 106-5, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Compression creep and long term tensile creep studies were conducted on cast and extruded binary NiAl in the temperature range 700-1200 K with the objectives of characterizing and understanding the creep mechanisms dominant in this alloy. Inverse and normal primary creep curves were observed in both compression and tension creep depending on stress and temperature although an asymmetrical response was observed under these two stress states. It was concluded that the primary creep of NiAl is limited by dislocation mobility. The stress exponents, n, for compression and tensile creep were similar varying between about 5 and 14. However, there were significant differences in the stress dependence of the activation energies for com-

pression and tensile creep. The true activation energy for tensile creep, Qc, was constant and equal to about 400 kJ mol-1 between 20 and 50 MPa but decreased to a constant value of 250 kJ mol-1 between 50 and 110 MPa. The activation energy was observed to be inversely stress dependent above 110 MPa. In contrast, Qc ≈ 300 kJ mol-1 for compression creep was constant between 25 and 70 MPa and inversely dependent on the true stress above 70 MPa. A detailed discussion of the probable dislocation creep mechanisms governing compressive and tensile creep of NiAl is presented. It is concluded that the non-conservative motion of jogs on screw dislocations influenced the nature of the primary creep curves, where the climb of these jogs involves either the next nearest neighbor or the six-jump cycle vacancy diffusion mechanism. The probable natures of the atom-vacancy exchange that occur within the core of an edge dislocation undergoing climb in NiAl are schematically examined.

10:15 AM
Creep Behaviour of Mg Alloys and Composites Investigated by Acoustic Emission: *Frantisek Chmelik*[1]; Zuzanka Trojanova[1]; Pavel Lukac[1]; [1]Charles University, Dept. of Metal Physics, Ke Karlovu 5, Praha 2 12116 Czech Republic

The application of different processing has allowed preparing magnesium-based materials with different grain size. The creep tests were conducted in the temperature range of 423-473 K under stress between 30 and 95 MPa. Acoustic emission was monitored during the test. The results demonstrate that the preparation technique can influence the creep behaviour. In the case of composites, the creep behaviour depends on the reinforcement and the matrix alloy. The analysis of the creep data of unreinforced alloys shows an increase in the stress exponent and an increase in the activation energy. No clear dependence of the stress exponent and the activation energy on temperature and applied stress is observed for composites crept at similar conditions. The acoustic emission activity is higher in the composite and it depends on the reinforcement. Acoustic emission indicates the initiation of failure earlier than that observed on the creep curve.

10:30 AM Break

10:45 AM
Recent Developments in Mechanisms of Five Power-Law Creep: *M. E. Kassner*[1]; [1]University of Southern California, Aeros. & Mechl. Engrg., OHE 430, CA 90089-1453 USA

This work will describe the latest developments on the understanding of the dislocation mechanisms for elevated temperature five power-law creep in materials. This will include a recent analysis of the applicability of the classic Taylor-hardening equation to creep in this regime. Additionally, there will be a discussion of the latest developments in the assessment of internal stresses in creep, including x-ray peak asymmetry analysis.

11:00 AM
Analysis, Representation, and Prediction of Creep Transients in Class I Alloys: *Eric M. Taleff*[1]; W. Paul Green[1]; Terrry R. McNelley[2]; Paul E. Krajewski[3]; [1]University of Texas, Dept. of Mechl. Engrg., 1 Univ. Sta., C2200, Austin, TX 78712-0292 USA; [2]Naval Postgraduate School, Dept. of Mechl. Engrg., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [3]General Motors Corporation, R&D Ctr., Bldg. 1-6, 30500 Mound Rd., Warren, MI 48090-9055 USA

Solute-drag creep in Class I alloys, also known as Class A alloys, is characterized by several features. Among these is the presence of "inverse" creep transients, which is a feature unique to these solid-solution alloys and the solute-drag creep mechanism. These creep transients can involve large changes in flow stress that occur over extended periods of strain and may have substantial impact on commercial hot-forming operations. A graphical construct for conveniently representing the magnitudes of creep transients, in general, and inverse transients, in particular, has been recently proposed. The present investigation develops methodology, based on the graphical construct, for the representation, analysis, and prediction of creep transients, with special emphasis on the inverse transients of Class I alloys.

11:15 AM
Impression Creep of a Mg-8Zn-4Al-0.5Ca Alloy: Lingling Peng[2]; *Fuqian Yang*[2]; Jian-Feng Nie[3]; James C.M. Li[1]; [1]University of Rochester, Dept. of Mechl. Engrg., Rochester, NY 14627 USA; [2]University of Kentucky, Dept. of Chem. & Matls. Engrg., Lexington, KY 40506 USA; [3]Monash University, Sch. of Physics & Matls. Engrg., Victoria Australia

The creep behavior of a precipitation hardenable Mg-8Zn-4Al-0.5Ca (wt%) casting alloy was determined by using the impression technique in the temperature range 403-623 K and under a punching stress between 1.68 MPa at 623 K and 60.4 MPa at 403 K. The alloy was solution treated for 4 hours at 325°C, water quenched and subsequently aged for 16 hours at 200°C (peak-aged) before the impression tests. Using a power law between the impression velocity and the punching stress, the activation energy is a function of the punching stress and changes from 75.3 kJ/mol at 6.71 MPa to 39.2 kJ/mole at 53.6 MPa. However, by using a hyperbolic sine function between the steady-state impression velocity and the punching stress, a single activation energy is found to be 76.5 kJ/mole, which is about half of the activation energy for lattice diffusion in Mg, 134-139 kJ/mole as summarized by Fujikawa.[1] It suggests that a single mechanism such as grain boundary fluid flow could be rate controlling. This research is supported by NSF through DMR-0211706 monitored by Drs. Guebre Tessema and Bruce A. MacDonald and the Kentucky Science and Engineering Foundation through KSEF-148-502-03-73. [1]S.-I. Fujikawa "Diffusion in Mg" Qin Jin Shu (Light Metals) 42, 822-825 (1992) (in Japanese).

11:30 AM
Predicting Harper-Dorn Creep Rates Using Data on Static Recovery: *Marek A. Przystupa*[1]; Yi Tan[1]; Alan J. Ardell[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095 USA

One of the distinctive features of the dislocation network theory of Harper-Dorn (H-D) creep is that it is capable of predicting steady-state creep rates based only on the knowledge of (1) the steady-state dislocation density and (2) the rates of static recovery of the network. Until now these predictions could not be rigorously tested because of the notorious lack of the high temperature data on static recovery for all materials tested in the H-D creep regime. In this talk we will present our recent high-temperature data on static recovery for polycrystalline aluminum. We show how the data can be used to obtain the important parameters in the dislocation network theory of H-D creep and subsequently used to predict the creep behavior. The resulting predictions of the H-D steady-state creep rates will be then compared with experimental measurements obtained by us and others, as well as with the predictions of the edge dislocation climb model under vacancy-saturated conditions developed over the years by Professor Langdon and his associates.

11:45 AM
Harper-Dorn Creep in Metals at Intermediate Temperatures Revisited: *Lubos Kloc*[1]; Jaroslav Fiala[2]; [1]Academy of Sciences of the Czech Republic, Inst. of Physics of Matls., Zizkova 22, Brno CZ-61662 Czech Republic; [2]Brno University of Technology, Faculty of Chmst., Purkynova 118, Brno CZ-61200 Czech Republic

Harper-Dorn creep, that is creep with apparent stress exponent close to one and independent of grain size have been observed in many metallic materials at temperatures close to the melting point of the material. Moreover, some observations of the similar behavior at intermediate temperatures of about a half of the melting point were interpreted as a "intermediate temperature" Harper-Dorn creep. The micromechanisms behind Harper -Dorn creep were a matter of large debate, but with no clear result. The stress and temperature dependencies of the creep rates derived from the constant stress, constant temperature experiments and ignoring transient "constant structure" effects in fact do not reflect the behavior of underlying micromechanisms directly. The previous results of intermediate temperature H-D creep are reinterpreted and completed with new observations to show that the direct relation between apparent stress exponent and micromechanisms involved in the deformation process is misconception.

12:00 PM
Creep Behavior of Mg-Al Alloys and its Comparison with Al-Mg Aalloys: *Woo-Jin Kim*[1]; Ho-Kyung Kim[2]; [1]Hong-Ik University, Dept. of Matls. Sci. & Engrg., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 S. Korea; [2]Seoul National University of Technology, Dept. of Auto. Engrg., 172 Kongnung-dong, Nowon-gu, Seoul 139-743 Korea

Creep behavior of coarse and fine grained Mg-Al alloys (AZ31) was investigated in a wide strain rate (2x10-7 ~ 7x10-2 s-1) and temperature range (443K ~ 823K) using double shear creep and tensile testing methods. Viscous glide controlled creep, dislocation climb creep and grain boundary sliding arose as the flow rate-controlling process. Effect of temperature, stress, alloying content and grain size on the transition of deformation mechanism was specially focused. The results were compared with those of the Al-Mg alloys of which creep behavior was well-known. The effects of Al addition to Mg and Mg addition to Al on solid drag creep were compared and discussed.

12:15 PM
Role of Grain Boundary Sliding in Creep of Sn-Rich Solder Alloys: Felipe Ochoa[1]; Xin Deng[1]; *Nik Chawla*[1]; [1]Arizona State Uni-

versity, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287 USA

The creep behavior of Sn-rich solder alloys is controlled by two major creep mechanisms: (a) Thermally-induced climb of dislocations over fine intermetallic particles (such as Ag3Sn) and (b) grain boundary sliding (GBS). In this talk, we report on a systematic study on the role of GBS in creep of bulk Sn-3.5 wt% Ag solder alloys at 60°C and 120°C. The solder microstructure was varied by cooling at two different rates: 24°C/s (finer microstructure) and 0.08°C/s (coarser microstructure). Fiducial lines were inscribed on the solder surface and creep tests were interrupted to quantify the evolution of microstructure and strain due to GBS. The microstructure evolution due to creep deformation was characterized using atomic force microscopy (AFM) and scanning electron microscopy (SEM). Measurements of the transverse offset in the fiducial marks twere carried out and the strain due to GBS was quantified. It will be shown that, in general, the strain due to GBS is quite inhomogeneous and is a small fraction of the total creep strain (in the primary and steady-state creep regimes), although the contribution of GBS varies with temperature and microstructure. A comparison of the Sn-rich alloy behavior with that of Pb-Sn alloys (by Langdon and co-workers) will be presented.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Keynote Session

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Monday PM Room: 2009
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Cheml. Engrg. & Matls., E. Lansing, MI 48824 USA; Hamish L. Fraser, Ohio State University, Dept. of Matl. Sci. & Engrg., Columbus, OH 43210 USA

2:00 PM Keynote
Automotive Research: Technical Trends and Challenges: *Alan Taub*[1]; [1]General Motors Corporation, R&D & Planning, Warren, MI 48090 USA

The population of the earth stands above 6.3 billion people today and in another 15 years will approach 7.5 billion. As world population rises, vehicle ownership is also expected to climb dramatically. In order to sustain increasing numbers of vehicles, the automotive industry must address important challenges in several key areas: energy, emissions, safety, congestion, and affordability. This talk will cover General Motors' current strategies on how to address these challenges, highlighting developments in advanced propulsion, vehicle electronics, lightweight and smart materials, and agile manufacturing. These technologies are key to enable the industry to extend the significant benefits of personal mobility to people around the globe.

2:30 PM Keynote
Materials and Manufacturing Challenges for the 21st Century Vehicles: *Subi Dinda*[1]; [1]DaimlerChrysler Corporation USA

Increased demands on safety and fuel economy for future automobiles require innovative technologies for cost effective applications of materials and manufacturing processes. This presentation highlights the strategy of new material applications coupled with novel manufacturing processes at DaimlerChrysler. Rapid emergence of new material demands equally rapid development of cost effective manufacturing processes. The challenges faced by the automotive industry in managing competing priorities of cost reduction, lightweighting, safety enhancement, and customer features will be discussed.

3:00 PM Keynote
Enabling, Enhancing, Empowering - The Role of Propulsion Materials Technologies in the Aerospace Industry: Daniel F. Paulonis[1]; *John J. Schirra*[1]; [1]Pratt & Whitney, 400 Main St., MSC 114-40, E. Hartford, CT 06108 USA

The aerospace industry is in the midst of unprecedented business drivers that have the potential to change its fundamental structure. After experiencing losses of $25B since the beginning of the new century, the industry is restructuring for sustainable, investable growth. Materials technologies will play a key role in this recreation as they enable advances in engine efficiency through increased operating temperatures and higher rotational speeds. Enhancements to airline profitability will be achieved through implementation of lower cost manufacturing processes for spare parts and more effective repair methods. Integration of advanced engineering tools with material damage evolution mechanisms will empower airlines to increase the safe and reliable utilization of their assets while planning for required maintenance. These objectives will be accomplished through sustained investment in fundamental materials research with a focus on engineering solutions, through the use of industry wide collaborative efforts and the implementation of dual use technologies.

3:30 PM Break

3:45 PM Keynote
The Hydrogen Economy - Materials Challenges and Opportunities: *James A. Spearot*[1]; [1]General Motors Corporation, R&D, Warren, MI 48090 USA

Recent debate in both government and technical forums has focused on the value, the possibility, and the timing of meeting future transportation fuel demands by use of hydrogen generated from renewable sources of primary energy. The justifications for and the criticisms against development of renewable energy supplies and hydrogen-fueled propulsion systems are reviewed, and the technical hurdles to be overcome in creating such a future vision are identified. If the vision of a hydrogen-fueled transportation system is to become reality, significant material inventions and developments will be required. The opportunities for critical materials research programs in the areas of hydrogen generation, fuel cell development, and hydrogen storage are described. The status of General Motors' progress in development of hydrogen-fueled, fuel cell-powered vehicles is used to demonstrate the potential that a clean, renewable-hydrogen fuel-based transportation system can provide in meeting societal goals.

4:15 PM Invited
An Overview of Hydrogen Storage for Transportation Applications: *James C.F. Wang*[1]; [1]Sandia National Laboratories, Livermore, CA USA

Hydrogen storage has been one of the highest technical priorities of the DOE Office of Hydrogen, Fuel Cells and Infrastructure Technologies (HFCIT). Currently, no storage technologies meet the hydrogen vehicle requirements. New materials and/or new technical approaches are needed to meet the DOE FreedomCAR hydrogen storage goals for 2010 and 2015. This paper will present an overview of current hydrogen storage technologies and some of the new developments underway in national laboratories, universities and industrial companies. A brief description of Sandia led DOE Center of Excellence for Metal Hydrides will also be presented.

Alumina and Bauxite: Bayer Process Chemistry: Part II

Sponsored by: Light Metals Division, LMD-Aluminum Committee

Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday PM Room: 2005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Robert Bitsch, Alcan, Bauxite & Alumina, Cedex, Gardanne, 13541 France

2:00 PM
Microstructure and Adsorption Behaviour of Sodium Aluminosilicate Polytipes in Sodium Aluminate Liquors: *Jonas Addai-Mensah*[1]; Jun Li[1]; Marek Zbik[1]; William Wilmarth[2]; [1]University of South Australia, Ian Wark Rsch. Inst., Mawson Lakes BLV, Mawson

Lakes, Adelaide, SA 5095 Australia; [2]Westinghouse Savannah River National Laboratory, Savannah River Tech. Ctr., Aiken, SC 29808 USA.

Sodium aluminosilicate (SAS) precipitation from SiO2-supersaturated sodium aluminate solution commonly occurs in alumina refining and high level nuclear waste (HLNW) liquor processing industries. In alumina plant heat exchangers, the precipitation of SAS dimorphs (sodalite and cancrinite) from spent liquors leads to unwanted fouling, impacting dramatically on heat transfer efficiency, liquor throughput and production costs. SAS precipitation fouling of HLNW evaporators may result in radionuclides (e.g. uranium) incorporation, posing a serious criticality concern and additional, technological processing challenge. In the present work, the micro and crystallo-chemical structures of four SAS polytypes (amorphous, zeolite A, sodalite and cancrinite) were determined and correlated with their solution uranium species adsorption behaviour. It was established that SAS phase-specific micro and crystallo-chemical structures play pivotal roles in uranium loading. Under similar conditions, the amorphous phase showed the greatest uranium uptake, with micro/mesoporosity being an important factor governing uranium loading for all 4 SAS adsorbents.

2:25 PM
Polymers as Anti-Scaling Materials in the Bayer Process: *Eoin P. Enright*[1]; Kenneth T. Stanton[1]; John Haines[2]; Teresa Curtin[1]; [1]University of Limerick, Matls. & Surface Sci. Inst., Natl. Technogl. Park, Limerick Ireland; [2]Aughinish Alumina Ltd., Aughinish Island, Askeaton, Co. Limerick Ireland

Scaling within the Bayer process is a widespread problem both in terms of cost of removal and the human risk associated with de-scaling operations. Here we present an overview of the possible use of polymer surfaces in anti-scaling applications. Various strategies may be employed including the use of materials as coatings that either do not scale or have a weak bond at the scale interface such that scale shedding is frequent and predictable. Preliminary results are shown from plant-based experiments where several representative polymeric materials were exposed to the liquor stream in a decanter overflow and scaling rates were measured. Degradation characteristics of these materials in Bayer liquor has been measured using Raman and Fourier-Transform Infrared (FT-IR) spectroscopy and these results are presented and discussed. Microscopy and X-ray analyses are also given.

2:50 PM
Reagents for the Elimination of Sodalite Scaling: Donald Spitzer[1]; Alan Rothenberg[1]; Howard Heitner[1]; Frank Kula[1]; *Morris Lewellyn*[1]; Owen Chamberlain[1]; Qi Dai[1]; Calvin Franz[1]; [1]Cytec Industries, 1937 W. Main St., Stamford, CT 06904 USA

Reagents have been discovered that can greatly reduce or even completely eliminate sodalite scaling in heat exchangers, pipes, etc. in lab, pilot plant, and plant tests. Most importantly, these results are achievable at practical dosages. Extensive downstream testing has not shown any negative effects even in the unlikely event that the reagents get through to precipitation. The reagents are, in fact, adsorbed onto red mud solids and should not accumulate in the circuit.

Alumina and Bauxite: HES and Control & Modelling

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday PM Room: 2005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Jean-Pierre Riffaud, Alumina Partners of Jamaica, Manchester, Jamaica W. Indies

3:15 PM
Studying So2 Emissions in the Calcination Area at Alumar: *Antonio Henrique S. P. Morais*[1]; *Valerio Araujo Gomes*[1]; *Aline Michelle O. Veloso*[1]; *Jonas Uizes Oliveira*[1]; *Julio C. Diniz Costa*[1]; [1]Consórcio de Alumínio do Maranhão - Alumar, EHS, BR-135, Km 18, Estiva, São Luís, Maranhão 65095-050 Brasil

One of the biggest environmental challenges in Alumina Refineries is the air emissions abatement. A proposed production increasing in calcination poses an obvious concern regarding future air emissions, such as SO2, due to the increase in heavy oil consumption. In order to comply with Brazilian regulations and to be aligned to the Alcoa 2020 Environmental Strategy a process evaluation throughout a mass balance was proposed to find out the total %S retained in the Alumina. and the stack emissions rate. The mass balance consisted of isokinetics samplings in the calciner stack, and simultaneously continuous control and measurement of all process parameters such as heavy oil consumption, alumina production, ESP Dust production and the %S in all lines. The main objective was to present the %S absorbed in the Alumina in the direct contact with flue gases and to provide information on which alternative for SO2 abatement should be chosen.

3:40 PM Break

3:55 PM
Experience with 3 x 4500 TPD Gas Suspension Calciners for Alumina: *Jens Fenger*[1]; Charles Wind[1]; Benny E. Raahauge[1]; [1]FFE Minerals Corporation, 77 Vigerslev Alle, Valby 2500 Denmark

In 2001, Queensland Alumina, Australia, launched their environmentally driven expansion project of replacing nine (9) rotary kilns by installing three (3) Gas Suspension Calciners, (GSC), each with a capacity of 4500 TPD. This was the first Calcination Plant ever, for Alumina, in which Stationary Calciners was equipped with Bag Houses for gas cleaning. The commissioning and project experience of the world largest Calcination Plant for Alumina and its impact on the environment will be presented.

4:20 PM
Improved Health and Safety Conditions and Increased Availability in Large Alumina Calcining Units: Hans W. Schmidt[1]; Michael Stroeder[1]; Gajendra Singh[2]; Marcilio Santana[2]; Joaquim Ribeiro[2]; Mike Cable[3]; John Priestrzeniewcz[3]; [1]Outokumpu Technology GmbH, Aluminium Business Sector, Ludwig-Erhard-Str. 21, Oberursel 61440 Germany; [2]Alumina do Norte do Brasil S/A, Km 12 Bacarena - PA Brazil; [3]Worsley Alumina Pty Ltd, Collie, WA Australia

Many existing alumina refineries are being expanded to capacities of more than 3 Mtpy and even up to 4.5 Mtpy. This requires the installation of larger calcination units for economic and operational reasons. For today's alumina calciners the requirements for health protection are on a significantly higher level than 5 years ago. This paper explains how these requirements can be met and in particular what measures can be taken to reduce the level of noise, vibration, heat radiation and dust emission to meet current occupational health and safety standards. At the same time ergonomics has been taken into account. Normal operation, process disturbances and scheduled shut downs of the calciners require diligent process safety attention e.g. for combustion control and dust management. These issues are considered in a HAZOP analysis and subsequently incorporated in operating and maintenance procedures. Whilst over the past decade calcination plants producing 2000 to 2200 tpd represented the largest available units, today typical capacities are in the range of 3000 to 4500 tpd. To maximize the economic value of such large units, the availability factor is one of the most important issues. This paper describes measures to increase the availability factor of these large calcination units to more than 95%. To achieve this, the areas of partial load operation, refractory design, and lifetime of mechanical equipment have been improved and the trip sensitivity and number of moving parts in the calcination system have been decreased.

4:45 PM
Model-Based Digester Temperature Control at Alumina Partners of Jamaica Refinery: *Astley Vincent Forrester*[1]; *Terry Snow*[2]; [1]Alumina Partners of Jamaica, Techl. Dept., PO Box 529, Arabi, LA 70032-529 USA; [2]Comalco Alumina Refinery - Gladstone, Advd. Control, PO Box 1479, Gladstone, Queensland 4680 Australia

The Alpart Alumina Refinery in Jamaica, a Kaiser high temperature design, is a high-energy consumer relative to other plants of similar design. As one part of the energy improvement program a digester temperature control improvement project was implemented with significant improvement in energy consumption. A model-based temperature controller was designed and successfully implemented. The form of the controller is Feed-Forward Predictive with IMC in parallel form for dead-time compensation of model error. Extensive use was made of dynamic simulation using the Simulink system to design and tune the control strategy. The control strategy was found to be robustly stable to various forms of severe process disturbances.

5:10 PM
Application of Advanced Control to Digestion in a Bayer Circuit: *Horace Lawrence*[1]; *Robert K. Jonas*[2]; [1]WINDALCO, Kirkvine Works, Kirkvine PO, Manchester Jamaica; [2]Honeywell, Process Solutions, 2500 W. Union Hills, MS: M15, Phoenix, AZ 85023 USA

Plant operations have been implementing advanced control techniques in the mineral processing industry for several years now. The Bayer process used for alumina refinery is no exception to this, with multivariable predictive control and expert systems being the preferred technologies for increasing throughput and reducing costs. Ultimately this is achieved by exploiting the installed distributed control system (DCS) infrastructure of the refinery. Other potential benefits that a user can get by installing such systems include having a consistent response to process changes and the continuous optimization of the process. This paper discusses the successful application of multivariable predictive control to the digestion circuit at Windalco's Kirkvine Works, what the benefits were and how this technology could be applied to other sections in the Bayer process.

5:35 PM
Mathematic Models of the Seed Precipitation Process of Sodium Aluminate Solution: Liu Chang Qing[1]; Zhang Ping Min[1]; Chen Qi Yuan[1]; Yin Zhou Lan[1]; [1]Central South University, Coll. of Chmst. & Cheml. Engrg., Changsha City, Hunan Province 410083 China

The computer-based process control take a key part in the communication, digitalization, and automatization of alumina production. It is a mathematic model depicting the relations of various factors that affect the alumina production process. Based on the theory of secondary nuclearation and agglomeration dynamics, alumina production process was analyzed. In the "two stages" sand alumina hydroxide production process, the mathematic model of agglomeration process in the "first stage" and secondary nuclearation process in the "second stage" were obtained respectively.

Aluminum Reduction Technology: Cell Development & Operations - Part 1

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday PM Room: 2003
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Knut Arne Paulsen, Hydro Aluminium, Karmøy Primary Production, Karmøy N-4265 Norway

2:00 PM
Current Efficiency in Prebake and Soderberg Cells: *Gary P. Tarcy*[1]; Knut Torklep[2]; [1]Alcoa, Inc, Hall Process Improvement, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA; [2]Elkem Aluminium Research, Fiskaaveien 100, N-4621, Kristiansand Norway

An isotope dilution technique was used to study 24 h current efficiency in 22 semi-randomly selected prebake pots and 21 randomly selected Soderberg pots at the Elkem smelters. A total of 110 measurements were made and compared with the 1128 current efficiency measurements made on Alcoa prebake pots using Alcoa's silver dilution technique. Results are discussed with emphasis on similarities and dissimilarities in prebake and Soderberg. Some theoretical proposals are made regarding the similarities and differences. Results for the prebake pots confirm the basic conclusions from the Alcoa studies including the relative importance of bath chemistry compared to noise (instability) and the impact of low alumina operation on current efficiency. Measurements of 24 h current efficiencies for Soderberg pots are scarce to non-existent. Only temperature and aluminium fluoride have similar influences in the two pot technologies.

2:25 PM
Automated Positioning of Prebaked Anodes in Electrolysis Cells, Part 2: *Jean-Pierre Gagné*[1]; Marc-Andre Thibault[1]; Robin Boulianne[1]; Gilles Dufour[2]; Claude Gauthier[2]; [1]STAS, 1846 Outarde, Chicoutimi, Quebec G7K 1H1 Canada; [2]Alcoa Canada, 1 Place Ville Marie, Montreal, Quebec Canada

During the production of aluminum, the anodes used in electrolysis cells have to be replaced frequently. Even with obtained the best trained operating crew, such operation - given the number of people involved and the methods that are used - is prone to variability and lack of consistency. STAS and Alcoa Canada have been working on developing an automated system for the vertical positioning of anodes in electrolysis cells. At the 2004 TMS Annual Meeting, the first part of this article was presented and the works related to the development and testing of three particular methods were explained. Further to these works, tests were carried out on 36 pots over a time frame of 49 days using one automatic method. The main objective was to verify the effects of this method on the electrolysis process and the reliability of the system. In this paper, we will describe this trial period.

2:50 PM
Anode Height Adjusted Automatically by PTM: *Carlos Adriano Barcellos de Jesus*[1]; [1]Alumar, Pot Rooms, BR 135, km 18, Distrito Industrial de Pedrinhas, São Luís, MA 65095-050 Brasil

Since Alumar start up, anode setting has been a big issue due to safety and hygiene concerns and also due to high level of extra sets. Aiming to carry out theses tasks, Alumar decided to fully reevaluate its anode setting system. Starting from this study Alumar has decided to implement a project which allows automatic anode height adjustment by the pot tending machine (PTM). The system to adjust anode height from the ECL crane cabinet consists basically on an encoder at the crane anode extractor, which converts the extractor movements into electric pulses. The results of this project have shown improvement in the safety and hygiene conditions to pot operator as well as a strong reduction in early anode failures.

3:15 PM
Pre-Heating Optimization Study of Smelting Cells: *Carlos Eduardo Zangiacomi*[1]; Victor Carlos Pandolfelli[2]; Leonardo Paulino[3]; Stephen J. Lindsay[4]; Halvor Kvande[5]; [1]Alcoa Aluminum, Reduction/ Potlining, Rodovia Pocos-Andradas, km 10, Poços de Caldas, Minas Gerais 37701-970 Brazil; [2]Universidade Federal de Sao Carlos, Matls. Sci. & Engrg., Rodovia Washington Luis, km 235, Sao Carlos 13565-905 Brazil; [3]Alcoa Aluminum, Process Engrg./Smelter, Rodovia Pocos-Andradas, km 10, Poços de Caldas, Minas Gerais 37701-970 Brazil; [4]Alcoa Aluminum, Primary Metals, 300 N. Hall Rd., TN 37701-2516 USA; [5]Hydro Aluminum, Drammensveien 264, Oslo, NO NO-0240 Norway

In this paper the author will present data taken from studies performed at Alcoa Poços de Caldas VSS smelter in Minas Gerais, Brazil. The objective of the study was to gain a sufficient grasp of the phenomena taking place during the preheating of a Hall-Heróult cell and then determine the optimum preheating rate and total preheating time required for a better operating performance of the reduction cells. The basis for the work was built upon cathode pre-heating best practices that have been published by Halvor Kvande. In these studies differential temperatures have been compared over time for surface mounted and sub-cathodic thermocouples by using a rigorous numerical-experimental Factorial Test analysis. The author offers conclusions on the experiment test and provides the systemic approach that will address the behavior of other relevant materials leading to the optimized preheating curve.

3:40 PM Break

3:55 PM
Line II Restart Process at ALUMAR - Brazil: *Agnello J.A. Borim*[1]; Eliezer Batista[1]; Elisio Bessa[1]; Sérgio Matos[1]; [1]Consórcio de Alumínio do Maranhão, Potrm., Br 135, Km 18 - Distrito Industrial de Pedrinhas, São Luís, Maranhão 65095-604 Brazil

On July 18, 2003 a flashover occurred at Alumar Line II rectifier room, causing a major blow up and fire which completely destroyed rectifier 23. The adjacent rectifier 22 and the whole control room and cabling system were severely damaged. It is known that both rectifiers had been operating at their nominal capacity until it occurred. As a result of the event, all the pots from line II had to be cut out. Some of them were tapped, but others had all the metal frozen in. So, this big variability of frozen metal layer into the pots was an issue that Alumar had never faced. This paper describes the root causes of the accident, the alternatives to recover the rectifiers, strategies to minimize the production losses under the new rectifier capacity and key aspects to restart the pots as fast as possible and with no incidents.

4:20 PM
The Use of Transversal Slot Anodes at Albras Smelter: *Ronaldo Raposo de Moura*[1]; Hânderson Penna Dias[2]; [1]Albras, Carbon Plant, Rodovia PA 483 Km 21 - Vila Murucupi, Barcarena, Pará 68.447-000 Brazil; [2]Albras, Potlines III & IV, Rodovia PA 483 Km 21 - Vila Murucupí, Barcarena, Pará 68.447-000 Brazil

The present article discusses the results obtained at Albras potlines with transversally slotted anodes. It is commonly believed in smelters that longitudinal oriented slots are more beneficial in pot operation when compared to transversal slots. However, due to the Albras green anode compactor design it was only possible to make longitudinal slots by cutting the anodes after baking. In 2002 Albras started experimenting the slotted anodes of both types in a few pots. As a result the use of

transversal slotted anodes was extended to two 30 pots sections. Data obtained over a period of six months on real pot resistance, anode effect frequency, anode deformation (spikes), noise, anode scrap levels and butt cracking incidence are presented and analyzed. Observations are made on how spike formation may be avoided. Finally the savings and benefits obtained from the use of this type of anode are evaluated.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Simulation & Control of Carbon Nanotube Formation

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Monday PM Room: 3018
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Jud Ready, Georgia Tech, GTRI-EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, College Park, MD 20742-2115 USA

2:00 PM Opening Remarks

2:05 PM Invited
Designing Carbon-Based Nanotechnology on a Supercomputer: *David Tománek*[1]; [1]Michigan State University, Physics & Astron. Dept., E. Lansing, MI 48824-2320 USA

The continuous reduction of device sizes, which is rapidly approaching the atomic level, calls for new approaches to design and test future building blocks of Nanotechnology. Computer modeling is an indispensable tool to interpret phenomena associated with the nanometer scale. A plethora of unexpected phenomena is displayed by carbon nanostructures. Due to the stability of the sp^2 bond, carbon nanotubes are thermally and mechanically extremely stable and chemically inert. They contract rather than expand at high temperatures, and are the ultimate thermal conductors. At the same time, nanotubes may be tuned into ballistic electron conductors or semiconductors. sp^2 bonded nanostructures may change their shape globally by a sequence of Stone-Wales transformations. Specific nanotube assemblies may even acquire a permanent magnetic moment. In nanostructures that form during a hierarchical self-assembly process, even defects may play a different, often helpful role. Efficient self-healing processes may convert less stable atomic assemblies into other, more perfect structures, thus answering an important concern in molecular electronics. Defects may even be used in nano-scale engineering to form complex systems such as carbon foam or nanotube peapods. Unusual behavior is expected of devices based on nanotubes, including nonvolatile computer memory elements or nanoVelcro. In this presentation, I will show how some of these challenging problems can be most efficiently addressed in simulations on recently available massively parallel supercomputers.

2:35 PM Invited
Molecular Dynamics Study of Catalyzed Single-Walled Carbon Nanotube Nucleation: *Kim Bolton*[1]; Feng Ding[1]; Arne Rosen[1]; [1]Göteborg University and Chalmers University of Technology, Sch. of Physics & Engrg. Physics

Molecular dynamics simulations based on an empirical potential energy surface were used to study iron catalyzed nucleation and growth of narrow single-walled carbon nanotubes (SWNTs). The simulations show that small (ca. 1 nm) iron particles are in the liquid phase at experimental growth temperatures, and that SWNTs grow from these particles at temperatures between 800 and 1400 K. In contrast, at temperatures below 600 K graphene sheets encapsulate the particle, and above 1600 K a three-dimensional (3D) soot-like structure is formed. Nucleation of these carbon (C) structures can be divided into three stages: i) at short times all C atoms dissolve in the iron-carbide (FeC) particle, ii) at intermediate times the FeC cluster is highly supersaturated in C and carbon strings, polygons and small graphitic islands nucleate on the cluster surface, iii) at longer times the FeC cluster is supersaturated in C and a graphene sheet, SWNT or soot-like structure is grown. The simulation shows that the growing SWNT maintains an open end on the FeC cluster due to the strong bonding between the nanotube end atoms and the cluster. These strong interactions also yield SWNTs that have similar diameters to the FeC particles. The growth mechanism on larger particles, which can be in the solid phase under experimental conditions, is similar to that described above, although C atoms show only limited diffusion into the cluster before adding to the end of the growing SWNT.

3:05 PM Break

3:30 PM Invited
Controlled Geometry of Carbon Nanotubes: *Sungho Jin*[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

By virtue of their exceptional electrical and mechanical properties, carbon nanotubes (CNTs) have been studied for many exciting, new applications such as electron field emitters, nano-scale manipulators, field-effect transistors, nano-interconnections, AFM probes, and bio or chemical interactions. In order to successfully utilize CNTs for such applications, the control of their size and morphology is essential. In this talk, various growth and microstructural controls of diameter, length, alignment, bending, and cutting of nanotubes will be discussed. Growth of patterned nanotubes or sharp nanocone arrays, multiple sharp bending of nanotubes for creation of zig-zag nanotubes, nano-scale shortening and opening of nanotubes, as well as coating or filling of nanotubes for nanocomposite formation will be described in relation to their potential applications.

4:00 PM Invited
Chirality Separation and Tuning the Electronic Structures of Carbon Nanotubes: *Young Hee Lee*[1]; [1]Sungkyunkwan University, Physics, Suwon, Kyungki 440-746 S. Korea

It is of crucial step to control the electronic structures of carbon nanotubes. The best way is of course to control the chirality during the synthesis, which is currently not available. One alternative is to separate nanotubes with specific chirality. We introduce gas adsorbates that lead to a selective etching of the nanotube edges. Among various types of simple gases, we found that it is only carbon dioxide that leads to a selective adsorption followed by a selective etching of zigzag nanotubes. We will introduce various approaches to choose either metallic or semiconductign nanotubes and propose a general strategy for chirality separation.

4:30 PM Invited
Growth and Properties of Carbon Nanotubes and Semiconducting Oxide Nanowires or Nanobelts: *A. M. Rao*[1]; [1]Clemson University, Dept. of Physics & Astron., Clemson, SC 29634 USA

One-dimensional nanostructures such as carbon nanotubes, semiconducting oxide nanowires or nanobelts exhibit unique fundamental properties that can be useful in several applications. I'll begin my talk by introducing a simple thermal CVD process in which doped nanotubes, and semiconducting oxide nanowires or nanobelts can be prepared in bulk quantities. Carbon nanotubes were prepared on bare quartz or oxidized silicon substrates using our thermal CVD process in which a liquid precursor, such as xylene, is used as the carbon source. Highly controlled doping of carbon nanotubes with nitrogen was accomplished by mixing appropriate amount of acetonitrile with xylene. The effect of nitrogen doping on the electronic and mechanical properties of carbon nanotubes will be presented. In addition to the synthesis and doping of carbon nanotube, nanowires and nanobelts of oxides, sulfides and nitrides of low melting metals such as gallium, indium and tin were also prepared using our CVD system. These nanostructures were synthesized using metal films and corresponding reactant gases at ambient pressure in a catalyst-free environment. Some of the fundamental properties of these nanostructures monitored using Raman spectroscopy, SEM, high resolution TEM, and vibrating reed measurements will be presented.

5:00 PM Invited
Carbon Nanotubes and Silicon Nanowires: *Z. F. Ren*[1]; [1]Boston College, Dept. of Physics, Chestnut Hill, MA USA

The carbon nanotubes aligned periodically have many potential applications in electronics, optics, etc. In this talk, I will discuss the aligned carbon nanotubes grown by plasma enhanced hot filament chemical vapor deposition on Ni films by magnetron sputtering, Ni dots by e-beam lithography, Ni dots by electrochemical deposition, and two-dimensional periodical nickel dot array by nanosphere self-assembly. The size of the nickel dot and spacing between them and location are tunable by the preparation methods. Two new techniques were developed to make triangular Ni dots and aligned carbon nanotubes. With these arrays, a variety of applications have been studied includ-

ing field emission, nanoelectrode, photonic band gap crystals, etc. In addition, I will talk about our recent studies on synthesis of silicon nanowires.

5:30 PM
Alignment of Carbon Nanotubes via Electrically Biased Thermal Filament Chemical Vapor Deposition: *Stephan Parker Turano*[1]; [1]Georgia Tech Research Institute, 925 Dalney St., Baker Rm. 114, Atlanta, GA 30332 USA

The focus of this research is to synthesize aligned carbon nanotubes (CNTs) on silicon substrates using transition metal catalysts such as Fe and Ni. CNTs are synthesized using thermal chemical vapor deposition (CVD) with temperature ranging between 700 and 900°C. In addition, an electric field was created in the deposition chamber using tungsten filaments under an electric bias. Results show that a significant amount of nanotube alignment occurs during the synthesis process. Coiled nanotubes, and nanotubes which exhibit perfect 90° bends, are also present in samples which are subjected to an electric field. This work suggests that CNT orientation can be controlled during synthesis and that CNTs may eventually be synthesized directly into electronic circuits or other devices.

Arsenic Metallurgy: Fundamentals & Applications: Removal of Arsenic and its Precipitation from Process Streams I

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Monday PM Room: 2014
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: George P. Demopoulos, McGill University, Metals & Matls. Engrg., Montreal, Quebec H3A 2B2 Canada; Tina Maniatis, Applied Biosciences, Salt Lake City, UT 84152 USA

2:00 PM
Arsenic Removal from Mine and Process Waters by Lime/Phosphate Precipitation: *Larry G. Twidwell*[1]; Jay McCloskey[2]; Michelle Gale Lee[3]; Jennifer Saran[4]; [1]Montana Tech of University of Montana, Sch. of Mines & Engrg., 1300 W. Park St., Butte, MT 59701 USA; [2]MSE-Technology Applications, Process Engrg., 200 Technology Way, Butte, MT 59701 USA; [3]MSE-Technology Applications, QA, 200 Technology Way, Butte, MT 59701 USA; [4]Kennecott Copper, Salt Lake City, UT 84044 USA

The removal of dissolved arsenic from mine wastewaters utilizing a lime/phosphate precipitation technique has been studied on a laboratory scale at Montana Tech of The University of Montana and on a pilot scale at MSE-Technologies Applications as a wastewater treatment process potentially capable of removing dissolved arsenic to <10 ug/L. Arsenic bearing lime and lime/phosphate slurries were subjected to laboratory extended time air-sparged aging for over four years. The lime slurries released their arsenic back into solution in a relatively short period of time, whereas the lime/phosphate slurries showed limited redissolution of arsenic. The developed process has been pilot scale tested by MSE-TA for the EPA Mine Waste Technology Program on two waters, ASARCO smelter blowdown water and Mineral Hill groundwater. The laboratory study results will be presented and discussed in this presentation. The pilot scale results will be presented and discussed in a companion presentation.

2:25 PM
Arsenic Removal from Mine and Process Waters by Lime/Phosphate Precipitation: Pilot Scale Demonstration: *Jay McCloskey*[1]; Michelle Gale Lee[1]; Larry G. Twidwell[2]; [1]MSE Technology Applications, Process Engrg., 200 Tech. Way, Butte, MT 59701 USA; [2]Montana Tech of University of Montana, Sch. of Mines & Engrg., 1300 W. Park St., Butte, MT 59701 USA

The removal of dissolved arsenic from mine wastewaters utilizing a lime/phosphate precipitation technique has been studied on a laboratory scale at Montana Tech of The University of Montana and on a pilot scale at MSE-Technology Applications as a wastewater treatment process potentially capable of removing dissolved arsenic to <10 ug/L. The developed process has been pilot scale tested by MSE-TA for the EPA Mine Waste Technology Program on two waters, ASARCO smelter blowdown water and Mineral Hill groundwater. Two other technologies were evaluated at the same time for comparison, e.g., alumina adsorption and ferrihydrite adsorption. The pilot scale results will be presented and discussed in this presentation. The background laboratory studies are presented in a companion presentation.

2:50 PM
Meeting Arsenic Standards in Drinking Water by January 2006: A Review of Available Technologies: *V. Ram Ramachandran*[1]; Ramesh Narasimhan[2]; [1]Consulting Engineer, 9650 E. Peregrine Place, Scottsdale, AZ 85262 USA; [2]Narasimhan Consulting Services, 3150 N. 24th St., Ste. D-104, Phoenix, AZ 85016 USA

Arsenic occurs naturally in rocks and soil, water, air, plants and animals. It can be further released into the environment through natural activities such as volcanic action, erosion of rocks and through human actions. Higher levels of arsenic tend to be found more in ground water sources than in surface water sources of drinking water. Compared to the rest of USA, western states have more systems with arsenic levels greter than 10 parts per billion(ppb). The US Environmental Protection Agency(EPA) has historically regulated arsenic in drinking water at 50 ppb, but the agency will lower the maximum contaminant level(MCL) for arsenic to 10 ppb by January 2006. This paper reviews briefly a)arsenic chemistry and removal mechanisms and b)the available treatment processes to attain the drinking water standard of 10 ppb by 2006. The combining of an appropriate removal method with an existing system depends on several factors such as nature of arsenic, the water source, existing plant equipment and processess, site conditions etc. The paper also includes one or two case studies from the State of Arizona.

3:15 PM
Coprecipitation of Arsenic with Fe(III), Al(III) and Mixtures of Both in a Chloride System: Robert G. Robins[1]; *Pritam Singh*[1]; Radhanath P. Das[2]; [1]Murdoch University, Div. of Sci. & Engrg./Chmst. Dept., Murdoch, WA 6150 Australia; [2]Regional Research Laboratory, CSIR/Dept. of Hydrometall., Bhubaneswar, Orissa 751013 India

The removal of arsenic from hydrometallurgical process and effluent streams and also from drinking water, has among other methods, utilised chemical coprecipitation with Fe(III) and Al(III) for many decades. It is considered that As(V) is the oxidation state that is most efficiently removed by chemical coprecipitation. The literature reports many studies in which an aqueous solution of either Fe(III) or Al(III) was the reagent that had been investigated for coprecipitation of As(V). There is argument as to whether Fe(III) or Al(III),used separately, is the most effective reagent for coprecipitation, and also concerning the optimal M(III):As(V) ratio for best removal. Many misconceptions have evolved from published work and this paper will address some of those and present experimental compari sons in evidence, More importantly we report the superior results from using a mixed Fe(III)-Al(III) reagent for coprecipitation of As(V), rather than the individual Fe(III) or Al(III) solutions separately.

3:40 PM Break

3:55 PM
The Effect of Silicate on the Adsorption of Arsenate on Co-Precipitated Ferrihydrite: *Pritam Singh*[1]; Wensheng Zhang[1]; David M. Muir[2]; Robert G. Robins[1]; [1]Murdoch University, Div. of Sci. & Engrg./Chmst. Dept., Murdoch, Western Australia 6150 Australia; [2]CSIRO Minerals, PO Box 90, Bentley, Western Australia 6982 Australia

Dissolved silicate interferes with the removal of As(V) from aqueous solutions by co-precipitation/adsorption on ferrihydrite. It causes an increase in the residual As(V) and Fe(III) in solution. The effect is a function of initial silicate concentration and pH of the solution from which co-precipitation/adsorption occurs. In the presence of silicate, the residual As(V) in water increases rapidly at pH>5. For a solution with initial 1 mg/L As and 30 mg/L Fe(III), when silicate is doubled from 20 to 40 mg/L, the coprecipitation/adsorption of As(V) on ferrihydrite at pH 7 results in five times higher residual As(V) (0.05 to 0.28 mg/L). At the same time, the residual Fe(III) in solution is doubled (approximately 4 to 8 mg/L). It is proposed that the silicate effect is due to a combination of complexation reactions between Fe(III), Si(IV) and As(V) species, and competition between As(V) and Si(IV) for adsorption sites on ferrihydrite.

4:20 PM
Coprecipitation of As(V) with Fe(III) in Sulfate Media: Solubility and Speciation of Arsenic: *Yongfeng Jia*[1]; Ning Chen[2]; George P. Demopoulos[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada; [2]Canadian

Light Source Inc., University of Saskatchewan, 101 Perimeter Rd., Saskatoon, SK S7N 0X4 Canada

Lime neutralization and co-precipitation of As(V) with iron(III) is the best and most economical method for the removal of arsenic from mineral processing solutions and effluents. However, the arsenate fixation mechanism in the coprecipitated solid is not fully understood. It was suggested by some researchers to be a ferrihydrite precipitation process during which arsenate is removed by adsorption on ferrihydrite. In this study, XRD, EXAFS and microprobe were employed for the speciation of arsenic and the effect of various factors were also investigated. The results indicate that the coprecipitation is a more complicated process than simple adsorption. Poorly crystalline ferric arsenate was observed for the first time to be present in the coprecipitated solid. In case lime as base, there exists some type of Ca-Fe-arsenate association. A general term "coprecipitate" is suggested to describe the precipitated arsenic-bearing solid instead of those widely used specific terms "arsenical ferrihydrite" or "arsenic-bearing ferrihydrite".

4:45 PM
Arsenic in Yellowknife, NWT, Canada: *W. R. Cullen*[1]; K. J. Reimer[2]; C. Ollson[3]; [1]University of British Columbia, Chmst. Dept., Vancouver, BC Canada; [2]Royal Military College of Canada, Environmental Scis. Grp., Kingston, Ontario Canada; [3]Jacques Whitford Environmental Limited, Ottawa, Ontario Canada

The Giant Mine began gold production in Yellowknife, Northwest Territories, Canada, in 1948. Soon after this is was decided to store the arsenic trioxide dust from the smelter underground. The current situation is that the mine has now ceased operations leaving 260,000 tons of 78% by weight arsenic trioxide dust stored in 15 underground chambers. The first part of the talk will outline the management options that include leaving the dust in place or removing it to the surface where further processing or stabilizing would be required. The processing of gold ore at both mines – the Giant and Con – in Yellowknife has resulted in tailings ponds and surface soils containing high concentrations of arsenic. This has added to a naturally elevated arsenic concentration in the area. Closure plans require the development of practical and cost-effective ways of mitigating the environmental impact of this contamination. The first step in such a process is to identify the actual environmental and human health risks. The second talk will describe the use of laboratory techniques (gastric fluid extraction) that simulate exposure scenarios. The results of these studies suggest that the risk is much less than would have otherwise been expected and could form the basis of remediation plans costing much less than would otherwise be the case.

Beta Titanium Alloys of the 00's: Applications II

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Monday PM Room: Salon 10/11
February 14, 2005 Location: San Francisco Marriott

Session Chairs: Rodney R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124 USA; K. O. Yu, RMI Titanium Company, Niles, OH 44446-0269 USA

2:00 PM
Recent Developments in Beta Strip Alloys: *John C. Fanning*[1]; [1]TIMET, PO Box 2128, Henderson, NV 89009 USA

The strip-producibility, good fabricability and excellent mechanical properties of beta alloys make them useful for a variety of fabricated sheet metal structures on airframes and aeroengines. In particular, TIMETAL 15-3 is used for ECS (Environmental Control System) ducting on the Boeing 777 and Airbus A380. For applications that require exposure to higher temperatures, such as the plug&nozzle assemblies on various civil and military aircraft, TIMETAL 21S is now widely used. This paper will summarize recent developments in the properties and processing of these alloys with regards to aerospace and other applications.

2:30 PM
Properties and Processing of TIMETAL® LCB: *Yoji Kosaka*[1]; Stephen P. Fox[1]; Kurt Faller[2]; Steven H. Reichman[2]; [1]TIMET, Henderson Techl. Lab., PO Box 2128, Henderson, NV 89009 USA; [2]TIMET Automotive, 900 Hemlock Rd., Morgantown, PA 19543 USA

TIMETAL® LCB was introduced more than 10 years ago targeting automotive suspension spring applications. The alloy development aim was to use a low cost formulation by selecting less expensive raw materials than typical beta titanium alloys. Following the first successful application of TIMETAL® LCB suspension springs to series production vehicles, the 2000 Volkswagen Lupo FSI, the springs have been used for Ferrari Challenge Stradale since 2003. The most recent development effort has been focused on the generation of metallurgical and mechanical service data of the alloy, and the establishment of low cost processing of coils and bars. This paper will introduce various properties of TIMETAL® LCB products that can be useful in the design and the consideration of spring applications. Recent progress in the processing of TIMETAL®LCB will also be introduced and discussed.

2:55 PM
Candidate Materials for Aerospace and Automotive High Strength Fastener Applications: *James G. Ferrero*[1]; [1]Perryman Company, 213 Vandale Dr., Houston, PA 15342 USA

There are many commercially available titanium alloys that have exhibited the capability of achieving high strengths. Many of these alloys have not been seriously considered for fastener applications due to their cost or availability as coil or bar. However, as new designs, increased material requirements and larger aircraft are being built the need to reduce weight and improve performance continues to be a major issue. The possibility of reducing weight by replacing currently used steel fasteners in various sizes is a great incentive. Over the past few years, many of these alloys have been processed to bar and coil product to evaluate there capabilities as potential fastener materials. This paper will review and summarize the mechanical properties, tensile, shear, notch tensile and available fatigue, as well as the microstructure of these candidate alloys.

3:25 PM Break

3:40 PM
Ti-200: A Titanium Alloy Developed for 200 ksi (1380 MPa) Applications: *Paul Bania*[1]; [1]TiPro LLC, 1011 Industrial Rd., Ste. #5, Boulder City, NV 89005 USA

A new titanium alloy has been developed by Ti-Pro LLC - designated "Ti-200" - for applications requiring tensile strengths at or above 200 ksi (1380 MPa). This paper will review the experimental studies leading to the final chemistry selection as well as some preliminary properties of the new alloy. Applications for the alloy include various small forgings/machined parts for automotive racing applications as well as high strength titanium fasteners.

4:05 PM
Mechanical Properties of Beta Processed Ti-17: *Andy Woodfield*[1]; [1]General Electric Aircraft Engines, Neumann Way, MD M-89, Cincinnati, OH 45215 USA

Abstract not available.

4:35 PM
Microstructure-Properties of Beta Titanium Alloy Castings: *E. Y. Chen*[1]; D. R. Bice[1]; [1]TiTech International, Inc., PO Box 3060, Pomona, CA 91769-3060 USA

The number of applications of titanium castings has expanded greatly in the last decade due to factors such as improved casting quality and mechanical properties as well as better shape-making capabilities. The bulk of the titanium casting manufactured has remained Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo, and CP-Ti. Beta titanium alloys have been widely used as forgings, but much less so as castings despite the potential benefits. This study investigates the castability and microstructure-properties of several beta and alpha + beta titanium alloys including Ti-10V-2Fe-3Al and Ti-15V-3Al-3Cr-3Sn. A comparison of the mechanical properties of the beta castings versus those of their forged counterparts will also be made.

Biological Materials Science and Engineering: Biological Materials II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Monday PM Room: 3009
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: George Mayer, University of Washington, Dept. of Matls. Sci. & Engrg., Seattle, Washington USA; A. G. Evans, University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

2:00 PM Invited
Mechanisms Governing the Inelastic Deformation of Bone: *A. G. Evans*[1]; C. Mercer[1]; P. Hansma[1]; M. He[2]; [1]University of California, Santa Barbara, CA USA

To understand the inelastic response of cortical and trabecular bone, a three-part investigation has been conducted. In the first, a flexural test protocol has been designed and implemented that monitors the axial and transverse strains on both the tensile and compressive surfaces of cortical bone. The results are used to assess the relative contributions of dilatation and shear to the inelastic deformation. A deconvolution has been used to affirm that the stress/strain curves in tension and compression are consistent with results in the literature. Unload/reload tests have characterized the hysteresis and provided insight about the mechanisms causing the strain. In the second part, a constitutive law representative of the deformation is selected. To demonstrate consistency, it is first used to reconstruct the flexural response. Thereafter, it is implemented to illustrate the coupled buckling and bending of ligaments that occurs in osteoarthritic trabecular bone loaded in compression. Stress/strain curves are calculated and shown to be consistent with measurements reported in the literature. The third part devises a model for the intrinsic stress/strain response of bone, based on a recent assessment of the nano-scale organization of the collagen fibrils and mineral platelets. The model is used to rationalize the inelastic deformation in tension, as well as the permanent strain and the hysteresis.

2:30 PM Invited
Growth, Structure, and Mechanical Response of Abalone Shells: *Marc Andre Meyers*[1]; Albert Lin[1]; Julie Muyco[1]; [1]University of California, Matls. Sci. Prog., La Jolla 92093-0411 USA

The growth of the nacreous layers (aragonite) was observed by inserting glass plates in the extrapallial layer for different time periods, removing them, and observing them by scanning electron microscopy. Details of the growth sequence were revealed. The mechanical properties and structure of mineral and organic phases was characterized by nanoindentation and atomic force microscopy.Individual protein chains were identified in the nanoorganic (~25 nm thick)layer between the aragonite tiles. This layer presented holes with a diameter of approximately 40 nm. Quasi-static compression and three-point bending tests were carried out. The mechanical response of the abalone is correlated with the microstructure and damage mechanisms. The mechanical response is found to vary significantly from specimen to specimen and requires the application of Weibull statistics in order to be quantitatively evaluated. The abalone exhibited orientation dependence of strength: the compressive strength when loaded perpendicular to the shell surface was approximately 50% higher than parallel to the shell surface. The compressive strength of abalone is 1.5 - 3 times the tensile strength (as determined from flexural tests), in contrast with monolithic ceramics, for which the compressive strength is typically an order of magnitude greater than the tensile strength. Quasi-static compressive failure in both shells occurred gradually, in a mode described as “graceful failure”. The shear strength of the organic/ceramic interfaces was determined by means of a shear test and was found to be approximately 30 MPa. Considerable inelastic deformation of these layers (up to a shear strain of 0.4) preceded failure. The mechanical response of the organic nanolayers and mesolayers (growth bands) determines the high tensile strength of the shell and its associated high toughness. Crack deflection, delocalization of damage, plastic microbuckling (kinking), and viscoplastic deformation of the organic layers are the most important mechanisms contributing to the unique mechanical properties of these shells.

3:00 PM Invited
Biomimetic Mineralization of Printed Polymer Gels: *Paul Calvert*[1]; [1]University of Massachusetts, Dept. of Textile Scis., N. Dartmouth, MA 02747 USA

The factors controlling biomineralization are not well understood in most cases. Nucleation on organic surfaces is believed to be important in some cases, in others nucleation occurs within a gel. Inhibitors are present which may control the crystal morphology or totally prevent growth. In some cases mineralization seems to arise from a localized source of the inorganic ions. In this work we have explored the role of immobilized enzymes in promoting mineralization of polymer gels. Printing methods were used to deposit gels with thicknesses from less than one micron up to hundreds of microns. We show that heavily mineralized gels can be formed in this way if they are thick enough that precipitation predominates over outward diffusion of the ions. The role of the gel matrix material in the process will also be discussed.

3:30 PM Break

3:45 PM Invited
Nanomechanical Characterization of Nanoscale Biomaterials: *Chwee Teck Lim*[1]; [1]National University of Singapore, Bioengrg. & Mechl. Engrg., 9 Engrg. Dr. 1, Singapore 117576

Much interest has been generated recently in the use of nanostructured biomaterials for tissue engineering so as to create biological alternatives for implants. Biodegradable polymers in the form of highly porous nanofibrous scaffolds are ideal in accommodating cells and guiding their growth for tissue regeneration in three dimensions. Besides biocompatibility, suitable porosity and sufficient surface area for cell attachment, these polymeric scaffolds should also have the structural integrity and mechanical strength to maintain the desired shape before the new tissue takes over. In fact, studies have shown that the mechanical constraints imposed on cells seeded in scaffolds can affect cell growth, migration and differentiation. Therefore, there is a need to study the structural and nanomechanical properties of individual nanofibers that make up the entire scaffold. However, due to the difficulty in isolating and manipulating individual nanofibers and measuring the very small force and deformation involved, little work has been done in this area. This talk will highlight some of the challenges as well as innovative experimental techniques that we have developed in our research group to investigate the nanomechanical properties of these nanoscale biomaterials.

4:05 PM
Dynamic Fracture of Bovine Cortical Bone: A Systematic Study of Strain Rate Effects: *Raghavendra R. Adharapurapu*[1]; Kenneth S. Vecchio[1]; Fengchun Jiang[1]; [1]University of California, Dept. of MAE, Matls. Sci. & Engrg. Grp., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

True clinical fracture of bones in bovine, race horses or humans occur predominantly during impact loading (for e.g. car accidents, falls or physical violence). Although, static fracture tests provide an estimate of fracture toughness or R-curve behavior in bones, the static toughness values may be ill suited for predicting failure under dynamic loading conditions due to the visco-elastic response of bone (i.e. strain rate dependent properties). Despite decades of the study on deformation rate dependency of cortical bone properties such as compression and fracture toughness, high quality dynamic fracture toughness data remains limited. Preliminary tests (compression and fracture toughness) have been conducted on dry and wet bovine cortical bone under both static and dynamic loading conditions with loading parallel and perpendicular to the bone axis (longitudinal and transverse respectively). The strain rate in compression tests varied between 10^{-3} to $10^{3}s^{-1}$, and the stress intensity rate varied between 10^{-3} to 10^{6} MPa-√m/s. While low strain rate tests were conducted on conventional mechanical testing machines, high strain rate experiments were conducted on a split-Hopkinson bar under compression and a novel three-point bend configuration. The influence of wetting agent on the failure properties was also investigated by testing bone exposed to a salt solution or acetone for different periods. The fracture morphology and the extent of microcracking of bone in each case were characterized using SEM, and an attempt is made to relate it to the rate dependency of fracture toughness of the cortical bone. Interpretation of the fracture mechanisms is conducted in terms of structural attributes of bone, such as the composite nature of bone with hierarchical structure

over different length scales that produce strong materials from weak constituents. It is believed that such understanding is crucial for mechanistic interpretation of cortical bone fracture phenomenon and eventually for predicting bone failure reliably.

4:25 PM
Creation of Nanostructured Hydroxyapatite (Synthetic Bone) by Hydrothermal Conversion of Seashells: *Kenneth S. Vecchio*[1]; Xing Zhang[1]; [1]University of California, Dept. of MAE, Matls. Sci. Grp., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

Hydroxyapatite is a bioactive material and the main component of human bone. Due to its biocompatibility and osteoconductive properties, it has many applications, such as bone graft substitutes, sustained-release drug delivery devices, purification of proteins, etc. Many of these medical implant applications require highly densified implants with mechanical strength high enough for load bearing. The major hurdle to synthesis of synthetic bone from calcite is the difficulty of making dense forms of hydroxyapatite, structured in a manner that impart good mechanical properties. On the other hand, shells and marine bones, which are composed of aragonite/calcite crystals, have dense, tailored structures that impart excellent mechanical properties to these structures. Seashells, such as conch, clams, and abalone, have hierarchical and optimally designed architectures with similar mechanical properties as hard bone. We have recently developed a way to convert the aragonite/calcite crystals within the shell to hydroxyapatite crystals, making the possibility of dense, well-structured, synthetic bone a realizable goal. This project is focusing on optimizing the hydrothermal conversion process to convert the aragonite/calcite to hydroxyapatite, while maintaining the architecture of the shell and marine bone structures. The applications for such synthetic bones include dental implants, biocompatible prosthetic interfaces, bone replacements and many other medical implants.

4:45 PM
Phase Transformation of the Nano-Sized Hydroxyapatite Synthesized by Hydrolysis Using In-Situ High Temperature X-Ray Diffraction: *Wei-Jen Shih*[1]; Jian-Wen Wang[2]; Moo-Chin Wang[3]; Min-Hsiung Hon[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., 1 Ta-Hsueh Rd., Tainan 70101 Taiwan; [2]Chung Hwa College of Medical Technology, Dept. of Environml. & Safety Engrg., 89 Wen-Hua 1st St., Rende Shiang, Tainan 71703 Taiwan; [3]National United University, Dept. of Matls. Sci. & Engrg., 1 Lien-Da, Kung-Ching Li, Miao Li 360 Taiwan

The biodegradable hydroxyapatite (HA) was synthesized by hydrolysis and characterized using high temperature X-ray diffraction (HT-XRD), differantial thermal analysis (DTA), thermogravimetry (TG), scanning electron microscopy (SEM), and FT-IR spectrometer. The in-situ phase transformation of nano-sized HA synthesized from $CaHPO_4$¡E$2H_2O$ (DCPD) and $CaCO_3$ with a Ca/P=1.5 in 2.5 M $NaOH_{(aq)}$ at 75°C for 1 h was investigated by HT-XRD from RT to 1500°C. The synthetic HA recrystallized at 600°C and maintained as the major phase until 1400°C. The HA steadily transformed to α-tricalcium phosphate α-TCP) and at 1500°C α-TCP became the major phosphate phase. The minor CaO phase appears at the temperature of 750°C due to the decomposition of CaCO3 and vanished at 1300°C. The Na^+ impurity from the hydrolysis process was responsible for the formation of $NaCaPO_4$ phase, appearing above 700°C and disappeared at 1300°C.

5:05 PM
Nucleation, Growth, Characterization and Biocompatibility of Biomimetic Apatite Layers Formed on Titanium Alloy: K. C. Baker[2]; M. A. Anderson[1]; S. A. Oehlke[1]; A. I. Aastachkina[2]; D. C. Haikio[2]; *J. Drelich*[1]; S. W. Donahue[2]; N. S. Istephanous[3]; [1]Michigan Tech, Matls. Sci. & Engrg., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Michigan Tech, Biomed. Engrg., Houghton, MI USA; [3]Medtronic USA, Inc., Matls. & Bioscis. Ctr., 710 Medtronic Pkwy., Minneapolis, MN 55432 USA

This study focuses on the growth, characterization and biocompatibility of bone-like apatite layers on Ti-6Al-4V alloy produced through diminished time duration chemical immersion. The method presented combines a pre-calcification step, in which samples were immersed in a boiling Ca(OH)2 solution to initiate and increase ion exchange. Subsequent immersion in a supersaturated calcium-phosphate solution (SCPS) produced homogenous coatings with thicknesses of 15-20 microns, 100% coverage and crystal sizes of 1-2 microns in as little as 4 days. Coated samples were favored biologically over non-coated samples by cultured osteoblast cells as evidenced through alkaline phosphatase activity assays. This study asserts that an industrially viable method of chemical immersion in a SCPS, coupled with simple pre-treatments can produce apatite coatings that favor positive biological interactions, conducive to osseointegration.

5:25 PM
A Biomimetic Approach to the Deposition of Zirconia Films on Self-Assembled Nanoscale Templates: *Junghyun Cho*[1]; [1]SUNY Binghamton, Mechl. Engrg., T. J. Watson Sch. of Engrg., PO Box 6000, Binghamton, NY 13902-6000 USA

A biomimetic synthesis technique is employed to deposit ceramic films on nanoscale organic materials. Specifically, phosphonate-based self-assembled monolayers (SAM) are used as the organic template, onto which a ZrO_2 film is grown in situ in an aqueous solution at near room temperature (≈80°C). This process mimics biomineralization involving the controlled nucleation and growth of the inorganic (ceramic) materials. It is shown that surface functionality of the SAM plays a crucial role by: i) providing surface nucleation sites for the covalently-bonded ceramic film, and ii) promoting electrostatic attraction between the SAM surface and the colloidal clusters or particles precipitated in the solution. The resultant zirconia films consist of sub-micron sized particles that are formed by an enhanced hydrolysis of zirconium sulfate. The mechanisms of film formation are systematically studied by tailoring the film structure from solution chemistry and SAM functionalities. In particular, the cross-sectional TEM work is performed to quantitatively analyze the film structure as well as interfacial region of the biomimetic ceramic films. Further, the integrated nanoindentation/AFM technique is extensively used to characterize the mechanical properties of the films. This growth mechanism is sufficiently general that it may be applicable to other oxide systems. Therefore, the ultimate goal of this study is to develop a process that can yield dense, solid ceramic structure with enhanced properties.

Bulk Metallic Glasses: Processing and Fabrication II

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Monday PM — Room: 3006
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Todd C. Hufnagel, Johns Hopkins University, Matls. Sci. & Engrg., Baltimore, MD 21218 USA; Robert W. Hyers, University of Massachusetts, Mechl. Engrg., Amherst, MA 01003 USA

2:00 PM
Anomalous Thermal Expansion in Undercooled Liquid Ti39.5Zr39.5Ni21: *Richard C. Bradshaw*[1]; Robert W. Hyers[1]; Jan R. Rogers[2]; Tom J. Rathz[2]; Geun W. Lee[3]; Anup K. Gangopadhyay[3]; Ken F. Kelton[3]; [1]University of Massachusetts, Mechl. Engrg. Dept., 160 Governors Rd., Elab 220, Amherst, MA 01003 USA; [2]NASA, Marshall Space Flight Ctr., MC SD46, NASA MSFC, Huntsville, AL 35812 USA; [2]University of Alabama, Ctr. for Automation & Robotics, Huntsville, AL 35899 USA; [3]Washington University, Dept. of Physics, St. Louis, MO 63130 USA

Quasicrystal alloys show great promise in the areas of low friction coatings, electronic applications and hydrogen storage. Discovered in 1984, the properties and behaviors of quasicrystals are still under study. Particular emphasis is being placed on the study of thermophysical properties in the undercooled liquid regime. The icosahedral ordering in the undercooled liquid is similar to the quasicrystal structure; the degree of ordering in the undercooled liquid should be reflected in changes in thermophysical properties. A non-linearity in thermal expansion has been observed in the undercooled liquid state in a Ti39.5Zr39.5Ni21 quasicrystal forming alloy. This non-linearity correlates with anomalous behavior in surface tension and specific heat as well as x-ray structure factor. Non-contact processing and optical measurement techniques allow access to the undercooled liquid. An overview of the processing and measurement technique is presented with the thermal expansion measurements.

2:20 PM
Short-Range Order Studies in Aero-Acoustic Levitation Processed Zr-Based Bulk Amorphous Alloy: *James J. Wall*[1]; J. K.

Richard Weber[2]; Peter K. Liaw[1]; Hahn Choo[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Containerless Research, Inc., Evanston, IL 60201 USA

Aero-acoustic levitation, was used to fabricate Zr57Ti5Ni8Cu20Al10 bulk metallic-glass at cooling rates of 2, 5, 10, 20, 100, and 300 K/s. In-situ levitation-calorimetry during processing showed the evidence of increasing the exothermic activity in the samples with decreasing cooling rate, suggesting an evolution of short-range order at the lower cooling rates. To investigate this phenomenon, neutron scattering experiments were performed on the ex-situ samples. Pair-distribution-function analyses showed that the atomic structure of the samples processed at a lower cooling rate exhibited a tendency to form more densely packed short-range atomic clusters. It is suggested that this behavior acted to lower the specific enthalpy of the specimens, enhancing the thermodynamic stability. This work is supported by the NSF International Materials Institutes Program DMR-0231320, with Dr. Carmen Huber as the program director.

2:40 PM

Exploration of Bulk Aluminum Glass: *James Michael Scott*[1]; Wynn S. Sanders[2]; Daniel B. Miracle[2]; Scott D. Bohnenstiehl[2]; Jenifer S. Warner[2]; [1]UES, Inc., 4401 Dayton-Xenia Dr., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Matl. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

Previous research has led to a new strategy for the prediction of new bulk metallic glasses. This strategy includes: application of a unique topological relationship via atomic size distribution plots, preferred radius ratios that enable efficient atomic packing, appropriate alloying element selection to depress the liquidus relative to the glass transition temperature and new structural modeling concepts. In addition, a wedge casting technique offers a simple method for alloy evaluation and gives an effective ranking of relative glass forming ability. Composition space of ternary marginal glass formers has been systematically explored via the addition of quaternary and higher order alloying elements based upon this strategy and use of this wedge casting technique. The results obtained from this systematic search will be presented and discussed.

3:00 PM

A Calphad Approach for Predicting the Bulk Glass-Forming Tendency of Zr-Ti-Ni-Cu-Al Alloys: *Hongbo Cao*[1]; Dong Ma[1]; Y. Austin Chang[1]; Ling Ding[1]; Ker-Chang Hsieh[2]; [1]University of Wisconsin, Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Sun Yat Sen University, Inst. of Matls. Sci. & Engrg., Kaohsiung Taiwan

A Calphad approach has been used to predict the compositions of Zr-Ti-Cu-Ni-Al alloys exhibiting low-lying-liquidus surfaces, which tend to favor the formation of bulk metallic glasses. The idea is to build on all thermodynamic information from the lower order constituent binaries and ternaries to obtain the thermodynamic properties of the higher order alloys. These thermodynamic properties enable us to predict those alloy compositions with low-lying-liquidus surfaces. The predicted alloy compositions of this quinary system for glass formation agree well with those determined experimentally. Using these calculations as a guide we identified a series of new alloys, which can be readily cast into glassy rods of 10 mm in diameter.

3:20 PM Break

3:40 PM

Producing Amorphous Joints in Metallic Glasses by Reactive Joining: *Jonathan C. Trenkle*[1]; Jiaping Wang[1]; Omar M. Knio[2]; Etienne Besnoin[2]; Tim P. Weihs[2]; Todd C. Hufnagel[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Reactive NanoTechnologies, 111 Lake Front Dr., Hunt Valley, MD 21030 USA

Reactive multilayer foils are comprised of nanoscale layers of materials that can sustain a self-propagating exothermic reaction. Al/Ni foils can be used to join metallic glasses with no evidence of devitrification in the glass. However, the presence of intermetallic products left by the reacted foil may degrade the mechanical properties of the joint. Because some of the characteristics of reactive foils (such as a negative heat of mixing of the components) are also characteristics of glass-forming alloys, it may be possible to design foils that react to form amorphous phases. To this end, we have investigated the heats of reaction, reaction velocities, and structure of Zr/Ni, Zr/Ni/Al, and Zr/(CuNi)/Al foils. We explore the influence of foil architecture on phase selection, and, in particular, on amorphous phase formation. We also discuss joining bulk metallic glasses using these reactive foils.

4:00 PM

Fabrication and Characterization of Ta-Based Amorphous Alloys: *Jiaping Wang*[1]; Laszlo J. Kecskes[2]; Todd C. Hufnagel[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]U. S. Army Research Laboratory, Aberdeen Proving Ground, MD 21005 USA

In most engineering applications low-density materials are desirable, but some specialized applications benefit from high-density materials. To this end, we have produced a series of Ta-Nb-(Fe,Ni) amorphous alloys with densities greater than 12 g cm-3 by mechanical alloying of the elemental powders. We have followed the progress of the amorphization by x-ray diffraction, scanning electron microscopy, and transmission electron microscopy. Thermal properties, including glass transition temperatures, crystallization temperatures, and liquidus temperatures were measured by differential scanning calorimetry and differential thermal analysis. We describe the mechanisms of amorphous alloy formation in the mechanically deformed powders, and the possibility of achieving even higher densities by making composite materials consisting of W particles in an amorphous Ta-alloy matrix.

4:20 PM

Consolidation of Fe-Based Amorphous Metal Powders by ECAE: Suveen N. Mathaudhu[1]; *K. Ted Hartwig*[1]; Ibrahim Karaman[1]; [1]Texas A&M University, Mechl. Engrg., 3123 TAMU, College Sta., TX 77843-3123 USA

Recent advances have allowed the fabrication of Fe-based bulk metallic glasses which have yield strengths two to three time that of common high strength steels. But limited structural applications have been found due to size limitations (~1 cm diameter max) inherent to the alloy systems used and the casting process. In this paper, hot equal channel angular extrusion (ECAE) in 90° tooling is used to consolidate Fe-based amorphous metal powder (Fe-Cr-Mo-Y-C-B) into a bulk amorphous metal rod with sizes equal to or greater than can be currently fabricated by casting. A fully dense and uniformly consolidated product is expected after only one extrusion. Hardness, DSC and other experimental results are reported.

4:40 PM

Nano-Hetero Structures in Zr-Base Glass Forming Alloys: *Jayanta Das*[1]; *Jürgen Eckert*[1]; Wolfgang Löser[2]; Sanat K. Roy[3]; Annett Gebert[2]; [1]Technische Universität Darmstadt, FB11 Matl. und Geowissenschaften, FG Physikalische Metallkunde, Petersenstrasse 23, Darmstadt D-64287 Germany; [2]Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden, Postfach 270016, D-01171 Dresden Germany; [3]Indian Institute of Technology, Dept. of Metallurgl. & Matls. Engrg., Kharagpur 721302 India

Bulk metallic glasses and nanostructured Zr-base multicomponent alloys are attractive candidates for advanced high strength materials for structural applications as well as for possible use as functional materials. Typically the strength of such alloys can be significantly higher than the values achievable for conventional microcrystalline materials. However, one major drawback for the use in engineering applications is the often limited macroscopic plastic deformability at room temperature, despite the fact that some of these alloys show perfectly elastic-plastic deformation behavior. This feature is correlated with localized deformation process, where high plastic deformation processes is accumulated in a very narrow region without contributing to macroscopic plasticity. To avoid such limitations, the concept of heterogeneous microstructure with partly glassy or nanostructured matrix and different length scale of second-phase dispersions has been recently employed to toughen the bulk metallic glasses or nanostructured materials derived from glass forming alloy compositions. We will present recent results obtained from Zr-base multicomponent structural in situ composite materials with unusual microstructure synthesized in a bulk form through inexpensive processing routes. This type of composite microstructure is able to achieve high strength together with high ductility by controlling the instabilities responsible for early failure.

Carbon Technology: Green Anodes

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland

Monday PM Room: 2007
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Matt Powell, Century Aluminum, Hawesville, KY 42348 USA; Zeno DeMori, Hilton Head Island, SC 29926 USA

2:00 PM
Capacity Creep Considerations for a Carbon Plant: *David William Glover*[1]; [1]Hatch Associates, 152 Wharf St., Brisbane Australia

Many smelters have been pursuing capacity creep to improve their economic position. As the amperage has been raised, the demand for raw materials such as power, alumina, and carbon has increased. Anode supply has become a constraint for many smelters and this paper is a discussion of the options, challenges, and opportunities available to smelters considering increasing their capacity.

2:25 PM
The Integration of Lean and Six Sigma - A Powerful Improvement Strategy for Carbon Plants: *Keith A. Sinclair*[1]; Rick Phelps[1]; Barry Sadler[2]; [1]Sinclair Associates, Inc., 12620 Ridgepath Ln., Knoxville, TN 37922 USA; [2]Net Carbon Consulting Pty Ltd., PO Box 286, Kangaroo Ground, Victoria 3097 Australia

Lean Manufacturing and Six Sigma have come to the forefront as models for manufacturing excellence. Six Sigma is driven from the strong relationship between reducing process/product variation and increasing business value - as represented by lead-time, cost, yield, and product quality. Lean Manufacturing is focused on the elimination of waste to reduce variation, shorten cycle times, accelerate product flow, and increase Customer "value in use." Although applied to some degree in Carbon Plants, they are not commonly used. The Authors feel there are several reasons for this: * Available information tends to focus on Lean/Six Sigma in discrete manufacturing. * There is little available experience in processes such as anode production. * Some concepts in Lean/Six Sigma have little relevance to existing Carbon Plants. These methods require careful integration to deliver full benefits - this is not always done well. Examples will be provided to demonstrate how Lean/Six Sigma can be successfully integrated into a wider framework and used to deliver sustained business value in process industries.

2:50 PM
Coke Quality Effect on the Grinding in the Air Swept Ball Mill Circuit: *Juraj Chmelar*[1]; Trygve Foosnæs[2]; Harald Arnljot Øye[2]; [1]SINTEF, Matls. & Chmst., Alfred Getz vei 2, Trondheim NO-7465 Norway; [2]NTNU, Dept. of Matls. Tech., Sem Sælands v 14, Trondheim NO-7491 Norway

Petroleum coke fines have been produced from three petrol coke qualities in an air swept ball mill with integrated air classifier under varying operating conditions. The pilot scale ball mill circuit is fully computer controlled with a laser based online particle size analyzer. The particle size is continuously monitored on a representative sample obtained from the air classifier outlet using a sample probe. The fines granulometry is specified through the computer controlling the ball mill circuit. The paper presents results obtained from the fines production with the following objectives; different product size, air sweeping velocity/particles residence time in the mill, circulating amount, mill throughput and specific energy consumption. The circulating load and air sweeping velocity/particle residence time has a critical effect on the particle size distribution steepness although the same top size product is produced. The morphology of the product was analyzed. The particle shape depends on the process control and the energy used for fines production.

3:15 PM
Fines Production for Anode Manufacturing: *Thore Moeller*[1]; [1]Claudius Peters Projects GmbH, Schanzenstrasse 40, 21614 Buxtehude Germany

For the anode production, 20-30% of the petroleum coke is used as dust in the fines fraction. Petroleum coke with an (HGI) 32-40 is ground to dust with 2000-5000 cm^2/g (Blaine) in a ball mill and are separated via the dynamic air swept classifier. During normal operation, these mills run during 60-90% of the time. When operating in shorter intervals the consistency of the dust fineness is not guaranteed. Operation with longer intervals and hence interruptions of several hours per shift, the segregation in the large silos of 30-50 ton capacity leads again to consistency problems. This presentation describes the operation of a vertical mill with integrated classifier for the anode industry, that was originally introduced in the coal and mineral industry. With this mill, dust can be produced during permanent operation which leads to consistent characteristics at minimum silo capacity.

3:40 PM Break

3:55 PM
Carbon Plant Performance with Blended Coke by Alberto Salvador: *Alberto Salvador Gomes*[1]; Reinhard Max Heilgendorff[1]; [1]ALBRAS Aluminio Brasileiro SA, Estrada Pa 483, Km 21, Vila Murucupi, Barcarena, Para 68447-000 Brazil

Quality deterioration and variability in the calcined coke market have resulted in routine changes in the property set of coke supplied to Albras. This deterioration has encouraged Albras to in investigate on-site, calcined coke blending and the consequent impact on carbon plant operation. This paper presents the results of industrial-scale testing of an on-site blend of two cokes - each produced from a single green coke.

4:20 PM
Finer Fines in Anode Formulation: *Francisco Figueiredo*[1]; [1]Consórcio de Alumínio do Maranhão, Smelter, BR 135, km 18, Electrode Dept., São Luís, MA 65095-604 Brazil

In order to provide continuous improvement and permit amperage increase in Potrooms, many efforts have been conducted to optimize anode formulation at Alumar. At the same time this new technique is an alternative to compensate calcined coke VBD (vibrated bulk density) deterioration. Carbon plant department studied the effect of finer fines in anode formulation and conducted trials to confirm observations. Green mill operation and process parameters were adjusted to increase 400 points in Blaine measurement (R&D Carbon Method). Results were measured in terms of green and baked anode properties. Baked apparent density, air permeability, electrical resistivity and flexural strength improved as expected with high statistical correlation. No cracks rate increase was observed and baked loss was the lowest in plant history.

4:45 PM
The SCAP-Rhodax® Process for Dry Mix Preparation in Anodes Plants: *Andre Pinoncely*[1]; Corinne Jouault[2]; Jean-Francois Andre[1]; Yann El Ghaoui[2]; Jean-Christophe Rotger[2]; Christian Dreyer[3]; [1]Solios Carbone, 32 rue Fleury Neuvesel, Givors 69702 France; [2]Alcan, LRF, St. Jean De Maurienne 73303 France; [3]Aluminium Dunkerque, Zip Ouest, Loon Plage 59279 France

Anode manufacturers have to ensure that their anode quality will allow a safe and optimum potline operation. This high anode quality must be obtained at the lowest price. The Solios Carbone Aluminium Pechiney - Rhodax® process, or SCAP-Rhodax®, which is dedicated to the production of a dry mix with an optimized G/S ratio, was developed following two objectives: reduce the investment and operating costs and ensure a high and stable anode quality. The final step of the Alcan and Solios Carbone research program was the construction of an industrial scale 35 tph pilot plant, which namely incorporates the use of a Rhodax® crusher, at the Aluminium Dunkerque smelter in France. This pilot plant allowed, from 2002 to 2004, the full validation of this very innovative process. Since May 2004, the SCAP-Rhodax® process ensures the whole anode production of the Aluminium Dunkerque smelter, on a continuous way. This process lead to a real breakthrough in paste plant technology and Aluminium Bahrain has already chosen it for its line 5 expansion. This new 35 tph industrial SCAP-Rhodax® reference will start up at the end of 2004. This paper recalls the process flow sheet, gives an updated synthesis of the industrial results demonstrated at the Aluminium Dunkerque prototype, and draws up the main figures of the first commercial reference coming soon on stream at Alba line 5.

Cast Shop Technology: Aluminum Melting: Strategies and Sourcing

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday PM Room: 2001
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Ctr. for Al Tech., Lexington, KY 40511 USA; Paul King, Albany Research Center, Albany, OR 97321 USA

2:00 PM
Implications of the Changing World of Aluminum Metal Supply: *Subodh K. Das*[1]; W. Jerry Long[1]; H. Wayne Hayden[2]; John A.S. Green[4]; [1]Secat , Inc., 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]MMPaCT, 123 Lake Hill Dr., Oak Ridge, TN 37830-4231 USA; [4]Consultant, 3712 Tustin Rd., Ellicott City, MD 21042-4826 USA

Geographic distribution of aluminum production has shifted, driven by energy and raw materials factors, and this transformation will likely continue in the future. A scenario for the U.S. aluminum industry is presented that illustrates the increasing impact of remelting, as compared to primary production by smelting of aluminum in the next few decades, with an emphasis on the magnitude of the opportunity for energy savings in each area. In the U.S., consumption will continue to grow while primary production will continue to shrink. Thus, the supply of metal to downstream fabrication processes will need to be increasingly met from re-melted ingot and scrap sources. Energy-efficient technologies for remelting and fabrication will play a more pivotal role in reducing energy consumption, environmental impacts, and imports. Areas of suggested research topics in the broad field of recycling, remelting and fabrication will be discussed as they apply to the changing aluminum world. Adapted from work originally appearing in August 2004 issue of *JOM*, pages 14-17.

2:25 PM
Scheduling Optimisation for Aluminium Smelter: *Neil Freeman*[1]; Jeffrey Dean Kelly[2]; [1]Honeywell, HPS, 5 Kitchener Way, Perth, Western Australia 6101 Australia; [2]Honeywell, Process Solutions, 300 Yorkland Blvd., N. York, Ontario M2J 1S1 Canada

Scheduling of cast house operations is difficult especially for multi-product casting operations. The cast house operations are generally batch oriented where as the pot line operations continuously produce hot metal which must be processed. This results in difficulties coordinating the pot line operations with the cast house operations with the net effect being sub optimal scheduling. New technologies incorporating Mixed Integer Linear Programming are able to not only solve this "closed" shop scheduling problem but also optimise this from both a logistics and quality perspective to minimise costs and maximise profit. There are considerable gains to be realised by optimally scheduling the flow of hot metal from reduction lines through the casthouse, particularly for multi-product casting operations. These gains will come from work flow improvements as well as melt loss minimisation, pot line superheat re-use, product quality enhancements and stock holding reductions. This paper discusses the problem, solution technology and benefits to be gained from scheduling optimisation. Additionally a case study will be presented where the technology has been successfully applied.

2:50 PM
Detection of Water Content in Aluminum Scraps with a Fast Neutron Source (Cf-252): *Miting Du*[2]; Qingyou Han[1]; Ray D. Peterson[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley, Oak Ridge, TN 37831-6083 USA; [2]Oak Ridge National Laboratory, Nucl. Sci. & Tech. Div., One Bethel Valley, Oak Ridge, TN 37831-6385 USA; [3]IMCO Recycling, Inc, 397 Black Hollow Rd., Rockwood, TN 37854 USA

Aluminum scrap and remelt secondary ingot (RSI) may contain water in their internal voids. The presence of water can be a serious safety issue for the aluminum remelting industries. This article describes a method that utilizes neutrons for the rapid detection of water in solid aluminum materials based on the fact that neutrons interact with hydrogen atoms in the water molecules. A fast neutron source (252Cf) and a portable "slow-down" (backscattering) neutron detector are used for moisture detection. Initial experiments carried out with aluminum turnings indicate that there is a linear relationship between the detected slow neutron counts and the hydrogen/water levels in aluminum scrap. The method has the potential of being used by the aluminum remelting industries for rapid, non-destructive detection of water in aluminum scrap or RSI.

3:15 PM
Quantifying Economic and Scrap Usage Impacts of Operational Uncertainty Within Alloy Production Planning: *Preston P. Li*[1]; Randolph E. Kirchain[1]; [1]Massachusetts Institute of Technology, Matls. Sys. Lab., Rm. E40-421, 77 Mass. Ave., Cambridge, MA 02139 USA

The intensive recovery and recycling of scrap will certainly play a central role in the long-term sustainable use of light metals. Yet, recognizing that producers are economic agents, environmental arguments alone are insufficient to promote scrap purchase and usage. Such efforts must be paralleled by economic incentives. This paper examines the potential for more efficient raw materials management through explicit consideration of operational uncertainties (e.g., uncertain demand for products) during production planning. Such uncertainties are considered within a two-stage recourse optimization framework. Both a conceptual framework and hypothetical case studies are presented, which demonstrate overall financial benefits and specific economic incentives for greater scrap use for aluminum alloy production. Case results also demonstrate that, although intuitive, alloy production planning based solely on expected outcomes leads to more costly production than planning derived from more robust analytical methods. By factoring in the penalties associated with different possible outcomes, the new scrap purchasing decisions better positioned the alloy producer to weather uncertain outcomes and promoted greater scrap reuse efficiency.

3:40 PM Break

3:50 PM
Industrial Application of Furnace Heel and Transferred Weight Measurement: *John Courtenay*[1]; Cees Castelijns[2]; Marcel Vossenberg[2]; [1]MQP Limited, 6, Hallcroft Way, Knowle, Solihull, W. Midlands B93 9EW UK; [2]Corus Aluminium NV, A. Stoccietlaan 87, 2570, Duffel Belgium

A new system for measuring the heel and transferred weight with an accuracy of +/-200kg has been developed and applied industrially at the Duffel works of Corus Aluminium N V in Belgium on a 50 tonne tilting holding furnace. The system, known as BatchPilot, was developed by BDH Tech of Chicoutimi and works on the principle of measuring changes in the hydraulic pressure in the furnace main cylinder. Since December 2003 a unit has been on industrial trial at Duffel and during June 2004 performance reached design values. The equipment has since been placed into full production usage with excellent results with respect to accuracy being achieved. The objective at Corus is to improve yield through reduction in the number of over length casts. The paper describes the technology and developments made during the trial period to improve operator friendliness and accuracy. The collection of data, its interpretation and inter-relation with furnace batching procedure are reviewed.

4:15 PM
EMIX+ Revolutionizes Circulation and Transfer of Liquid Aluminium in Furnaces: *Owen Stephen Tollerfield*[1]; [1]Solios Thermal, Ops., Midland House, Ounsdale Rd., Wombourne, Wolverhampton, W. Midlands WV5 8BY England

Solios Thermal have completed the successful production start-up of their EMIX+ system, to displace liquid aluminium in a 20-Tonne capacity Sidewell Furnace. The vertical component of the EMIX+ not only facilitates full bath circulation and clean scrap submergence, but also controlled transfer. As such, it has the potential to eliminate the tilting furnace altogether. First indications have also shown high circulation rates and rapid submergence of light-weight, de-coated scrap and silicon alloy chips. Results include increased metal recovery rates, increased alloy dissolution rates and decreased batch times. This paper confirms the effectiveness of the EMIX+ in the cast house environment. Field results are presented to reinforce and confirm all theoretical aspects. With intrinsic safety, low maintenance requirements (no moving parts) and total flexibility, the EMIX+ presents a revolutionary solution to electromagnetic stirring and transferring. Solios Thermal will continue to develop the EMIX+ for both the primary and secondary aluminium sectors.

Cast Shop Technology: Melt Treatment: Fluxing, Alloying and Grain Refinement

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Monday PM Room: 2002
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Christian Pluchon, Alcan Pechiney CRV, Unité de Recherche Fonderie, Voreppe 38340 France

2:00 PM
Mathematical Modeling of the Chlorine Fluxing of Aluminum: *Autumn Fjeld*[1]; Suchitra Edussuriya[1]; James W. Evans[1]; Aniruddha Mukhopadhyay[2]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg., MS 1760, Berkely, CA 94720 USA; [2]FLUENT

Chlorine fluxing is an essential process in the removal of impurities from aluminum, e.g. Na, Li in the primary aluminum industry and Mg in the secondary industry. Such impurities are removed by injecting a mixture of chlorine and argon gas below the surface of the molten aluminum. In some cases this is done using simple tubes ("wands" or "lances"), but more sophisticated devices have been developed where the gas is injected through a rotating impeller which simultaneously agitates the melt, while breaking up and dispersing gas bubbles throughout the liquid. Ideally, the chlorine in the bubbles reacts with the impurities to form solid or liquid chloride layers floating on the aluminum. In reality, however, much of the injected chlorine reacts to form gaseous aluminum chloride. In turn, some of this byproduct reacts with the impurities in the melt. This reaction scheme introduces inefficiency into the process as a fraction of the aluminum chloride escapes without reacting. Thus, product is lost and the aluminum chloride reacts with atmospheric moisture to produce HCl emissions. This inefficiency is dependent upon factors such as the residence time of bubbles in the melt and the surface area of the bubbles. In previous issues of Light Metals we have described a capacitance probe used to measure bubbles in liquid aluminum and its use in an A622 fluxing unit at Alcoa. In the present paper we describe a mathematical model designed to complement the experimental measurements. At the heart of the model is the computation of liquid and gas velocities using the software FLUENT®. This computational fluid dynamics package computes the velocities in the realistic 3D geometry for the turbulent two-phase flow. Bubble residence time distributions and gas fraction at various points in the melt are also computed. The former are important in that they are then coupled to a model for the reaction to predict chlorine utilization efficiency; the latter are valuable because they allow comparison with the capacitance probe measurements. Research supported by DOE under grant DE-FC07-01ID14192.

2:25 PM
The Choice of Additions to NaCl-KCl Mixture for Aluminum Refining from Alkali and Alkali-Earth Impurities: Anatoly Georgievich Zholnin[1]; *Sergey Borisovich Novichkov*[2]; Alexander Georgievich Stroganov[1]; [1]Mosoblprommontazh, der.Ratmirovo, Ul.Naberezhnaya, 4, Voskresensky region, Moscovskay oblast 140207 Russia; [2]Russkiy Aluminiy, Prokatniy Div., Verhniy Taganskiy Typic, 4, Moscow Russia

By thermodynamic calculation was shown that the fluoride additions of alkali and alkali-earth elements to the NaCl-KCl equiweight mixture lead to the formation of any quantity of NaF in the flux. The formation of NaF leads to the aluminum saturation with sodium. The more equilibrium concentration of NaF in the flux, the more is equilibrium concentration of sodium in aluminum. Therefore all fluoride additions of alkali and alkali-earth elements lead to the aluminum saturation with sodium. The design data was confirmed experimentally on the crucible fusions. In contrast to the fluoride of alkali and alkali-earth elements, the AlF3 addition dos not result to the formation of NaF in the flux. The AlF3 addition efficiently reduces the content of sodium in aluminum, but it is very expensive. At the same time, it was experimentally discovered, that the negative influence of fluoride additions of alkali and alkali-earth elements on the sodium saturation may be neutralized by MgCl2.

2:50 PM
Evaluation of Molten Metal Losses Using Flux Protecting Covers: *Alfredo Flores Valdés*[1]; *José Escobedo Bocardo*[1]; Fidel Barajas Guevara[1]; [1]CINVESTAV, Metall., Manzana 18, #100, Fraccionamiento Molinos del Rey, Ramos Arizpe, Coahuila 25900 México

This work describes a statistically designed experimental procedure for the development of molten aluminum protecting fluxes. The composition of fluxes was based on the equimolar NaCl-KCl composition, adding to complete the formulation different amounts of fluorides such as NaF, KF, LiF, and Na3AlF6. Regarding the results obtained at a laboratory scale, the best performance was registered for a formulation containing 90 wt % NaCl-KCl, plus 10 wt % Na3AlF6, as the molten metal losses reduced from around 12 wt % to around 5 wt %. Furthermore, this formulation was tested at industrial trials level in a 4000 kg molten aluminum capacity reverberatory furnace, reducing the molten metal losses from around 10 wt % to less than 6 wt %.

3:15 PM
A Methodology for Analyzing Se in Mn Electrolytic Metal and Al Materials Coming From the Casthouse: Raquel Antolín[1]; Tomás Posada[1]; *Gregorio Borge*[1]; Juan Carlos Raposo[1]; Gorka Arana[2]; Nestor Etxebarria[2]; [1]Bostlan, S.A., Techl. Dept., Poligo Industrial Trobika, s/n, Mungia 48100 Spain; [2]University of the Basque Country, Analytical Chmst., POB 644, Bilbao 48080 Spain

Production of Mn electrolytic metal can be improved by the addition of selenium derivatives. Se is then added to the production of aluminium when Mn compacts are added to the molten bath in the casthouse. Problems related to Se toxicity during aluminium processing, and specially during the recycling of aluminium drosses, have been reported in the last years at different TMS conferences. In spite of this, actual content of Se is rarely certified or even reported in the documentation supplied by Mn electrolytic metal producers, although Se concentrations are essential to classify Mn lots. This work tries to solve controversies about real content of Se in Mn additives with the development of an analytical method HG-ICP-OES (hydride generation-inductively coupled plasma-optical emission spectroscopy) which is inexpensive, easy to handle and adequate in terms of high sensibility and reproducibility for industrial metallic matrixes. A laboratory reference material (LRM) of Se in electrolytic Mn has been generated to verify the goodness of the measurements performed. The new methodology allows to control precisely and accurately the Se concentration in Mn raw material lots, and in Al baths and drosses, thus a more adequate answer to issues of Se fate in aluminium production chain can be given.

Characterization of Minerals, Metals and Materials: Characterization of Structural Engineering Materials - I

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Monday PM Room: 2010
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Masafumi Maeda, University of Tokyo, Inst. of Industl. Sci., Tokyo 153-8505 Japan; Donato Firrao, Politecnico di Torino, Dip. di Scienza dei Materiali e Ingegneria Chimica, Torino 10129 Italy

2:00 PM
The Effect of Delta-Ferrite in P92 Steel on the Formation of Laves Phase and Cavities for the Reduction of Fatigue Life: *Jung Woong Baek*[1]; Soo Woo Nam[1]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. & Engrg., 373-1, Guseong-dong, Yuseong-gu, Daejeon 305-701 S. Korea

Modern tempered martensite 9~12%Cr steels are introduced as excellent materials for power generation. Although the 9~12%Cr steels have fully martensite microstructure, they have some tendency to form the delta-ferrite during welding and other procedure. It is known that delta-ferrite has harmful effects on the high temperature mechanical properties because of its poor creep ductility and toughness. Compared with inside of grain, grain boundaries are the preferential sites for precipitation of second phase particles, however it is confirmed from analyses of microstructural observation and diffraction

pattern that Laves phase is preferentially formed inside of delta-ferrite rather than lath boundary and inside of martensite. Therefore, creep cavities are found to be formed along the Laves phase interface during creep-fatigue test. It is hypothesized that the brittle Laves phase is responsible for the reduction of the creep-fatigue life by the formation of creep damage of cavities.

2:25 PM
Analyses of Stress Corrosion Cracking in X-52 Steel Line-Pipes: *Jian Li*[1]; Mimoun Elboujdaini[1]; [1]Natural Resources Canada, CANMET-MTL, 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

Failures of X-52 steel line-pipes due to stress corrosion cracking (SCC) have been a primary concern in oil and gas industry. However, the mechanisms of crack initiation and propagation have not been well understood. In this work, SCC from a section of line-pipe was thoroughly investigated. Features of intergranular cracks (IGSCC) and transgranular cracks (TGSCC) were characterized with optical microcopy, scanning electron microscopy (SEM), focused ion beam (FIB) microscopy and transmission electron microscopy (TEM). The effect of carbide on crack propagation is emphasized.

2:50 PM
Effect of Heat Treatment on the Microstructure and Texture Evolution of Modfied 316LN Stainless Steel: *Steven T. Downey*[1]; Peter N. Kalu[1]; Ke Han[2]; [1]FAMU/FSU College of Engineering, Dept. of Mechl. Engrg., 2525 Pottsdamer St., Tallahassee, FL 32310-6046 Leon ; [2]National High Magnetic Field Laboratory, Magnet Sci. & Tech., 1800 E. Paul Dirac Dr., Tallahassee, FL 32301 USA

Chemically modified 316LN Stainless steel is currently used by the National High Magnetic Field Laboratory to jacket superconductors used in the Superconducting Outsert Conduit of the 45T Hybrid Magnet System. The principal reason for using this material is its ability to withstand the effects of the Nb3Sn reaction heat treatment. This investigation focuses on the effect of heat treatment on the microstructure and texture evolution of the modified 316LN stainless steel. Optical microscopy, X-Ray texture, ESEM and related electron microscopical tools were employed for the analysis. Although annealing at 700°C, for times ranging from 1 to 100 hours, resulted in a negligible grain growth and texture change, there was substantial increase in precipitate formation. A correlation between the heat treatment and the microstructural and texture development is presented.

3:15 PM
Effect of Cryogenic Treatment on the Mechanical Properties of Steel (4340) and Aluminum Alloy (7075): *Saeed Zhirafar*[1]; Martin Pugh[1]; [1]Concordia University, Dept. of Mechl. & Industrial Engrg., 1455 de Maisonneuve Blvd. W., Montreal, Quebec H3G 1M8 Canada

Over the last few years, interest has been shown in the effect of very low temperatures during the heat treatment cycle on the performance of the steels. “Cryogenic Tempering” the process of deep-freezing materials at Cryogenic temperatures (-196°C), has been reported to optimize the mechanical properties of some steels, normally tool steels, being treated. This paper reports on experiments carried out to determine the hardness, toughness and durability of cryogenically treated Steel (4340) and Aluminum alloy (7075) as compared to those conventionally treated.

3:40 PM Break

3:50 PM
Imaging of Deformation Microstructure in Ni-Based Superalloys: *Peter Maxwell Sarosi*[1]; Gopal Babu Viswanathan[1]; Deborah Whitis[2]; Michael Mills[1]; Raymond Unocic[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]AEGE, Matls. Sci. & Engrg., Cincinnati, OH USA

The exellent creep tensile, fatigue and crack growth properties of nickel based superalloys have made them the favored, high temperature structural material used in the turbine section of jet engines. However, higher operating temperatures needed to increase enige efficiency degrade the superalloy’s structural properties including creep. The creep deformation mechanisms in these alloys can be complex with different deformations mechanisms arising as a function of applied stress temperature and microstructure. Computer simulations of HRTEM images and bright field images of stacking faults were compared with experimental results in order to determine the nature of the stacking faults which are necessary for the development of physically based creep models.

4:15 PM
Vapor Pressure Measurement of Phosphorous in Molten Carbon Saturated Iron: *Takashi Nagai*[1]; Masao Miyake[1]; Hisao Kimura[1]; Masafumi Maeda[1]; [1]University of Tokyo, Inst. of Industrial Sci., Internatl. Rsch. Ctr. for Sustainable Matls., 4-6-1 Komaba, Meguro, Tokyo 153-8505 Japan

The hot metal pretreatment is common practice for dephosphorization. To develop more effective process, reliable thermodynamic data of phosphorous in molten carbon saturated iron is required. In this research, thermodynamic properties of phosphorous in molten iron were investigated. The vapor pressure of P2 in equilibrium with carbon-saturated molten iron containing phosphorous was measured by double Knudsen cell mass spectrometry. The measurements revealed that the activity of phosphorous in molten iron saturated with carbon obeys Henry’s law in the range of phosphorous content below 0.72 wt%.

4:40 PM
An EBSD Study of Oxidation Behavior of Iron-Base Alloy in Supercritical Water Reactor: *Lizhen Tan*[1]; Todd R. Allen[1]; [1]University of Wisconsin, Dept. of Engrg. Physics, 1500 Engrg. Dr., Madison, WI 53706 USA

The supercritical-water-cooled reactor (SCWR) system is recommended in the DOE Generation IV reactor program due to its high thermal efficiency and plant simplification. At supercritical water conditions of 500°C and 25 MPa, one of the properties of most concern for materials selection is oxidation behavior. This behavior is generally evaluated by means of weight or thickness change. In this work, a new analytical method, electron backscatter diffraction, was employed to study the oxidation behavior of alloys exposed to SCW. Microstructural evolution including phase, grain size and shape, and texture, from bulk to the interface between oxides and SCW is revealed by this technique. This methodology is important to understand oxidation mechanisms and sheds light on the study of the effect of Grain Boundary Engineering on oxidation resistance.

5:05 PM
Precipitates Characterization of Forged and Direct Quenched Ti-Nb-V HSLA Steels: *Y. Sikaddour*[1]; M. Hadji[2]; [1]Université des Sciences et de la Technologie Houari Boumedienne (USHB), Faculté de Génie Mécanique & Génie des Procédés, Dépt. des Scis. des Matériaux, BP 32 EL Alia Bab, Ezzouar 16000 Alger; [2]Université de Blida, Dépt. des Scis. des Matériaux, El Soumaa Blida 09000

The mechanical properties of HSLA steels depend mainly on the state and the forms of precipitates induced by their thermoméchanical history. The investigation reported here is a characterisation of precipitates in forged and direct quenched Ti-Nb-V HSLA steels. The objective is to develop microalloys forging steels in order to enhance mechanical properties of the hot formed steels parts while simultaneously eliminate the need for heat treatment of the steel, which can reduces energy consumption and processing time as well as the material inventories resulting from intermediate processing steps. It has been shown that the precipitates formed between 900° and 1100°C are essentially the fine TiN and Ti Carbonitrides (Ti (C,N)) which are more stable at high temperature than other microalloying carbides or nitrides and can therefore control the austenite grain size.The experiments revealed, however, the presence of sulfide (Mn,Ti) S and carbosulfide $Ti4C_2S_2$ which doesn’t contribute to the precipitation hardening in 0,25% Ti alloy and not in other % Ti alloys this, explain the hardness decrease. Many kinds of precipitates in spherical and lengthen forms are observed in grain boundary, it was essentially (Ti,C) N , Nb (C,N) and very complexes Carbonitrides; which retain austenite grain growth during thermomechanical treatment at high temperature. Titanium additions and the effect of the temperature forging are also investigated.

Characterization of Minerals, Metals and Materials: Characterization of Industrial Products

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Monday PM Room: 2012
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Louis J. Cabri, Cabri Consulting Inc., Ottawa, Ontario K1S 5P5 Canada; Markus A. Reuter, Delft University of Technology, Delft 2628 RX Netherlands

2:00 PM
Characterization of Waelz Kiln Products from the Recycling of EAF Dust: *Tzong T. Chen*[1]; John E. Dutrizac[1]; Glenn Poirier[1]; [1]Natural Resources Canada, CANMET-MMSL, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

The Waelz kiln process is currently the most efficient commercial technique for processing EAF dust to recover the contained zinc as zinc oxide. In this process, EAF dust is converted to a Fe-rich slag and a Zn-rich Waelz oxide that is subsequently processed for the recovery of zinc. Grab samples from an European zinc recycling plant were studied. The EAF dust contains ~27% Zn which occurs mainly as ZnO and ZnFe2O4. During processing, the Zn is converted to ZnO, together with minor amounts of Pb(OH)Cl, PbCl2, NaCl and KCl, in the Waelz oxide product. The Fe is converted to metallic Fe, Fe oxide and FeO(OH), together with gypsum, CaO/Ca(OH)2, Ca2SiO4, Mn-Fe oxide, various other oxides and Ca-Al-Fe-Mg-Mn silicates, in the slag.

2:25 PM
Characterization of In-Service Concrete and Construction Materials for Failure Analyses and Performance Evaluation: *Ann Marisa Hagni*[1]; [1]Construction Technology Laboratories, Inc., Microscopy Grp., 5400 Old Orchard Rd., Skokie, IL 60077 USA

Construction materials that have been placed into service are routinely analyzed to determine cause of failure, distress, deterioration, or change in physical appearance. This information is used to evaluate the existing condition, as well as the expected future performance, of the concrete or other construction material. Optical microscopy, SEM-EDS, and XRD are key tools in identifying freeze-thaw damage, corrosion of steel reinforcement, chemical attack (sulfate, acid, alkaline), alkali-aggregate reaction, and shrinkage/swelling (plastic shrinkage, thermal expansion) in concrete. Raw materials, such as aggregates, are analyzed by mineralogical techniques to determine deleterious constituents. Cement and clinker feed materials are evaluated to optimize processing and final product, and water/cement ratio and air-void contents in concrete are determined by petrography during product development and after years of service. Examples of identification and significance of construction materials, and how mineralogical techniques are uniquely utilized in problem solving, will be discussed.

2:50 PM
Characterization of Pigments Produced by Soft-Chemical Methods: Azucena Arellano[1]; Sandra Bribiesca[1]; *Moisés A. Carreón*[1]; [1]Universidad Michoacana de San Nicolás de Hidalgo, Inst. de Investigaciones Metalúrgicas, Edificio, CP 58000, Morelia, Michoacán México

In the present work both physical and chemical characterization of black pigments synthesized via co-precipitation of Fe, Mn and Co nitrates is made. The effect of synthesis pH and the effect of chemical synthesis environment was studied. The resultant phases were characterized by means of XRD, IR spectroscopy, SEM, EDS and nitrogen porosimetry. It was found that the electrokinetic behavior of the metallic species in solution as well as the chemical environment (i.e. oxidant or inert) played a critical role in the final textural, compositional and morphological properties of the resultant pigments. XRD showed the formation of hematite when the synthesis was conducted on inert environment and magnetite in oxidant environment. SEM studies allowed us to study the effect of pH on the morphology of the synthesized phases.

3:15 PM
Fracture Mode Analysis of Phosphor Bronze Dropper Clamp in Railway Catenary System: Jung-Nam Kim[1]; *Jeongguk Kim*[1]; Song-Chul Lim[2]; Kae-Myung Kang[2]; Sung-Tae Kwon[1]; [1]Korea Railroad Research Institute, Railroad Safety Rsch. & Testing Ctr., 360-1 Woulam, Uiwang, Kyunggi 437-757 S. Korea; [2]Seoul National University of Technology, Matls. Sci. & Engrg., Seoul 139-743 S. Korea

Failure mode analysis was performed for phosphor bronze dropper clamp (DC), which is used as a holder between messenger wire and contact wire in railway catenary system. Several analysis techniques were employed for two different types of samples, a fractured DC during service and as-received new DC, to investigate the fracture mode of the DC. The chemical composition analysis of the DC was conducted to evaluate the chemical quality of the DC, and mechanical tests such as tension withstand strength test and holding strength test, were performed to verify the standard values. After mechanical testing, Scanning electron microscopy (SEM) characterizations with the energy dispersive X-ray (EDX) analyses were performed to observe the fracture surfaces of two samples. Through the SEM characterization of fracture surfaces, it was observed that the in-service fractured DC sample experienced stress corrosion cracking due to corrosive environment during service, while the tension tested sample showed ductile fracture morphology with dimples.

3:40 PM Break

3:50 PM
Characterization of Mold Fluxes for Continuous Casting of Steel: *Danny Rao Pratap Singh*[1]; E. Wei[1]; Y. D. Yang[1]; C. Feng[1]; I. D. Sommerville[1]; A. McLean[1]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Toronto, Ontario M5S 3E4 Canada

During continuous casting of steel, the properties of mold fluxes strongly affect the casting performance, steel quality and environment of the casting operation. In this study, a high temperature microscopy technique was used to investigate the melting behavior of mold fluxes, and a drip test method was used to determine their melting rate. The results showed that free carbon was a dominant factor in governing the melting behavior of the fluxes, and the melting rate increased with increasing carbon reactivity and with decreasing carbon content.

4:15 PM
Research Methods of Iron Ore Sinters: Improvement in their Quality Assessment: *Marilia Sacramento de Magalhaes*[1]; Roberto Parreiras Tavares[2]; Paulo Roberto Gomes Brandao[1]; [1]Federal University of Minas Gerais - UFMG, Mining Engrg. Dept., Rua Espírito Santo, 35/721, Belo Horizonte, MG 30160-030 Brazil; [2]Federal University of Minas Gerais - UFMG, Metallurgl. & Matls. Engrg. Dept., Rua Espírito Santo, 35, Belo Horizonte, MG 30160-030 Brazil

Iron ore sinters are the main burden of blast furnaces for pig iron production. The evaluation of these agglomerates is very important due to their properties affect indirectly the productivity and costs of production of the steel industry. Besides classical methods commonly used in sintering plants: particle size evaluation, bulk chemical analyses, tumbler and/or shatter tests and reduction degradation and reducibility indexes, microscopy techniques allow to enhance the sintering process assessment. For investigation of the phases and microstructures, reflected-light and backscattered electrons images were used; for microanalytical chemical examination, energy-dispersive X-ray spectrometry; for Kikuchi patterns registers, electron backscattering diffraction. As complementary data, magnetic susceptibility measurements were applied to characterize the magnetic behavior. Hematite, magnetite, silicoferrite of calcium and aluminium and silicates are formed during the sintering. Hematite and Mg-bearing silicate are also remnants of ores and fluxes, respectively. Phase associations and their proportions are some aspects appraised by such procedures.

4:40 PM
Quantitative Characterization of Phases Present in Aluminum Drosses: *Adriana Gómez Gómez*[1]; Nelson Batista de Lima[2]; Jorge Alberto Soares Tenório[1]; [1]Escola Politécnica - Universidade de São Paulo, Metallurgl. & Matls. Engrg., Av. Prof. Mello Moraes, 2463, São Paulo, São Paulo 05508-900 Brasil; [2]Instituto de Pesquisas Energéticas e Nucleares, X-Ray Diffraction Lab., Av. Lineu Prestes, 2242, São Paulo, São Paulo Brasil

The white dross is a result of the aluminum oxidation during its primary production. White dross is mainly composed of metallic aluminum, Al2O3, with minor amounts of AlN and some oxides. This great variety of phases does complicated the quantitative characterization. This work shows a quantitative method to determine the main phases of white aluminum drosses by means of X-ray diffraction (the Rietveld Method). Actually, the Rietveld Method is maybe the most

useful method for obtaining quantitative phases information in multi-component mixtures, this method permits relatively quick quantitative phase analysis by fitting the calculated X-ray diffraction profile with the observed one. The analyses were done for the material smaller than 45 micron, to obtain the suitable material size the aluminum dross was grounded by ten minutes, several times. The grounded material was screened, and the material smaller than 45 micron was taken to quantitative XRD analysis.

5:05 PM
Rheological Characterization of Viscoelastic Composite Systems Used in Oil Industry: *Sayavur Ispandiyar Bakhtiyarov*[1]; Geilani M. Panahov[2]; Eldar M. Abbasov[2]; [1]Auburn University, Mechl. Engrg., 202 Ross Hall, Auburn, AL 36849-5341 USA; [2]Azerbaijan National Academy of Sciences, Inst. of Math. & Math., Baku Azerbaijan

Currently the oil industry requires a development and application more "smart" materials, which will resist high range of temperatures and pressures, shear conditions, and chemical environment. Application of "self-healing" viscoelastic composite materials in many technological processes of oil production has been proven to increase an efficiency and quality of the operations. Unfortunately, the rheological and transport properties of these composite materials were not studied sufficiently. In this paper we present the results of the development of optimal recipes of thixotropic viscoelastic composite material with "self-recoverable" properties. A systematic and fundamental study of rheophysical properties and hydrodynamic behavior of composite material is realized in these studies. Laboratory experiments were carried out to determine important thermophysical, rheological and mechanical properties of the test materials using both the standard commercial and in-house made equipment. The obtained results provide a valuable information for computer modeling of the viscoelastic fluid flow in pipes of complex geometry.

Computational Aspects of Mechanical Properties of Materials: Atomistic-Scale Modeling

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Monday PM Room: 3012
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Diana Farkas, Virginia Tech, Dept. of Matls. Sci., Blacksburg, VA 24060-0237 USA; Ju Li, Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

2:00 PM Invited
Minimum Energy Paths of Thermal Activation at Crack Tips: Ting Zhu[2]; *Ju Li*[1]; Sidney Yip[3]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 494 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Massachusetts Institute of Technology, Dept. of Mechl. Engrg., Cambridge, MA 02139 USA; [3]Massachusetts Institute of Technology, Dept. of Nucl. Engrg. & Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA

We study thermally activated processes of crack-tip dislocation emission (Cu), crack kink nucleation and migration (Si), and chemically enhanced slow crack growth (SiO2+H2O). The nudged elastic band and dimer methods, guided by mechanical insights, are implemented to explore the atomistic energy landscapes and determine the minimum energy paths (MEPs) and saddle-point energies. In Cu (PRL 93, 025503), we find at 75% of the strain energy release rate for spontaneous dislocation emission, the activation energy for 3D bow-out emission is 1.1 eV, significantly higher than the continuum estimates. This is likely due to (a) negligence of surface deformation energetics, and (b) the dislocation core energies in continuum models have not been calibrated against atomistic calculations. In Si, we compute the crack kink formation and migration energies for different crack front orientations on the (111) cleavage plane, to explain the experimentally observed crack propagation directional anisotropy. In SiO2, we compute the activation pathways of water attacking strained Si-O-Si bridge. The stress-dependent activation energies are obtained, and we have found three competing MEPs, that are rate-controlling at low, medium and high stresses, respectively.

2:35 PM
Surface Energy of Silicon Calculated by Molecular Dynamics Method: *Takashi Mizuguchi*[1]; Ken-ichi Ikeda[1]; Fuyuki Yoshida[1]; Hideharu Nakashima[1]; Hiroshi Abe[1]; [1]Kyushu University, Interdisciplinary Grad. Sch. of Engrg. Scis., kasugakoen 6-1, Kasuga, Fukuoka 816-8580 Japan

Silicon shows a sharp brittle-ductile transition and is a brittle material at room temperature. Because the surface energy is considered to affect the fracture behavior of a brittle material, it is important to investigate the surface energy just after fracture in order to clarify the details of fracture mechanism. This study was carried out to clarify systematically the crystal orientation dependence of surface energy just after fracture by molecular dynamics calculation. The surface energy after annealing at 1098K was also calculated and compared with that just after fracture. It was revealed that surface energy of (111) had the minimum value. This agrees with the fact that the cleavage plane of silicon is (111). It was also found that the surface energy after 1098K annealing was lower than that just after fracture. It is concluded that the decrease in surface energy is brought about by the rearrangement of surface atoms.

2:55 PM
Ab-Initio Based Classical Potential Describes Dislocation Motion and Solute Effects in BCC Molybdenum: *Richard G. Hennig*[1]; Dallas R. Trinkle[2]; Thomas J. Lenosky[1]; Chris Woodward[2]; John W. Wilkins[1]; [1]Ohio State University, Dept. of Physics, 174 W 18th Ave., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Wright Patterson AFB, OH 45433 USA

Molybdenum's high strength and high-temperature stability makes this refractory metal very attractive for use in advanced process technologies; however, the pure metal is very brittle. Solutes can soften molybdenum; though, a microscopic understanding is missing. To describe plastic deformation requires knowledge of how dislocations—the defects responsible for crystal plasticity—evolve under stress. An ab initio based classical potential extends the accuracy of density functional theory to the relevant length scales for dislocations. The classical potential of the modified embedded atom type accurately predicts defect energies, phonon dispersion, melting, dislocation structures and Peierls stresses of the bcc phase. The atomistic effect of solutes on the dislocation core and the Peierls stress is systematically determined as a function of solute size and modulus misfit. The results explain the experimental observations of low temperature softening by Re and hardening by Hf. Supported by DOE grant DOE-DE-FG02-99ER45795 and NSF grant NSF-FRG-739792.

3:15 PM
Atomistic Study of Screw Dislocation - Obstacle Interactions in BCC Mo: *Hyon-Jee Lee*[1]; Jae-Hyeok Shim[1]; Brian D. Wirth[1]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA

The effect of irradiation on materials produces large increases in strength, which are attributed to dislocation resistance from irradiation-induced point defect clusters. Large-scale molecular dynamics (MD) simulation simulations have been used to study the sequence of events controlling the interaction between screw dislocations (of 100 burgers vector length) and the commonly observed radiation obstacles such as voids or interstitial loops in body centered cubic (BCC) molybdenum. The MD simulations have been performed using a Finnis-Sinclair potential. Considering the unique non-planar core structures of BCC screw dislocation, we first report the behavior of screw dislocation motion as a function of temperatures and applied stresses. Then, we introduce defects into the system and observe their interaction behavior with screw dislocations in both dynamic and static conditions.

3:35 PM
Computer Simulation of Solute Effects on Dislocation Motion in Model Fe-Cu and Fe-C Solid Solutions: *Kanit Tapasa*[1]; David J. Bacon[1]; Yuri N. Osetsky[2]; [1]University of Liverpool, Dept. of Engrg., Brownlow Hill, Liverpool L69 3GH UK; [2]Oak Ridge National Laboratory, Computer Scis. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA

Glide of an edge dislocation in iron containing solute atoms has been investigated using atomic-scale computer simulation. Models for two different solutes types have been considered, namely substitutional copper and interstitial carbon. The influences of solute at up to 1at%Cu and 0.5at%C have been treated. The dependence of dislocation velocity on stress and temperature under dynamic conditions has been investigated and compared with similar modelling for pure iron.

In addition, the attraction and repulsion of a dislocation due to either a single solute atom near the dislocation glide plane or pairs of solutes across the glide plane have been studied in terms of the interaction energy and the resolved shear stress for glide (Peierls stress) at T = 0K. The results are discussed in relation to the dynamics of dislocation drag and predictions of dislocation-defect interactions obtained from elasticity theory.

3:55 PM Break

4:05 PM Invited
Dislocation Motion and Dislocation-Twin Interactions in Zirconium: Atomistic Modeling and Experiments: *S. G. Srinivasan*[1]; M. I. Baskes[1]; A. Misra[1]; R. J. McCabe[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

We will describe our recent atomistic calculations of dislocation motion and dislocation-twin interactions in hexagonally close packed zirconium metal (Zr). A semi-empirical modified embedded atom method potential that includes angular forces, describes the interactions between Zr atoms. The stresses to move basal, prism, and pyramidal edge and screw dislocations in single crystal Zr are computed as a function of temperature. We find that at low temperature the basal dislocations move much easier than the prism dislocations. The stress needed to move the prism dislocation decreases significantly at higher temperature. We also will present results on the nucleation of dislocations from Zr free-surfaces and from twin boundaries, and determine the stress needed to transmit dislocations through a twin boundary. We will relate our atomistic results to detailed electron microscopy of plastically deformed Zr.

4:40 PM
Interaction of 1/3<1120>(0001) and 1/3<1120>{1100} Edge Dislocations with Point Defect Clusters in α-Zirconium: *Roman E. Voskoboinikov*[1]; Yuri N. Osetsky[2]; David J. Bacon[1]; [1]University of Liverpool, Matls. Sci. & Engrg., Dept. of Engrg., Brownlow Hill, Liverpool, Merseyside L69 3GH UK; [2]Oak Ridge National Laboratory, PO Box 2008, MS-6138, Oak Ridge 37831 USA

Atomic-scale computer modeling has been carried out for two edge dislocations with the same Burgers vector 1/3<1120> but different glide planes, (0001) and <1100>, in the hcp structure to investigate their interaction with typical point defect clusters found in high-energy displacement cascades. The dislocation in the basal slip plane splits into two partials whereas the one in the prism plane remains undissociated. The family of self-interstitial atom defects consists of a triangular cluster, an irregular 3D cluster and glissile loops with two different shapes and orientations. The population of vacancy defects includes a prismatic and pyramidal vacancy clusters. During interaction full or partial absorbtion of point defect clusters, pinning of dislocation line by cluster or due to formation of immobile segment on the dislocation, push of cluster in the glide direction occur depending on the dislocation slip plane and cluster type. Stress-strain curves have been obtained for all the simulated cases.

5:00 PM
On the Mobility and Interactions of Twinning Disconnections in the HCP Metals: *Anna Serra*[1]; David J. Bacon[2]; Yuri N. Osetsky[3]; [1]University Polytechnic of Catalonia, Dept. Applied Math. III, Jordi Girona 1-3, Barcelona 08034 Spain; [2]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK; [3]Oak Ridge National Laboratory, Computer Scis. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA

Conventional simulation of the atomic structure of twin boundaries and defects such as twinning dislocations uses periodic boundary conditions along the dislocation line and fixed conditions in the other two directions, so that extensive defect motion cannot be considered. A new method has been developed to simulate a twin boundary containing a disconnection defect, i.e. a step with dislocation character, with full periodicity in the boundary plane. It may be used for investigating either the static or dynamic properties of such interfaces as the defects in them move over large distances. The method is described here and is used to investigate the motion of twinning disconnections in {10-12} and {11-22} boundaries as a function of the applied stress and temperature. In addition some processes in the boundary, such as the creation of disconnection dipoles and the creation of crystal dislocations, are investigated.

5:20 PM
Atomic-Scale Modeling of Dislocation-Stacking Fault Tetrahedra Interactions in Copper: *Yuri N. Osetsky*[1]; Roger E. Stoller[2]; David J. Bacon[3]; [1]Oak Ridge National Laboratory, Computer Scis. & Math., PO Box 2008, MS-6138, Oak Ridge, TN 37831 USA; [2]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, MS-2008, Oak Ridge, TN 37831 USA; [3]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

The present paper reports recent progress in atomic-scale dislocation dynamics achieved by exploring large-scale simulation of realistic dislocation and SFT density and SFT size for both irradiation (~2-4nm) and ageing (>10nm) conditions. We have considered gliding edge and screw dislocations with the Burgers vector ½<110> interacting with perfect SFTs of size of up to 12nm over the temperature range from 0 to 600K. A variety of different reactions have been observed, such as dislocation climb, cross-slip and formation of superjogs at the dislocation line and ledges on the SFT. These cannot be rationalized without taking into account specific atomic-scale structure of dislocations and SFTs. The atomic-scale mechanisms observed are discussed and tentative interpretation of experimental results is given.

5:40 PM
Dislocation Bypass Mechanism on Cobalt Precipitate in Copper: Atomistic Approach: *Jae-Hyeok Shim*[1]; Brian D. Wirth[1]; Hyon-Jee Lee[1]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720 USA

Large-scale molecular dynamic simulations of the interaction between dislocations (edge and screw) and precipitates in Cu-Co alloys have been performed using 3-5 million atoms. Coherent Co precipitates show different resistance to the leading and trailing Shockley partial dislocations. A temperature dependent bypass mechanism of the trailing partial dislocation is observed irrespective of the dislocation type. At low temperatures, the trailing partial bypasses with the Orowan mechanism, whereas it bypasses with the shear mechanism at high temperatures. The combination of this change in bypass mechanism and the dynamic effect due to the phonon drag explains the reported anomalous temperature dependence of the critical resolved shear stress in Cu-Co alloys. Incoherent Co precipitates show more complicated dislocation interactions and bypass. Initial interactions involve dislocation annihilation and re-nucleation at the precipitate interface accompanied by interface dislocation generation which glides into the precipitate.

Computational Thermodynamics and Phase Transformations: Materials Design and Development

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Monday PM Room: 3005
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Koenraad G.F. Janssens, Sandia National Laboratories, Albuquerque, NM 87111 USA

2:00 PM Invited
Computational Materials Design Applications of Thermodynamics and Phase Transformations: *Charles J. Kuehmana*[1]; [1]QuesTek Innovations LLC, 1820 Ridge Ave., Evanston, IL 60201 USA

Computational Thermodynamics and phase transformations are an essential tool for the quantitative design of advanced materials. Computational thermodynamics provides information of complex phase equilibria controlling processability as well as the underlying thermodynamic inputs for detailed microstructural evolution simulations. Precipitation simulations provide control of strengthening and grain-pinning dispersions during complex thermal and mechanical processing. Displacive phase transformation models allow control of process temperatures and mechanical behavior influenced by shear transformations. Examples of the use these modeling techniques in the design of bulk metallic glasses, ultrahigh-strength precipitation strengthened stainless steels and high-strength shape memory alloys will be reviewed. Such tools can also address robust design including scalability and intrinsic process variation as well as accelerated qualifi-

cation of materials to establish confidence in a materials minimum guaranteed properties with limited available experimental data.

2:30 PM Invited
Designing Elastic Properties of Titanium Alloys: *Shigeru Kuramoto*[1]; Hideaki Ikehata[1]; Naoyuki Nagasako[1]; Junghwan Hwang[1]; Tadahiko Furuta[1]; Kazuaki Nishino[1]; Takashi Saito[1]; [1]Toyota Central R&D Labs., Inc., Advd. Metal Lab., 41-1, Yokomichi, Nagakute, Aichi 480-1192 Japan

We utilized a result of ab initio calculations to design Gum Metal, a new multifunctional titanium alloy with low Young's modulus and high strength. Elastic constants were calculated in several Ti-X binary alloys, and elastic moduli in several orientations of single crystal and those of polycrystal were estimated to obtain a basic principle to determine a compositional limitation of the alloy with low modulus. It was revealed that computational results of Young's modulus were essentially consistent with experimental results of the binary alloys, and elastic moduli in a few orientations of single crystal were expected to be minimized at the specific alloy composition which is represented by valence electron number per atom (e/a) of 4.24. Experimental results of binary, ternary and quaternary titanium alloys also exhibited substantial dependence on e/a, and some evidences of the elastic anomaly were observed in microstructure after deformation.

3:00 PM Invited
Virtual Aluminum Castings: The Ford Experiment in Integrated Computational Materials: *John E. Allison*[1]; [1]Ford Motor Company, Rsch. & Advd. Engrg., MD 3182 Scientific Rsch. Lab., Dearborn, MI 48124-2053 USA

Integrated Computational Materials seeks to unify materials models across length and time scales and knowledge domains. It has the ability to unify analysis of manufacturing, design and materials into a holistic system. It offers a solution to the paradoxical industrial need to quickly develop durable components at the lowest possible cost. This talk will describe Virtual Aluminum Castings, the Ford experiment in Integrated Computational Materials. VAC is a comprehensive, integrated and experimentally verified suite of CAE tools for optimization of cast aluminum components and processes. Our vision is that castings will be designed and virtually cast, heat treated and tested for durability, all on a workstation long before components are fabricated. This talk will review recent progress in integrating models for the Al-Si-Cu alloys typically used in automobile engine structures, including models for phase equilibria and microsegregation, aging response, and the influence of microstructure on properties. The need for a unifying activity in computational materials science in the form of a "grand challenge" will also be outlined.

3:30 PM Break

3:40 PM Invited
Trace Element Effects on Precipitation in Al-Cu-Mg-(Ag, Si) Alloys: *Aiwu Zhu*[1]; Gary J. Shiflet[1]; Edgar A. Starke[1]; [1]University of Virginia, Matls. Scis., 116 Engr.'s Way, Charlottesville, VA 22904 USA

Metastable 2nd phases play major roles for determining the mechanical properties of age-hardenable aluminum alloys. The purpose of alloy design is to enhance the precipitation of the desired 2nd phase(s) among all the competing phases. Trace elements or impurities are the sources for clusters or segregates in the quenched solid solutions and hence control the nucleation of the coherent or semi-coherent precipitates. This talk describes application of thermodynamic and first principle calculation tools to identify those elements that are either helpful or harmful. Cluster variation method based on FCC tetrahedron approximation is employed to indicate the cluster formation in the solid solutions. The energy term is calculated using first principle total energy based on LDA/GGA and the overall cluster volume relaxation or the effect of atomic size difference is considered. This FP-CVM method, together with the quasi-chemical model based on CALPHAD databases, will be compared with 3D-AP and TEM experimental observations of cluster formation prior to the precipitation in Al-Cu-Mg-(Ag, Si) alloys.

4:10 PM Invited
Aspects of Quality Assurance in a Thermodynamic Mg Alloy Database: *Rainer Schmid-Fetzer*[1]; [1]University of Clausthal, Inst. of Metall., Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

The thermodynamic Mg alloy database, which is an ongoing long-term development project at the University of Clausthal, currently comprises 17 components and 277 phases. Quality assurance is a major concern for such a large number of components and interactions. This requires meticulous work and scrutiny during all steps, the initial generation, extension, maintenance, and updating of the database. The set of procedures (or protocol) used for quality assurance in our Mg database will be outlined. Examples will be given on how to detect and eliminate artifacts or errors in thermodynamic descriptions and the resulting phase diagrams. Standard tests on the thermodynamic functions or parameters will be shown that are of practical use in checking consistency and plausibility. The link to key experiments will be highlighted. It is suggested that such a protocol may be useful also for other thermodynamic databases. The typical final user, applying the database to a real multicomponent material or process, will generally not have sufficient time, resources, and experience to perform the quality check himself. It is thus helpful to quantify if the database developer performed quality assurance following a standard protocol.

4:40 PM Invited
Alloy Design of Creep-Resistance Magnesium Alloys from Stacking Fault Energy Calculated by First-Principles: *Kenji Higashi*[1]; Tokuteru Uesugi[1]; [1]Osaka Prefecture University, Dept. of Metall & Matls. Sci., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

The first-principles calculations apply the optimizing substitutional-alloying-elements can enhance the creep resistance of Mg-based alloys, which is estimated as the materials parameter expressed in terms of stacking fault energy (SFE), based on both macroscopic phenomenological studies and mesoscopic dislocation theory. As the results, it is demonstrated that both Y and Ca among the elements investigated in the present paper are the promising elements to improve a resistance of creep controlled by dislocation-climb in the Mg-based alloys. This work presented the alloying effect on the SFE by limited additional elements in Mg-based alloys because of less computational time. Now this work is very limited examples to demonstrate the importance of atomistic analysis of the materials characteristics. In the present paper, however, we challenged to shed light on the mechanism of creep deformation in atomic scale from the first principles.

5:10 PM Invited
Computational Crystal Structure Prediction with High-Throughput Ab Initio and Data Mining Methods: *Dane Morgan*[1]; Gerbrand Ceder[1]; Stefano Curtarolo[2]; [1]Massachusetts Institute of Technology, Matls. Sci., 77 Mass. Ave., Cambridge, MA USA; [2]Duke University, Mechl. Engrg. & Matls. Sci., 144 Hudson Hall, Box 90300, Durham, NC 27708 USA

Crystal structure prediction is an essential step in rational materials design. Unfortunately, there is no general tool for reliably predicting crystal structures of new alloys. Total energy ab initio approaches can be used to accurately compare energies of different candidate structures, but developing a manageable list of candidate structures for comparison is still very challenging. A powerful new tool to tackle this problem is "high-throughput" ab initio computation, which makes use of robust automated techniques to perform many thousands of calculations. High-throughput ab initio can be enhanced with data mining techniques, which can be used to accelerate structure prediction in new alloys. We have used high-throughput methods to calculate over 14,000 full ab initio structural optimizations on 80 intermetallic binary alloys, and implemented a novel data mining scheme that shows potential to dramatically reduce the time necessary for identify stable crystal structures in new alloys.

Converter and Fire Refining Practices: Operations and Modernization

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee
Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Monday PM Room: 2016
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Theo Lehner, Boliden Mineral AB, Rönnskär Smelter, Skelleftehamn S 932 81 Skelleftehamn; Alistair G. Ross, Inco Ltd, Canadian Smelting & Copper Business, Copper Cliff, ON P0M 1N0 Canada

2:00 PM
The Operation and Improvements of PS Converters at Tamano Smelter: *Souichiro Tanaka*[1]; M. Hamamoto[1]; M. Hashimoto[1]; Shintaro Udo[1]; [1]Hibi Kyodo Smelting Co., Ltd., 6-1-1 Hibi Tamano, Okayama Pref. 706-8511 Japan

The Tamano Smelter of Hibi Kyodo Smelting Co. Ltd. was commissioned in 1972. Since start up, the anode production capacity has

increased from 101,000 tpy to 290,000 tpy, as a result of the application of continuous improvements at each phase of the expansion projects. The Tamano Smelter had developed several innovations, including the original coke combustion technology for Flash Smelting Furnace operations (T-FSF), and has also been able to extend the operating life of the Peirce-Smith converters. This paper will present several improvements implemented at the Tamano Smelter, and will discuss relevant P-S converter operation.

2:30 PM
Current Practices at the Converter and Anode Furnace Operations of PASAR: *Romeo Urban Pagador*[1]; Mirardo Celeste Malazarte[1]; Tomas Wilfred Gonzales[1]; [1]PASAR Corporation, Smelter Div., Leyte Industl. Dvlp. Estate, Isabel, Leyte 6539 Philippines

After privatization in 1999, PASAR Corporation initiated a plant modernization program to optimize the performance of the smelter by increasing plant availability. This initiative required operational adjustments to the converting and anode furnace procedures, to support the higher throughput in the flash furnace, while continuing to meet government environmental regulations. The major shift in production direction was to eliminate the 30-35 day maintenance shutdown every two years, and replace it with a 15-day shutdown. This paper discusses the changes implemented in the converter and anode operation that have resulted in an increase of 10% in flash furnace throughput, an 18% increase in blister output, to170 MT per cycle, and a converter campaign tonnage increase of 10%. Since privatization, PASAR has instituted a series of 'management of change' programs that have facilitated an industrial cultural change needed to achieve the company's vision, mission and strategic goals.

3:00 PM
Technical Developments and Environmental Protection with the PS Converter at the Sumitomo Toyo Smelter and Refinery: *Harumasa Kurokawa*[1]; Masaru Takebayashi[1]; Naoki Kubo[1]; Shuuji Endou[1]; [1]Toyo Smelter & Refinery, Techl. Sect., Otu 145-1, Funaya, Saijo, Ehime 793-0005 Japan

Since the initial start-up, the Toyo Smelter & Refinery has been operating in harmony with the ambient environment, and improvements have been implemented with technologies specifically developed for further protecting the environment. Toyo Smelter & Refinery is now planning to increase the copper production capacity from 280,000 tpy to 450,000 tpy, over a period of several years. In 2003, the first step of this smelter expansion and modernization plan was implemented. The production capacity of the P-S converter was increased dramatically, as it became possible to operate under a '2 hot-2 blowing' schedule. In 2005, an additional converter will be installed, in order to allow operation under a '3 hot-2 blowing' schedule. This report will describe the technical improvements on the P-S converters, as well as describe the Toyo expansion program and discuss the technologies applied to the fugitive gas collecting system.

3:30 PM Break

3:45 PM
Converter Modernization and Operational Improvement at the Pirdop Smelter: *William A. Enrico*[1]; Ivailo Vasilev[1]; Nikolai Naidenov[1]; Evgeni Marinov[1]; Dimo Kirilov[1]; [1]Umicore Med JSC, Pirdop Smelter, 2070 Pirdop Bulgaria

Umicore acquired the Pirdop Smelter in a privatization offering by the Bulgarian government in September 1997. Since that time, an investment program has been conducted to increase capacity to 225 000 tons per year of anode copper, while improving the environmental performance to meet Bulgarian and European standards. A significant part of this investment program involved reconstruction of the three Peirce-Smith converters and the associated gas handling system. During the period June, 2000 through April, 2002, converter capacity was increased, the existing water-cooled hoods were replaced, the waste heat boiler was replaced by two evaporative coolers, the dry ESP's were rebuilt and extended, and a Converter Control System (CCS) installed. From 2002 until the present, a continuous improvement of operating and environmental performance has been achieved. This paper will describe the converter reconstruction project, the operational, maintenance, and environmental improvements achieved since then and additional projects presently under consideration.

4:15 PM
Copper Concentrate Smelting in Peirce Smith Converters at Onahama Smelter: Toshiyuki Kawai[1]; *Michio Nishiwaki*[1]; Shosaku Hayashi[1]; [1]MMC, Onahama Smelter & Refinery, Onahama Japan

Copper concentrate smelting directly in P-S converters was started at Onahama Smelter in 1983. Thereafter, the tonnage of concentrate smelted through the P-S converters increased, such that by 2002, it equaled the tonnage smelted through the reverberatory furnaces. In 2003, 56% of the total copper concentrates treated at Onahama Smelter were processed directly through the P-S converters. Technical improvements are reviewed and advantages of direct smelting in P-S converters are reported.

4:45 PM
Studies of Heat Exchange within the Working Space of an Electrical Nickel Anode Refining Furnace: *V. M. Paretsky*[1]; *Andrey V. Tarasov*[1]; *G. S. Nus*[1]; [1]State Research Institute of Nonferrous Metals, State Rsch. Ctr. of Russian Federation, 13, Acad. Korolyov St., 129515, Moscow Russia

The effect of the degree of nickel oxide reduction and the reaction gas composition on the furnace productivity has been estimated by computations and the established relationship is in good agreement with the practical data obtained in the process of electric smelting at the Severonickel smelter. An increase in the reduction degree within a range of =0.65 - 0.9 results in an increase in the furnace productivity by 19% to 37% and in a decrease in the unit electric power requirement by 16% to 27% depending on the reaction gas composition. The agreement between the computed and practical data has confirmed that the decisive factor for the process of treatment of reduced nickel oxide in an electric-arc furnace under the given conditions is the specific heat absorption, which is dependent on the nickel oxide reduction degree and the reaction gas composition.

Extractive Metallurgy: Hydrometallurgy

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Monday PM Room: 2018
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Jim Hoffmann, James Hoffmann and Associates, Houston, TX 77242 USA; Dirk Verhulst, Altair Nanomaterials Inc., Reno, NV 89502 USA

2:00 PM
Computational Modelling of Reactive Multi-Phase Flows in Porous Media: Applications to Metals Extraction and Environmental Recovery Processes: *Mark Cross*[1]; Chris R. Bennett[1]; Diane McBride[1]; T. Nick Croft[1]; James Gebhardt[2]; [1]University of Greenwich, Ctr. for Numerical Modlg. & Process Analysis, Old Royal Naval Coll., Park Row, London SE10 9LS UK; [2]PERI, 1945 S. 1100 E., Ste. 100, Salt Lake City, UT 84106 USA

There are a number of metallurgical processes where liquid and gas containing reactants flow through a porous medium interacting in such a way as to cause the extraction of metallic components. This might happen in a leach process (as in gold or copper, for example) or in some kind of environmental recovery process on spent dumps or heaps. In either class of process, there is a challenge to develop comprehensive models that enable reliable predictions of how the process will behave over the long term. In particular, there is considerable interest in predicting the base metal and other contaminant levels in the out-flowing liquid streams from the dumps or heaps, and in the case of environmental recovery, their impact upon the local ecological system in the geographical neighbourhood. The computational modelling of these families of processes present considerable challenges to the relevant research community demanding concomitant capabilities to represent: a) The liquid flow through heterogeneous porous media structures subject to variably saturated flow conditions; b) The flow of gas through the porous structure; c) The complexity and possibly dynamically changing nature of the porous media structure, and the flow conditions of either or both the gas and liquid flows; d) The transport of reactants and products of reaction in both the liquid and gas flow; e) The interaction of the liquid and gas flows with the solid porous structure with regard to a range of chemical reactions; f) The simultaneous mass and heat transfer that occurs; g) The role of microorganisms as a catalyst for reactions. Aside from the huge amount of knowledge that must be acquired to characterise each of the above features, there is a major challenge in developing a computational modelling framework that can encompass the chemical, physical, bio-

logical, engineering and environmental features that comprise chemical reactive porous media processes, whether as industrial production or environmental recovery operations. The objective of this paper is to outline these challenges and to describe how one such framework has been developed over the last few years, which can cope with arbitrarily complex geometries and, because the computational challenges are so significant, run efficiently on high performance parallel clusters.

2:25 PM
Coupling of Electrocoagulation and Alternative Energy Sources for the Removal of Arsenic Contaminants in Water: An Environment-Friendly Materials Processing: *Jewel A. Gomes*[1]; Mehmet Kesmez[1]; Michael Weir[1]; Bonnie Ardoin[1]; Praveenkumar Daida[1]; J. R. Parga[2]; David L. Cocke[1]; [1]Lamar University, Gill Chair of Chmst. & Cheml. Engrg., PO Box 10022, Beaumont, TX 77710 USA; [2]Institute Technology of Saltillo, Dept. of Metall. & Matls. Sci., V. Caranza 2400, Saltillo Coah C.P. 25000 Mexico

Arsenic contamination of well-water in different countries has caused signs and symptoms of arsenic poisoning. Although several physico-chemical treatments are presently used for the removal of arsenic compounds from drinking water, an electrochemical treatment, electrocoagulation, is a promising processing technology and currently is the most efficient (above 99% removal) and environmentally friendly technique, particularly if combined with alternative energy sources. Wind and fuel cell technologies can be considered the most promising developmental clean power sources. We will discuss new insights into the materials processing mechanisms and show the coupling of electrocoagulation with fuel cells and simulated wind energy sources. Different electrocoagulation plate materials were considered, along varied currents from the fuel cell to produce maximum arsenic removal. Coupling these technologies will remove carcinogenic arsenic from water sources, and will exhibit efficient usage of these energy sources by producing pure water for human and agricultural consumption.

2:50 PM
The Morphology Control of Cobalt Compounds and Co3O4 Powder Produced by Homogeneous Precipitation Process: Chen Song[1]; Zhang Duo Mo[2]; Wang Li Jun[1]; Luo Yuan Hui[1]; Zhang Li[1]; [1]General Research Institute for Non-ferrous Metals, Mineral Resources, Metall. & Matls., No.2 Xin jie kou wai da jie St., Beijing 100088 China; [2]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yue Lu Dist., ChangSha, Hu Nan 410083 China

The precipitation from cobalt nitrate and chloride solution in the presence of urea yield dispersed cobalt compounds with different chemical compositions and morphologies. The effects of agitation and concentration of cobalt solution and Urea on precipitation morphology were investigated experimentally. In closed systems the particles were identified as cobalt basic carbonate with needle type shape. In open system, spherical and plate-like precipitations of cobalt basic cyanate are formed. The chemical formula of cobalt compounds was confirmed. As Co(OH)0.83(CO3)0.49 Ay 0.14H2O(in tightly-capped reactor: A stands for NO3- Cl-; y[A] molecular weight<12.9) and Co(OH)0.83(NCO)XAy0.14H2O. (in open reactor A stands for: NO3-;Cl-;42x+ y[A]molecular weight<44.82). The precursor particles transform to Co3O4 on calcining. The shap of Co3O4 particle with porous character has inheritance with precipitation. The particles formation follows by the principle of nucleation-aggregation in aqueous solution. The aggregation has played an important role. During the calcine, the aggregation was destroyed partly.

3:15 PM
Precursor Synthesis of Fibrillar Nanocrystalline Nickel Powder: Wu Jian Hui[1]; *Zhang Chuan Fu*[1]; Zhan Jing[1]; Li Chang Jun[2]; Bai Meng[3]; [1]Central South University, Sch. of Metall. Sci. & Engrg., Changsha, Hunan 410083 China; [3]JiangXi Copper GuiXi Smelter HuaXin Metal Liabilities Co., Ltd, Guixi, Jiangxi 335424 China

The composition and morphology of the precursor of fibrillar nanocrystalline nickel powder prepared by oxalate precipitation are characterized by XRD, IR and SEM analysis, and the effects of temperature, nickel ion concentration, pH value, surfactant and drying process on the morphology and dispersion of the precursor are investigated in detail. The experiment results shows that fibrillar precursor is complicated nickel salt. The precursor in well dispersive and fiber can be obtained under conditions that are proposed: precipitation temperature is 60~70 C; the nickel ion concentration is 0.6~0.8 mol/L; pH value is 8.4~8.6 and addition of PVP surfactant is 0.5%wt.

3:40 PM Break

3:55 PM
The Use of Functionalized Adsorbents in the Recovery of Copper-Complexed Cyanide from Gold Leach Solutions: *Steven F. McGrath*[1]; Rainer Bauder[1]; [1]MR3 Systems, Inc., 435 Brannan St., Ste. 200, San Francisco, CA 94107 USA

The presence of copper minerals in ores that are processed by cyanide leaching for gold recovery causes numerous problems. Dissolution of copper in the leach solution parasitically consumes cyanide and oxygen, retards gold dissolution and interferes with gold recovery processes. Additionally, the presence of copper cyanide complexes in the tailings of gold mine operations is problematic because the destruction of cyanide is impeded when the molecule is complexed with metals, and it persists in the environment. Extraction of copper cyanide complexes from the leach solution and recovery of the cyanide will improve the economics of a mining operation and mitigate the environmental liability associated with having residual weak acid dissociable (WAD) cyanides in tailings. Ion exchange materials have historically been incorporated in processes designed to solve these problems. This paper presents some recent advances in applying functionalized adsorbents to assist in cyanide recovery and copper removal from cyanide leach solutions.

4:20 PM
A Novel Perspective Method for Serpentinite Treatment: *Nshan H. Zulumyan*[1]; Zaruhi H. Hovhannisyan[1]; Aghasi R. Torosyan[1]; Sona E. Ghazaryan[1]; [1]National Academy of Sciences of the Republic of Armenia, Inst. of Gen. & Inorganic Chmst., 2-tup., Argutyan St. 10, Yerevan 375051 Armenia

A novel thermal-acid method for treatment of serpentinous ultrabasic rocks has been found. It allows extract completely magnesium and iron in the form of salts, hydroxides or oxides, as well as SiO_2 in the form of aquagel. It should be noted that up to present two methods are known to prepare aquagel by sol-gel processes: a) from the soluble silicates and b) from silica compounds like to SiH_4, $SiCl_4$ or $Si(OR)_4$. The suggested method can be seen as third one. The aquagel obtained from the serpentinites has specific behavior. For example after hydrosilicagel filtration 1 mol of SiO_2 can quite stable hold more then 64 mol of water and has particles in nanosize dimensions. According to the preliminary estimations from 1 tone of rock it is possible to extract 400kg MgO, 70-80kg Fe_2O_3, 150kg SiO_2 and 300-320kg active silica fine-grained powder.

Friction Stir Welding and Processing III: High-Temperature Materials

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Monday PM — Room: Nob Hill C/D
February 14, 2005 — Location: San Francisco Marriott

Session Chair: Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM Keynote
An Investigation into the Effect of Substrate Thickness and Machine Characteristics on Friction Stir Processing of Ni Al Bronze Alloy 95800: *Leo Christodoulou*[1]; William A. Palko[2]; Christian Fuller[3]; [1]DARPA/DSO, 3701 N. Fairfax Dr., Arlington, VA 22203 USA; [2]Naval Surface Warfare Center, Carderock Div., 9500 MacArthur Blvd., W. Bethesda, MD 20817 USA; [3]Rockwell Scientific, Structl. Metals, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

The effect of substrate thickness and equipment characteristics on the mechanical and microstructural properties of friction stir processed (FSP) cast Ni Al bronze (NAB) alloy 95800 was investigated using single-pass 0.5 inch deep FSP regions in plates ranging from 0.5 to 1.5 inches thick. Two different friction stir units were compared to determine the effect of equipment characteristics. Visual observation showed that the radiated energy generated at the tool/plate interface was different for the two FSP machines, and the average plate temperature was higher for the unit exhibiting the higher amount of radi-

ated energy. Metallographic examination showed only minor differences in the macrostructure and microstructure of the FSP zone (for a given plate size) produced by either FSP unit. However, increasing plate thickness caused an increase in strength and a reduction in tensile ductility. Methods to mitigate the loss of tensile ductility were also examined.

2:30 PM Invited
Tungsten - Rhenium Tooling for Friction Stir Welding: James Downs[1]; Todd Leonhardt[1]; *Don Mitchell*[1]; [1]Rhenium Alloys Inc., 1329 Taylor St., Elyria, OH 44036 USA

Tungsten 25% rhenium was selected due to its enhanced physical properties, which are a high modulus of elasticity, good ductility, lower ductile brittle transition temperature and a high recrystallization temperature. The initial FSW tooling has gone through a evolution in processing from producing a lower density rod with poorer properties to high density high strength FSW tool, used for applications involving the welding of thick sections of various steels and/or titanium alloys. A discussion of the processing, properties and microstructures will be presented with a review of early production of tungsten 25% rhenium rods to present tungsten-rhenium alloys.

2:50 PM
Friction Stir Welding of Nitinol: *Blair London*[1]; Jennifer Fino[2]; Alan R. Pelton[2]; Christian Fuller[3]; Murray Mahoney[3]; [1]Cal Poly State University, Matls. Engrg. Dept., San Luis Obispo, CA 93407 USA; [2]Nitinol Devices & Components, 47533 Westinghouse Dr., Fremont, CA 94539 USA; [3]Rockwell Scientific Company, 1049 Camino dos Rios, Thousand Oaks, CA 91360 USA

Nitinol, a shape-memory alloy, is difficult to fusion weld to itself and to other metals, which can make processing operations expensive and can limit applications for the alloy. Void-free friction stir welds were produced in 3 mm and 6 mm thick nitinol plates using a polycrystalline cubic boron nitride tool. The weld microstructures were documented with optical and scanning electron microscopy. The austenitic and martensitic transformation temperatures of the weld regions were measured using differential scanning calorimetry. The FSP nugget showed a slightly reduced average grain diameter (33 mm) from the base metal (43 mm). The austenite finish (Af) transformation temperature was lower in the processed region (-22.5°C) compared to the base metal (15.0°C). An important characteristic of a welded nitinol structure is its ability to undergo deformation processing. Preliminary results of hot-rolling studies on friction stir processed nitinol will be presented.

3:10 PM
Friction Stir Welding of MA 957 Oxide Dispersion Strengthened Ferritic Steel: *Bharat K. Jasthi*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; Glenn J. Grant[3]; Santosh Koduri[3]; Darrell R. Herling[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Mfg. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [3]Pacific Northwest National Laboratory, Matls. Sci. Div., K2-03, 902 Battelle Blvd., Richland, WA 99352 USA

This paper describes the results of friction stir welding MA 957. This material cannot be welded by conventional means because the ferritic steel structure contains extremely fine oxide particles such as yttria that rise in the molten weld pool. In this investigation friction stir welding was used in an attempt to obviate the fusion welding difficulties. Metallographic analysis of the post friction-stir welded material was performed to determine the distribution of the oxide dispersion and the likely structural implications resulting from its disturbance during friction stir welding.

3:30 PM Break

3:50 PM Invited
FSW of Beta Titanium Alloy Sheet: *A. P. Reynolds*[1]; Elizabeth Hood[1]; [1]University of South Carolina, Dept. of Mechl. Engrg., Columbia, SC 29208 USA

Alloy Beta 21S sheet was friction stir welded over a wide range of welding parameters. Excellent weld quality and mechanical properties were observed for all conditions. The textures in all of the welds was measured by OIM. When suitably rotated, all textures correspond to standard torsion textures observed in other BCC alloys. The required rotations varied with the welding parameters. Grain misorientation distributions and weld nugget grain size were also quantified as functions of weld parameters.

4:10 PM
Interface Microstructure in Dissimilar Friction Welded Joint: *Raghavan Ayer*[1]; Russell Mueller[1]; H. Jin[1]; Shiun Ling[1]; Steven Ford[2]; [1]Corporate Research Laboratory, Exxon Rsch. & Engrg. Co., 1545 Rt. 22 E., Annandale, NJ 07920 USA; [2]ExxonMobil Upstream Research, PO Box 2189, Houston, TX 77252-2189 USA

The interface microstructure of a Fe/Ni dissimilar Friction Stir welded joint was investigated to understand the mechanism of bonding during the Friction Stir Welding (FSW) process. The microstructure of the joint was characterized in detail by transmission electron microscopy and the remnant plastic strain across the interface determined by micro x-ray line broadening. The studies revealed that although interface diffusion of the elements was limited to a 5-10 ?Ým range and the effective diffusivities were significantly enhanced by the point defects introduced by the plastic deformation during joining. The measured x-ray strain profiles were effective in delineating the various zones (e.g. heat affected zone, thermo-mechanically affected zone) during FSW. The results of the study will be presented and the implications on the FSW of high temperature materials discussed.

4:30 PM
Friction Stir Welding of Dual Phase Steel Sheets: *M. P. Miles*[1]; J. W. Pew[1]; T. W. Nelson[1]; [1]Brigham Young University, Coll. of Engrg. & Tech., 265 CTB, Provo, UT 84602 USA

Dual phase steel is a high strength sheet steel with potential for downgauging in automotive applications, compared to standard lower strength drawing quality steels. It has good formability, but its weldablity is fair. Therefore, in cases where dual phase steel sheets are joined laser welding results in high peak hardness in the weld nugget, lowering its ductility. As an alternative, friction stir welding was investigated as a possible method of joining dual phase steel sheets. Initial results indicate that the hardness in the weld nugget is less than that of laser welded sheets, which should result in better weld formability in tailored blank applications. Tension and formability testing was used to compare the performance of the laser welded sheets with friction stir welded sheets.

4:50 PM
Friction Stir Processing of Ferrous Alloys Using Induction Preheating: *Uma Ramasubramanian*[1]; Bryan Matthew Tweedy[1]; William J. Arbegast[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA

The Advanced Materials Processing Center (AMP) at the South Dakota School of Mines and Technology is evaluating forces, temperature and wear on PCBN high temperature pin tools (HTP) with and without induction preheating. The objective is to show that induction preheating reduces the stresses on the pin tool thereby extending pin tool life. Friction stir plunge testing was carried out on 6.4 mm (0.25 inch) 1018 steel plates and 12.7 mm (0.50 inch) grey cast iron plates with force and temperature data collected. A friction factor with and without induction preheating is determined which can be used to assess the degree of interaction (plasticizing) between the pin tool and the flowing metal. The microstructures of the plunges were characterized and micro hardness measurements were taken across the transverse section of the weld to distinguish the different metallurgical zones and are related to maximum temperatures observed.

5:10 PM
The Effect of Friction Stir Processing on the Microstructure and Strength of Cast Ti-6Al-4V: *Mary Juhas*[1]; Paul Pavka[1]; David Norfleet[1]; Anthony P. Reynolds[2]; Jim Williams[1]; [1]Ohio State University, Coll. of Engrg., 2070 Neil Ave., Rm. 323 Hitchcock Hall, Columbus, OH 43210 USA; [2]University of South Carolina, Mechl. Engrg., 300 Main St., Rm. A127, Columbia, SC 29208 USA

The fully lamellar as-cast structure of Ti-6Al-4V is characterized by relatively low strength but high toughness and good fatigue crack growth resistance. In most Ti alloys fatigue crack initiation resistance is enhanced by higher strength, especially in the long life regime (HCF). We have used friction stir processing (FSP) to modify the surface microstructure of cast and HIPd Ti-6Al-4V slabs. FSP creates a very fine grained structure which typically has increased strength. The hypothesis is that higher flow stresses equate with better fatigue crack initiation resistance. We will describe the FSP method and show the effect of this process on the microstructure. A new technique to determine local flow stress variations is used. This technique uses a focused ion beam to create small, cylindrical pillars and a nanoindentor apparatus to compress these pillars, yielding a load displacement curve. These results will be presented and discussed. This work sponsored by ONR.

Frontiers in Solidification Science: Morphological Evolution and Mushy Zone Phenomena II

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Monday PM Room: 2020
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Michel Rappaz, Ecole Polytechnique Federale de Lausanne, Lausanne Switzerland; Jeff J. Hoyt, Sandia National Laboratory, Albuquerque, NM 87122 USA

2:00 PM Invited
Analysis of Solidification Microstructures During Wedge-Casting: *J. H. Perepezko*[1]; K. Hildal[1]; [1]University of Wisconsin, Dept. Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

Microstructure evolution is a function of both alloy composition and solidification thermal history. With a wedge-shaped mold, castings can be subjected to a range of cooling rates differing by orders of magnitude, typically 1-1000 K/s in a single run. With proper time-temperature measurements of the quenching melt and heat transfer analysis, wedge-casting is an effective tool to explore the spectrum of alloy solidification microstructures. The applications range from a determination of the critical cooling rate for glass formation, the measurement of the carbon equivalent in cast iron or the secondary dendrite arm spacing and porosity distribution in cast aluminum alloys. A wedge-casting mold equipped with thermocouples and fiber optic sensors has been applied to the analysis of microstructure evolution and phase selection as a function of cooling rate and undercooling in a bulk glass forming Fe-Cr-B alloy.

2:35 PM Invited
Prediction of Dendrite Growth Directions: *Alain Karma*[1]; [1]Northeastern University, Dept. of Physics, Boston, MA 02139 USA

Dendrites are well-known to have preferred growth directions relative to their crystal structure. In metals with an underlying cubic symmetry, <100> directions are often, albeit not always, selected. This raises the basic question: what determines the preferred growth directions of a dendrite? The traditional answer to this question has been that these directions correspond to maxima of the interfacial energy. This answer has been widely confirmed by solvability theory and phase field simulations for the simplest case where the solid liquid gamma plot is parameterized by a single anisotropy parameter, epsilon_1, which measures the magnitude of the four fold symmetric variation of gamma in a (100) plane. In this parameterization, only <100> growth directions are possible for positive values of this parameter. Even though the interfacial energy is very weakly anisotropic in metal systems, recent molecular dynamics simulations have revealed that two anisotropy parameters turn out however to be necessary to represent the entire gamma plot, where the second parameter epsilon_2 is the coefficient of the next higher order term in a cubic harmonic expansion of the gamma plot. The above question can therefore be rephrased by asking what are the preferred dendrite growth directions in this two-dimensional parameterization (epsilon_1,epsilon_2) of the gamma plot. This talk examines the answer to this question through quantitative phase-field simulations of dendritic evolution in two and three dimensions and the extension of solvability theory to higher order gamma plots. The main conclusion is that dendrite growth directions need not always correspond to extrema of the gamma plot and hence to low index <100>, <110>, and <111> directions. Most relevant for interpreting recent experiments in Al alloys is the finding that the growth direction can vary continuously from <100> to <110> over a domain of the (epsilon_1,epsilon_2) parameter space, which encompasses observed growth directions near <320>. Existing predictions of anisotropy values from atomistic simulations for pure Al fall close to this domain, which makes the selection of experimentally observed non-<100> orientations by various alloying elements quite plausible.

3:10 PM Invited
Coarsening of Dendritic Microstructures: R. Mendoza[1]; D. Kammer[1]; K. Thornton[2]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Engrg., 2220 Campus Dr., Cook Hall, Evanston, IL 60208 USA; [2]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI USA

We investigate the evolution of dendritic microstructures during coarsening using three-dimensional reconstructions and computations. We have measured the morphology of these evolving microstructures using measurements of the interfacial shape distribution, the probability of finding a patch of interface with a given pair of principle curvatures, the spatial anisotropy of the microstructure using the normals to the interfaces, and the genus of the microstructure. Three-dimensional phase field calculations are employed to follow the evolution of the interfacial shape distribution. Using the phase field calculations we compute the average time rate of change of a given pair of principle interfacial curvatures and the flux in probability space. We show that this flux predicts qualitatively the evolution of the interface shape distribution and can be used to understand the mechanisms responsible for coarsening in these topologically complex systems. A comparison between the predictions of the calculations and experiments will be given.

3:45 PM
A Statistically Significant Analysis Technique of Microsegregation in Multicomponent Alloys: *Muthiah Ganesan*[1]; David Dye[1]; Peter D. Lee[1]; [1]Imperial College London, Dept. of Matls., Exhibition Rd., London, England SW7 2AZ UK

Instrumental errors in microsegregation measurements in multicomponent alloys complicate the treatment of randomly sampled data, leading to mis-estimation of segregation parameters such as the partitioning coefficient and segregation range. A new, physically reasonable, alloy-independent data treatment algorithm is presented that is capable of separating these errors from the underlying segregation trends. In comparison to other data treatment strategies such as the Flemings-Gungor, this new approach exploits all the information in the microprobe data set and assign each measured location a unique fraction solid. Artificial tails commonly seen in the solute distribution profiles are also minimised if not eliminated, matching the expected results from a conventional Scheil analysis of solidification partitioning. Improved estimates of the dendrite tip partitioning are obtained. This new technique is applied to examine microsegregation and heat treat ability response of four successive generations of single crystal superalloys castings.

4:05 PM Break

4:15 PM Invited
Experimental Study of the Stability of Lamellar Eutectic Growth in Bulk Samples: *Gabriel Faivre*[1]; Silvere Akamatsu[1]; Sabine Bottin-Rousseau[2]; [1]CNRS, 140 rue de Lourmel, Paris France; [2]UPMC, 140 rue de Lourmel, Paris France

We shall present real-time observations of the directional-solidification microstructures of a transparent non-faceted binary eutectic alloy (CBr_4-C_2Cl_6) in bulk (100- to 500-µm-thick) samples. The two-phase growth front is observed from the top through the liquid and a wall of the glass container with a long-distance microscope. At near-eutectic concentrations, the growth pattern is essentially lamellar. The direction of the lamella plane is arbitrary at the onset of the solidification, but a preferred direction normal to the container walls is progressively established after a long time of pulling. We study the stability of the lamellar pattern as a function of the interlamellar spacing λ at a fixed pulling velocity V - or, equivalently, as a function of V at fixed λ. The lower stability limit is due to a lamella termination instability that occurs at λ˜0.7 λ_m, where $\lambda_m \sim V^{0.5}$ is the minimum-undercooling spacing (in our case, λ_m˜14 µm for V=1 µms^{-1}). The upper stability limit occurs at l˜0.85 lm, and is due to a zigzag bifurcation. The zigzag patterns are stable up to about 1.1 λ_m. Above this limit, they undergo a lamella break-up instability that leads either to a decrease of the spacing through the creation of new lamellae, or to the formation of topological line defects (phase jumps), which resemble the “line faults” that are commonly observed in the cross-sections of bulk metallic samples. Preliminary results about the competition between lamellar and rod-like patterns that takes place at off-eutectic concentrations will also be presented.

4:50 PM Invited
Morphological Stability of Lamellar and Rod Eutectic Growth: Andrea Parisi[1]; *Mathis Plapp*[1]; [1]CNRS/Ecole Polytechnique, Lab. PMC, Ecole Polytechnique, Palaiseau 91128 France

The morphological stability of lamellar and rod eutectic coupled growth is investigated by means of three-dimensional phase-field simulations, both for a generic eutectic alloy with a symmetric phase diagram and for the transparent organic alloy carbontetrabromide-hexachloroethane. The instabilities that limit stable steady-state growth at large spacings are identified for both lamellae and rods. For lamellae,

a zig-zag instability occurs, which can saturate or lead to the breakup of the lamellae into rods or labyrinth structures, depending on the initial spacing and the volume fractions. The simulation results are in good quantitative agreement with recent experimental observations. Rods undergo a shape transition with increasing spacing from circular over oval to a dumbbell-like section, followed by splitting into smaller rods or a transition to lamellae. The implications of these findings for the lamella to rod transition and the selection of the final microstructural patterns are discussed.

5:25 PM Invited
Spatio-Temporal Microstructure Evolution in Directional Solidification Processes: *Shan Liu*[1]; Jehyun Lee[2]; Rohit Trivedi[1]; [1]Iowa State University, Ames Lab.-USDOE, 235 Wilhelm Hall, Ames, IA 50010 USA; [2]Changwon National University, Dept. Metall. & Matls. Sci., Kyungnam S. Korea

Directional solidification has been extensively used to disclose the relationship between microstructure lengthscales with processing variables. Diffusion-only thermosolutal transport has usually been assumed to develop theoretical models for microstructure evolution, however this is not necessary correct for a solidification process conducted on ground. In this paper, we will explore the versatile microstructures formed in directionally solidified off-eutectic Al-Cu alloys. In a single solidification process, primary phase, rod eutectic and lamellar eutectic can grow at different parts of the growth interface due to the convective mass transfer in the bulk melt. For this spatio-temporal microstructure formation, we develop a local model which takes into account the local composition, temperature gradient, morphology and lenthscale variation and it is found that this kind of noisy microstructure can be understood if the local growth conditions can be well specified.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Nanostructures and Nanocomposites II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Monday PM Room: 3020
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Rajiv K. Singh, University of Texas, Austin, TX USA; Steve Pearton, University of Florida, Matls. Sci. & Engrg., Gainesville, FL 32611 USA

2:00 PM Invited
Aggregation Behavior of SiO2 and Al2O3 Nanopowders in Aqueous Solution With Various Ion Concentrations to Generate Dispersion-Strengthened Ni Coatings: Gabriele Vidrich[1]; *Hans Ferkel*[1]; [1]TU Clausthal, Inst. of Matls. Sci. & Tech., Agricolastr. 6, Clausthal-Zellerfeld 38678 Germany

One approach to generate nanoparticle-strengthened metal matrix material is co-deposition of metal ions and nanoparticles from metal plating baths containing dispersed nanoparticles. The aggregation behavior of the nanoparticles in the plating bath is of importance because this has a strong influence on their homogeneous distribution in the plated metal layer. Therefore, the aggregation tendency of the nanoparticles in a nickel plating bath of different nanopowder and ion concentrations as a function of the pH value was investigated by Photon correlation spectroscopy and Zeta potential measurements. An influence of the pH value on the particle aggregation can be observed, with the strongest influence close to the isoelectric point (IEP). An increasing ion concentration shifts the IEP to higher pH values. This lead to a much less pronounced compression of the particles electrolytic double layer than expected, directly influencing the Zeta potential and the aggregation behavior. The results are discussed.

2:30 PM
Synthesis of Nanostructured WC by Solid State Combustion Process: *Hyung Il Won*[1]; Hayk H. Nersisyan[1]; Chang Whan Won[1]; [1]Chungnam National University, RASOM, Daejeon, Yusong 305-764 S. Korea

A new cost-effective process to produce nanostructured WC powder has been developed. WC nanostructured powder was produced from the WO3-NaN3-C mixture by combustion synthesis (CS) method. The formation of WC nanostructured powders and the effects of processing parameters on the microstructure, phase and size of as-synthesized nanopowders were investigated in this study. The x-ray analysis that is performed to identify the phase composition of final products suggested the formation of monophase hcp-WC powder at the combustion temperature 1050-1150°C. Beyond of these condition reaction products are multiphase and contain WC, W2C and metallic W. Diffraction lines of WC phase are width, the average crystal size which is determined from the broadening of corresponding X-ray spectral peaks by Scherrer formula corresponds to 50 nm. The morphology of WC powder obtained by TEM indicates that WC particles are soft agglomerated, spherical in shape and nanometer in size (about 20-60 nm).

2:45 PM Invited
Combustion Synthesis of Transition Metals Nanoparticles: *Hayk H. Nersisyan*[1]; Chang Whan Won[1]; [1]Chungnam National University, RASOM, Daejeon, Yuseong 305-764 S. Korea

In the present work new methodology to synthesize transition metals nanopowders (W,Mo,Ta,,Ti) under the combustion mode is developed. The preparation of metal nanoparticles was performed by reduction of MexOy in the presence of alkali metal salt as a particle size controling agent (PSCA). In the hot zone of solid flame melted salt forms protective shells around of transition metal particles formed by rapid reduction of oxide and prevents them from the agglomeration and future growing. As-synthesized Me particles have a size about 20-100 nm and specific surface area 5-15 m2/g. The salt layer also gradually reduces the pyrophoric activity of metal nanoparticles and makes them handling during the processing. Unique properties such as unusually large shrinkage, and 90-95% relative density were recorded at a temperature 800-1400°C. The technology developed was tested in a pilot technological reactor and a large-scale production possibility was discovered.

3:15 PM Break

3:30 PM
Self-Lubricant Nanocomposite Coatings Deposited by Plasma Spraying: *Xinqing Ma*[1]; Tony DeCarmine[1]; Danny T. Xiao[1]; [1]Inframat Corporation, 74 Battersin Park Rd., Farmington, CT 06032 USA

Ceramic coatings have the greatest long-term growth potential for improved wear and corrosion resistances in a variety of application cases, such as diesel engine components for ships, urban buses, locomotives, earth moving vehicles, and power generating stations, and thereby have a strong economic thrush and environmental impact. In comparison to conventional ceramic coatings, nanostructured ceramic coatings like nano-Al2O3/TiO2 have demonstrated superior performance in terms of wear, erosion and corrosion as well as mechanical properties. In this work, oxide additive acting as “soft” lubricant phase was the first introduced to a nanostructured alumina/titania matrix phase for forming a self-lubricant nanocomposite coating. The nanocomposites had been fabricated into lubricant coatings with a single layer or a functionally graded structure using a plasma spray technique. Tribological test results for the nanocomposite coatings demonstrated 4 times increase in sliding wear resistance and 3-5 times increase in abrasive wear resistance in under the tested conditions. The lowest coefficient of friction of ~0.18 was measured on the nanocomposite coating with an optimal lubricant (iron oxide or iron sulfide) content in pin-on-disk test in ethanol. Based on morphologies and wear behavior analyses, the wear mechanism was proposed for the nanocomposites. The nanocomposite coatings have exhibited the advantages of cleavability, chemical stability, low friction and high wear resistance, and will have a potential for various applications that require high lubricity at ambient and elevated temperatures.

4:00 PM Invited
Nanostructured Ceramics for Medical Applications: *Roger J. Narayan*[1]; [1]Georgia Institute of Technology, Biomatls. & Bioengrg., Sch. of Matls. Sci. & Engrg., 771 Ferst Dr. NW, Atlanta, GA 30332-0245 USA

Nanostructured ceramics may possess unique capabilities for interacting with cells, proteins, or DNA. We have developed diamondlike carbon-metal nanocomposite films, hydroxyapatite nanocomposite films, and bioglass nanocomposite films using a novel multiphase target pulsed laser deposition process. Transmission electron microscopy has shown that these films possess nanoparticle or nanolayered composite structures. These films exhibit unique mechanical, wear, corro-

sion, and biological properties. Future prospects for nanostructured ceramics in medicine are described.

4:30 PM Invited
On the Nanoscale Particles of Metals and Ferrites: *Jitendra Kumar*[1]; [1]Indian Institute of Technology, Kanpur India

Nanosize particles of noble and transition metals dispersed over aluminium oxide support have been prepared by thermal evaporation under vacuum and studied by TEM with regard to their morphology, phase(s), etc. and changes that occur in vacuum, oxygen and hydrogen at elevated temperatures. These particles are shown to exhibit phase(s) corresponding to bulk or a distorted face centred cubic in case of gold and platinum in some situations. A number of phenomena observed (namely, coarsening of particles, faceting with well defined shapes, wetting of substrate, formation of oxide/hydrides and emergence of core-and-ring or torus shape particles) have been described. Evidence has been advanced to support the Ostwald ripening mechanism for growth of particles. Some recent results on the formation of specific shapes of silver particles have also been discussed. Nanosize particles of zinc substituted manganese ferrites have been synthesized by coprecipitation method and investigated for their phase(s), surface area and magnetic properties. It is shown that the products invariably correspond to single fcc phase but with the lattice parameter decreasing with increase in zinc content. BET surface area increases with zinc substitution - indicating emergence of progressively small size. The low saturation magnetization and high Curie temperature of manganese ferrite particles in comparison to respective bulk values and changes occurring in characteristics as a consequence of partial substitution of manganese with zinc are attributed to gradual decrease of particle size, cation redistribution and/or magnetic dead layer on the surface.

5:00 PM
Distribution of Metallic (Pt, Pd) Nanoparticle Catalysts in Ceramic Substrates: *M. Shamsuzzoha*[1]; Earl T. Ada[1]; R. G. Reddy[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Bulk platinum and palladium have been known to be chemically inert, but show remarkably high activity as catalysts when finely dispersed as nanoparticles (<10 nm) in a ceramic substrate. In this respect, we have studied the structure, morphology and distribution of these Pt and Pd nanoparticles in gadolinium-doped ceria support by TEM and XPS. The analysis results are discussed in relation to their catalytic activity in autothermal reforming of iso-octane. Prior to reforming both metallic catalysts which are highly crystalline and often twinned appear to distribute randomly in the microstructure of the ceria support. The nanoparticle size ranges between 5 - 10 nm. A special feature found on these metal-support systems was the smooth interface between the Pt or Pd metal and the ceria support. After autothermal reforming, the metal catalysts show evidences of crystalline defects as their twinned structure show gradual dissipation. The smooth metal-support interface seem to be preserved in these catalysts even after the reforming process.

5:30 PM
Ion Beam Mixing for Processing of Nanostructure Materials: *Sufian Abedrabbo*[1]; Dia-Eddin Arafah[1]; N. M. Ravindra[2]; [1]University of Jordan, Dept. of Physics, Amman 11942 Jordan; [2]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

The novel technique of Ion Beam Mixing (IBM) has been utilized to process various nanostructure materials and thin films for various applications. Examples include shallow alloy formation of band gap engineered Si-Ge for solar cell applications and metallic thin films deposited on semiconductors. Characterization techniques focus on structural variations due to Argon beam irradiation of a variety of fluences by Rutherford Backscattering (RBS) and shallow defects and deep trapping level states by Thermo luminescence (TL).

Functional Thin Films for Sensors: Novel Synthesis Methods and Applications of Functional Thin Films

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: Anis Zribi, General Electric Global Research Center, Niskayuna, NY 12309 USA; Jeffrey Fortin, GE Global Research, Niskayuna, NY 12309 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Choong-Un Kim, University of Texas, Materials Science and Engineering, Arlington, TX 76019 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Gerald Schultz, GE Infrastructure, Sensing, Wilmington, MA 01887-4498 USA

Monday PM — Room: 3022
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Jeffrey Fortin, GE Global Research, Micro & Nano Struct., Niskayuna, NY 12309 USA; Seung H. Kang, Agere Systems, Device & Module R&D, Allentown, PA 18109 USA; Gerald Schultz, GE Infrastructure, Sensing, Wilmington, MA 01887-4498 USA

2:00 PM
Surface and Bulk Passivation Layer of Silicon Nitride for Solar Cell Applications: *Chuan Li*[1]; Bhushan Sopori[1]; N. M. Ravindra[2]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA; [2]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

It is well known that silicon nitride layers, particularly films deposited by plasma-enhanced chemical vapor deposition (PECVD), are not only useful antireflection (AR) coatings for Si solar cells, but also can serve other functions in Si solar cell fabrication. In this study, the surface and bulk passivation effects by PECVD hydrogenated silicon nitride (SiN: H) layers will be presented. Model calculations of the surface recombination velocities at the Si-SiN interface on wafers coated with nitrides deposited by various techniques are presented and discussed. A complete model of H "storage" during nitridation and its subsequent diffusion into the bulk of the Si is established and employed to explain the bulk passivation effect after nitridation. Optimization of the nitridation technique is also suggested.

2:25 PM Invited
Building Selectivity and Sensitivity in High Temperature Gas Sensors: *Prabir K. Dutta*[1]; [1]Ohio State University, 120 W 18th Ave., Columbus, OH 43085 USA

The change in resistance of semiconducting metal oxides upon exposure to gases is the basis for many gas sensors. Additives are often added to modify the chemical reactivity on the metal oxide surface. However, as the temperatures at which the sensing reactions are carried out increases, there is the possibility that the additive can also interact with the metal oxide and alter its electrical properties. We have considered several strategies for increasing sensitivity and selectivity. We have examined sputtered films of titania for sensing of carbon monoxide (CO) at 550°C. Film thickness was varied from 24-1000 nm by varying the sputtering time. The films were amorphous as prepared and converted to rutile upon thermal treatment. Highest sensor sensitivity, as measured by the resistance change of the film upon exposure to CO was noted for the ~240 nm film, and characterization focused on this film. A strategy will be presented for determining total NOx (NO + NO2) in gas streams at temperatures greater than 400°C. By using a Pt-loaded zeolite Y as a catalyst filter bed placed before a sensor, NOx species in the gas stream are brought to an equilibrium concentration of NO and NO2 that is determined by the background oxygen concentration and the filter temperature. The equilibrated NOx is then measured with a yttria stabilized zirconia (YSZ) sensor using a chromium oxide sensing electrode. This design provides the opportunity for miniaturization as well as removes the need for an air reference. We have also developed a novel Pt-zeolite filter-TiO2 sensor that responds selectively to hydrocarbons in the presence of CO. We propose that the response to propane is from the water that is liberated as a result of the oxidation reaction, and this hypothesis is based on the observation that the resistance of TiO2 at temperatures of 500-600°C decreases upon exposure to water vapor.

2:50 PM
Fabrication and Characterization of Small AFM Cantilevers Using Silicon Carbide Thin Films: Moon Jong Seok[1]; Jee Hae Geun[1]; Hyun Jae Seong[1]; Park Jin Ho[1]; *Moon Ok Min*[1]; Kim Seong

Hyun[3]; Choi Young Jin[3]; Park Jeong Ho[2]; Lee Nae Eung[2]; Boo Jin Hyo[1]; [1]Sungkyunkwan University, Chmst., 300, Chunchun-Dong, Jangan-Gu, Suwon 440-746 Korea; [2]Sungkyunkwan University, Matls. Engrg., 300, Chunchun-Dong, Jangan-Gu, Suwon 440-746 Korea; [3]Korea Electronics Technology Institute, 455-6, Masan-Ri, Jinui-Myun, Pyungtaek 451-865 Korea

We have designed and tested small AFM cantilever by using silicon carbide thin films. This work reports the application of high quality silicon carbide thin films grown by metal-organic chemical vapor deposition (MOCVD) method using single molecular precursors for micro-electro-mechanical systems (MEMS) applications. In this study, we have used and compared three kinds of the precursor molecules such as diethylmethylsilane(DEMS), 1,3-disilabutane(DSB) and tetramethylsilane(TMS) in this study. After these silicon carbide thin films were grown under most suitable condition, we tried to fabricate the AFM cantilevers using standard microfabrication processes, and then to operated it with conventional AFM equipment to characterize it. We studied their superior mechanical properties by comparing both Si and Si3N4 cantilevers. The cantilevers are 200-500§¬ thick, 5-10micron wide and 10-50micron long. Tips were grown on the cantilevers by electron-beam deposition method.

3:05 PM Invited
Design and Optimization of Polymeric Sensor Materials Using Combinatorial Chemistry Tools: *R. A. Potyrailo*[1]; [1]Biosciences, Combichemistry and Characterization Technologies, GE Global Rsch. Ctr., One Rsch. Cir., Niskayuna, NY 12309 USA

Abstract not available.

3:30 PM Break

3:50 PM Invited
New Sensing Materials and Devices: *Omowunmi A. Sadik*[1]; [1]SUNY at Binghamton, Dept. of Chmst., PO Box 6016, Binghamton, NY 13902 USA

The development of new electrode materials has expanded the range and classes of detectable compounds using electroanalytical methods. Conducting electroactive polymers (CEPs) have been demonstrated to have remarkable sensing applications through their ability to be reversibly oxidized or reduced by applying electrical potentials. For sensing applications, new approaches are required to synthesize CEPs that can combine the role of matrix immobilization template with signal generation. We have reported the synthesis of polyamic acid (PAAs) and the subsequent application of the polymers for sensor fabrication. This presentation will focus on the correlation of the electrochemical behavior of precursors (dianhydride and diamine compounds) with electrodeposition of PAA. The properties of the PAAs layers have been studied by cyclic voltammetry, scanning electron microscopy and FTIR. We will also discuss the development of a new class of polymer-stabilized metal nanoparticles using polyamic acid, polyoxydianilines and polyimides. Novel applications for sensing and metal removal will be discussed.

4:15 PM
Novel Electrospun Mesoporous Nanofibers for Gas Sensing Applications: *Rashmi Rao*[1]; Duraiswamy Srinivasan[1]; Anis Zribi[1]; [1]General Electric, Global Rsch. Ctr., Niskayuna, NY 12309 USA

Abstract not available.

4:40 PM
Designing Nanostructures for Sensor Applications: *Yiping Zhao*[1]; [1]University of Georgia, Physics & Astron., Athens, GA 30602 USA

Nanostructured materials have shown great potential in improving the sensitivity and reliability of chemical and biological sensors. The ability to control the geometric shape (size, separation, orientation, alignment, etc.) of nanostructures, and to integrate nanostructures from different materials becomes one of the great challenges for sensor fabrication. Here, I want to discuss a simple physical vapor deposition technique that could help to overcome this barrier. This so-called glancing angle deposition technique can fabricate well-aligned three-dimensional nanostructures through computer programming. By rotating the substrate in both polar and azimuthal directions, one can fabricate desired nanostructures, such as nano-rod arrays with different shapes, nano-spring arrays, and even multilayer nanostructures. This method offers full three-dimensional control of the nanostructure with the additional capability of self-alignment. There is almost no limitation on materials that can be fabricated into desired nanostructures. I will discuss the current status of the glancing angle deposition technology, its potential applications in sensor development, and its future challenges.

General Abstract Session: Composites and Coatings

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Monday PM Room: 2011
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Mark L. Weaver, University of Alabama, Dept. of Metallurgl. & Matls. Engrg., Tuscaloosa, AL 35487-0202 USA

2:00 PM
Reaction Products of Al/TiC Composites Fabricated by Pressureless Infiltration Technique: *Kon Bae Lee*[1]; Hoon Kwon[1]; [1]Kookmin University, Sch. of Advd. Matls. Engrg., 861-1, Jongnung-dong, Songbuk-ku, Seoul 136-702 Korea

The interfacial reaction products in a 5052 Al/TiCp composite fabricated by the pressureless infiltration method were analyzed using SEM, EDS, and TEM. Since the spontaneous infiltration of molten Al-Mg alloys into the powder bed containing TiC particles occurred at 800 C for 1 hour under a nitrogen atmosphere, it was possible to fabricate 5052 Al alloy matrix composites reinforced with TiC particles. During fabrication of composites, reaction products of various morphology and size were formed in the Al matrix as well as in the vicinity of the TiC particles by the interfacial reaction between the Al alloy and the TiC particles. From the EDS and SADP analysis, it could be identifid that Al4C3, Al18Ti2Mg3, Ti2AlC, Al3Ti and TiAl were formed as interfacial reaction products.

2:25 PM
Kinetics Process of Aluminium Alloy Infiltrated the Cenosphere Particle: *L. L. Wu*[1]; G. C. Yao[1]; Y. H. Liu[1]; T. J. Luo[1]; [1]Northeastern University, Grad. Student of Sch. of Matl. & Metall., Liaoning, Shenyang 110004 China

Scanning electron microscope analysis of cenosphere fly ash reinforcement aluminium alloy composites showed that along with the increasing of heat preservation time, the quantity of cenosphere fly ash that the aluminium alloy infiltrated increased gradually. Aluminium alloy going into the cenosphere fly ash had followed two kinds of circumstances. First, aluminium alloy by way of the eyelet on particle wall entered the inside of cenosphere fly ash, and reacted with the air that present in the grain. Reduction of pressure in the interior of the grain caused molten aluminium to permeate into the cenosphere fly ash. If the particle was pressurized, aluminium alloy reacted with the oxide on the outside wall first. Due to the volume difference between the reactant and the product, hole on the airproof wall was formed. The grain had vacuum inside and made the aluminium alloy permeate inside the particle to form uniformity composite.

2:50 PM
The Production of TiC Reinforced Aluminium Matrix Composites by Elemental Carbon Addition: *I. Kerti*[1]; [1]Yildiz Technical University, Dept. of Metall. Matl. Engrg., Istanbul Turkey

Nowadays, with the highly developing modern technology, the interest on and the usage of metal matrix composites have been constantly increasing because of their properties that are much better than conventional materials. Discontinuously reinforced aluminium-based metal matrix composites (MMCS) are canditate materials for applications in the automotive, transportation, aerocpace, construction industries, because of their low density, high strength, elastic modulus, fatigue strength, wear resistance and low thermal expansion. A new production method of TiC reinforced aluminium matrix composite is the addition of elemental carbon into Al-Ti liquid phase. In this method, graphite and amorf carbon ore used as elemental carbon. The dissolved or the solid state carbon in Al-Ti alloys, react with the dissolved Ti and produce a thin reinforced phase of TiC in the molten state matrix alloy. The adventages of this method over the other are practicaly easy production, requiring no advanced technology, low cast of production, no wetting problems encountered in conventional composite materials production techniques, no impurity problems between matrix-reinforced phase interface which effects mechanical prop-

erties negatiely and shorter production time which is the most importent one of all.

3:15 PM
Influence of Deposition Parameters on the Microstructures and Properties of Ti-Cr-Al-N Based Overlay Coatings: *G. Mark Calhoun*[1]; Feng Huang[1]; Mark L. Weaver[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 35487-0202 USA

Recent research efforts have established that Ti-Cr-Al-N based alloys offer the potential for extending the operating temperatures of structural titanium aluminide intermetallics such as Ti-48Al-2Cr-2Nb in excess of 800°C. The present study addresses the influences of deposition parameters the microstructures, mechanical properties, and oxidation resistance of Ti-Cr-Al-N overlay coatings deposited via direct current (DC) magnetron sputtering. Coatings deposited without substrate heating were found to be amorphous when nitrogen flow ratios were maintained below 8% whereas crystalline coatings consisting of Ti and Cr-based nitrides were observed at higher nitrogen flow ratios.

3:40 PM Break

4:00 PM
Low Percolation Threshold Composites Consisting of PMMA and Carbon Black: Runqing Ou[1]; Sidhartha Gupta[1]; Charles Aaron Parker[1]; *Rosario A. Gerhardt*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

A conductive filler is normally added to an insulating polymer to render conductivity to the composite in applications like electromagnetic interference shielding and electrostatic dissipation. Since the filler is usually the more expensive component, it is advantageous to lower the percolation threshold. In this study, a low percolation threshold of 0.4 % vol. was achieved in PMMA/carbon black nano-composites. These composites were fabricated using a procedure involving mechanical mixing and compression molding. The average size of the CB nano-particles used was 21 nm with a DBPA of 175 ml/100g. SEM images of the composites reveal a pseudo-crystalline structure, where PMMA constitutes the pseudo-crystal grains and carbon black forms an interconnected network at the grain boundaries. The electrical properties of these composites as well as the composites made by solution mixing were determined by ac impedance measurements. It was found that the new method lowers the percolation threshold significantly.

4:25 PM
Investigation of the Effects of Fatty Acids on the Compressive Strength of the Concrete and the Grindability of the Cement: Ali Tugrul Albayrak[1]; *Muzaffer Yasar*[1]; M. Ali Gurkaynak[1]; Ismet Gurgey[1]; [1]University of Istanbul, Dept. of Chem. Engrg., Avcilar, Istanbul 34850 Turkey

In cement industry, a great energy consumption has been observed during grinding of clinker. Nowadays, to reduce this consumption, some waste products, which don't have economic utility, have been used as grinding aids. In this investigation, we have examined the effects of sunflower oil (SO), oleic acid (OA), stearic acid (SA), myristic acid (MA) and lauric acid (LA) on the fineness and strength of the cement have been examined. These aids were added into clinker in certain ratios based on the cement clinker weight and the grinding has been done for a definite time at the same condition. The results obtained were as follows: All of the fatty acids used increased the fineness as compared with the cement without the grinding additives. SO and OA decreased the strength significantly, LA decreased it to a lesser extent and SA increased it definitely according to the common cement. But MA didn't alter the strength of the cement as much as SA. Also the covering of the balls influences the grinding of cement clinker unfavourably.

4:50 PM
Electromechanical Response of Piezoelectric Composite Materials: *Ronit Kar Gupta*[1]; T. A. Venkatesh[1]; [1]Tulane University, Mechl. Engrg., 400 Lindy Boggs Ctr., New Orleans, LA 70118 USA

Recognizing the potential for the use of piezoelectric materials in a number of applications as sensors and actuators, there has been a continuing research and development effort to synthesize monolithic materials with enhanced coupled properties. Because the sensing or actuating actions of monolithic piezoelectric materials are limited, the composite approach to piezoelectric materials provides a unique opportunity to access a new design space with optimal mechanical and coupled characteristics, hitherto inaccessible through monolithic materials. Through finite-element based numerical modeling, a systematic methodology for predicting a complete set of coupled properties of the piezoelectric composites as a function of the poling characteristics, size, shape, and distribution of the constituent phases are obtained. Furthermore, a general method towards characterizing the coupled constitutive response of composite materials is also identified. Model predictions are compared with experimental results for a number of polymer-based and ceramic-based piezoelectric composites.

5:15 PM
Influence of an Organic Interlayer on the Tribomechanical Behavior of TiN Thin Films: *Patrick Jonathon Henry*[1]; Shelby Shuler[2]; Mark L. Weaver[1]; Shane Street[2]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]University of Alabama, Dept. of Chmst., Tuscaloosa, AL 35487 USA

Previously we have shown that the incorporation of dendrimer self-assembled monolayers (SAMs) can increase adhesion between metallic films to silicon-based substrates. This study centers on the influence of a dendrimer SAMs on the tribological response of TiN-based films. TiN films have been deposited via reactive DC magnetron sputtering onto SAM containing and SAM-free glass substrates using varying sputtering conditions (i.e., power, working gas pressure, and N/Ar mixture). The resulting microstructures and tribological properties are reported and discussed relative to prior observations for metallic thin films.

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Monday PM
February 14, 2005
Room: 3008
Location: Moscone West Convention Center

Session Chairs: Uwe Köster, University of Dortmund, Dept. of Biocheml. & Cheml. Engrg., Dortmund 44221 Germany; Tsutomu Ishimasa, Hokkaido University, Div. of Applied Physics, Sapporo 060-8628 Japan

2:00 PM Invited
Influence of Local Structure and Alloy Composition on Glass Formation and Stability: *Kenneth Franklin Kelton*[1]; [1]Washington University, Dept. of Physics, Campus Box 1105, One Brookings Dr., St. Louis, MO 63130 USA

The icosahedral quasicrysal (i-phase) is the primary crystallizing phase in a growing number of glasses. In many cases, a nanoscale microstructure is observed, likely reflecting a low nucleation barrier due to the presumed icosahedral short-range order in undercooled metallic liquids and glasses. Our recent x-ray diffraction data from electrostatically levitated metals and alloys, which support this conclusion, are presented and discussed. Since many glasses do not form quasicrystals, however, but frequently develop a similar devitrified nanostructure, other crystallization processes must be in competition. In many cases, the compositions of the amorphous and devitrified phase are different, making diffusion effects also important. The consequences of the structures of the glass and liquid and of diffusion-limited nucleation on glass formation and stability are discussed. Supported by NASA under contract NAG 8-1682, and by the NSF under grant DMR 03-07410.

2:30 PM Invited
Electronic Structure Based Approaches to Metallic Glass Stability: *Donald M. Nicholson*[1]; [1]Oak Ridge National Laboratory, CSMD, One Bethel Valley Rd., MS 6164, Bldg. 5700, Oak Ridge, TN 37830 USA

At least three factors make the determination of the electronic structure of metallic glasses difficult. First, the structure is not known from experiment; in the best circumstances only the density and pair correlations are known. Second, there is no periodicity and hence no simplifying Bloch theorem. Third, the amount of computer time required to model the electronic structure as the liquid is cooled through the glass transition is unattainable. Here, efforts to calculate the electronic structure over the past thirty years will be discussed; these include nearly free electron, effective medium, and supper cell approaches. The importance of the calculated stability of competing

crystalline phases will also be emphasized. Finally, progress in using electronic structure based forces and energies to construct classical force fields will be demonstrated by determination of the glass transition temperature and evaluation electronic, magnetic, structural, and kinetic properties of the resulting glass.

3:00 PM Break

3:20 PM Invited
Characteristic Local Structures of Bulk Metallic Glasses: *Eiichiro Matsubara*[1]; [1]Tohoku University, IMR, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

Bulk metallic glasses (BMGs) show a clear glass transition and a large temperature span of supercooled liquid region more than 50K. In order to distinguish these new amorphous alloys from the conventional ones, they are often called bulk amorphous alloys or BMGs. Inoue introduced three guiding principles for formation of BMG, i.e. the multicomponent systems, the atomic size difference between the constituents, and the large negative heat of mixings between them. We have studied atomic structures of various BMGs, such as Zr-, Pd- and Fe-based systems by x-ray diffraction techniques. We discussed characteristics of local atomic structures in contrast with those for the conventional amorphous alloys in order to understand the origin of their structural stability. In the presentation, I will summarize these structural studies of BMGs, including the recent results on the structures of BMGs in the medium-range region by our collaborative research with the groups by electron and neutron diffractions.

3:50 PM Invited
Formability of Bulk Metallic Glasses: *Takeshi Egami*[1]; [1]University of Tennessee/Oak Ridge National Laboratory, MSE/Physics, 208 S. College, 1413 Circle Dr., Knoxville, TN 37996 USA

The glass formability depends critically upon alloy composition, and it has been known that the atomic size plays a major role. Earlier we developed a theory of glass formability based upon the atomic size ratio and the atomic level stresses such atomic size mismatch creates, which explained very well the composition limit for glass formation for binary glasses. However, recent development of bulk metallic glasses challenged this theory, which did not address the question of ease of glass formation that differentiates bulk metallic glasses from regular metallic glasses. We have extended the theory and explained the difference in the ease of glass formation, based upon the concept of distributed local glass transition and the complexity of the competing crystalline phase. It should be emphasized that this theory focuses on kinetics, not energetics, in contrast to the Hume-Rothery rules. However, there is a subtle connection, which we will discuss.

4:20 PM Invited
An Atomic Structural Model for Metallic Glasses: *Daniel B. Miracle*[1]; [1]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

An atomic structural model for metallic glasses has very recently been established. Using relative atomic sizes as the primary variable, this structural model is derived from the requirement to efficiently fill space in a system of unequal spheres. Three separate comparisons with experimental data have been conducted to validate this model. The first is comparison with partial radial distribution functions, the second is with atomic coordination numbers, and the third is a prediction of metallic glass constitution. The agreement between experiment and predictions is good in all three cases, including a compelling ability to explain the compositions in a wide range of simple and complex glasses based on Zr, Pd, rare earth metals, Al, Mg and Fe. The basic features of this model will be described and the specific atomic arrangements that provide the ability to stabilize metallic glasses will be discussed. Insights gained from this model will be explored.

4:50 PM Invited
Structural Origin of the Stability of Pd-Ni-P Model Bulk Metallic Glasses Against Crystallization: *Faisal M. Alamgir*[1]; Himanshu Jain[2]; David B. Williams[2]; [1]Brookhaven National Laboratory & Hunter College, Physics Dept., 695 Park Ave., New York, NY 10021 USA; [2]Lehigh University, Dept. Matls. Sci. & Engrg., 5 E Packer Ave., Bethlehem, PA 18015 USA

The fundamental origins of the stability of the $(Pd\text{-}Ni)_{80}P_{20}$ bulk metallic glasses (BMGs), a prototype for a whole class of BMGs, were explored. While many properties of these BMGs have been characterized, their stability against crystallization has not been understood in terms of the basic structure. Therefore, we have investigated the crystallization behavior in the $(Pd\text{-}Ni)_{80}P_{20}$ glass system from the atomic and electronic structural perspective, using X-ray photoelectron spectroscopy and X-ray absorption spectroscopy, respectively. We find that the $Pd_{60}Ni_{20}P_{20}$ glass, which loosely marks the Pd-rich end of BMG formation, is stabilized through a low enthalpy component to the Gibbs free energy. The $Pd_{30}Ni_{50}P_{20}$ glass, which is near the Ni-rich end of BMG formation, is stabilized due to a large entropic cost for crystallization. The $Pd_{40}Ni_{40}P_{20}$ glass, the best glass former in the compositional range, receives stability from the kinetic hindrances imposed by having to create both a Ni-rich as well as a Pd-rich phase close to the compositions of Ni_3P and Pd_3P.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Intermetallic Growth in Lead-Free Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Monday PM — Room: 3014
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Sung K. Kang, IBM T.J. Watson Research Center, Microelect. Pkgg. Tech., Yorktown Hgts., NY 10598 USA; Darrel R. Frear, Freescale Semiconductor, Tempe, AZ 85284 USA

2:00 PM
A Reliability Issue for Pb-Free Solder Joint Miniaturisation: *Zhiheng Huang*[1]; Paul Conway[1]; Changqing Liu[1]; *Rachel Thomson*[2]; [1]Loughborough University, Wolfson Sch. of Mechl. & Mfg. Engrg., Loughborough, Leicestershire LE11 3TU UK; [2]Loughborough University, Inst. of Polymer Tech. & Matls. Engrg., Loughborough, Leicestershire LE11 3TU UK

As solder joints become increasingly miniaturised to meet the severe demands for future electronic packaging, it is vitally important to answer the question as to whether the solder joint size and geometry could become a reliability issue and therefore affect the implementation of the Pb-free solders. In this study, different sizes of copper bond pads are formed by depositing a copper seed layer onto silicon wafer, followed by definition of the pads through a photolithographic process with a spin-coated photoresist and subsequent electroplating to build-up the copper thickness. Different sizes of Sn-3.5(wt.%)Ag solder bumps and microstructure formed by solder dipping for different times are presented and compared. A two-dimensional thermodynamic-kinetic model is developed to assist the understanding of the kinetics of the interdiffusion and the formation of interfacial intermetallic compounds. The experimental results and theoretical predictions suggest that the solder bump size and geometry can influence the as-soldered microstructure, and therefore this factor should be taken into consideration for the design of future reliable untrafine Pb-free solder joints.

2:30 PM
Effect of Solder Composition on Morphology and Size Distribution of Cu_6Sn_5 Intermetallic Compound Grains in Reactions Between SnPb Solder and Cu: *Jong-ook Suh*[1]; Andriy M. Gusak[2]; King-Ning Tu[1]; [1]University of California, Matls. Sci. & Engrg., 6532 Boelter Hall, 405 Hilgard Ave., Box 951595, Los Angeles, CA 90095-1595 USA; [2]Cherkasy State University, Theoretical Physics, Cherkasy Ukraine

During the soldering reaction between molten SnPb and Cu, scallop-type Cu_6Sn_5 intermetallic compound grains form at the interface. By varying the solder composition from the eutectic point, we observe a change of the morphology of scallops from rounded grains to facetted grains. When molten pure Sn reacts with Cu, the Cu_6Sn_5 grains are highly facetted. Ripening of the interfacial intermetallic compound was also investigated, and size distribution and average size change were measured as a function of time. These experimental data were

compared with the kinetic model proposed in our previous study. (Phys. Rev. B66, 115403, 2002). The result showed that the average radius of intermetallic compound scallops follows $t^{1/3}$ dependence. Size distribution was also in good agreement with the proposed kinetic model.

2:50 PM
Effect of Cu Additives on Sn Whisker Formation of Sn(Cu) Finishes: *Hui-Ju Kao*[1]; Wen-Cheng Wu[2]; S. T. Tsai[2]; Cheng-Yi Liu[1]; [1]National Central University, Cheml. & Matls. Engrg., No. 300, Jungda Rd., Jhong-li, Taoyuan 320 Taiwan; [2]Yageo Corporation Nantze Branch, 16, W. 3rd St., Kaohsiung 811 Taiwan

Due to the Pb-free solder implementation, pure Sn and Sn(Cu) finishes on lead-frame and metal terminals of passive devices are commonly used. During a period of storage at room temperature, Sn whiskers were found to form on the surface of Sn(Cu) finishes, which causes serious reliability issues. In this study, we have investigated the effect of Cu additives on the Sn whisker formation of Sn(Cu) finishes. Four different Sn(Cu) finishes were studied, which were pure Sn, Sn0.7Cu, Sn1.0Cu, and Sn3.0Cu. According to the preliminary results, we found that Sn whisker formation was retarded by increasing Cu content in Sn(Cu) metal finishes. Also, we found that the density of Sn whisker formation deceased with increasing of thickness of Sn(Cu) finish layer. In this talk, we will report the detail mechanism of alloying and thickness effects on the Sn whisker formation. Also, how does the interfacial reaction between Sn(Cu) finishes and Cu substrates affect Sn whisker formation will be discussed as well.

3:10 PM
Nanoindentation of Intermetallics Formed in Pb-Free Solder Joints: Experiments and Simulation: *Xin Deng*[1]; Mark Koopman[2]; Nik Chawla[1]; Krishan K. Chawla[2]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA; [2]University of Alabama, Dept. of Matls. Sci. & Engrg., Birmingham, AL 35209 USA

A knowledge of the elastic properties of Cu and Ag-based intermetallics, formed during reflow of Sn-rich solder joints on Cu, is extremely important in understanding and predicting the mechanical behavior of the joint. Bulk testing of these intermetallics is problematic because of the difficulty in achieving fully-dense materials, and because the microstructure in bulk form is often quite different from that observed in the joint. In this study, we have used nanoindentation to probe the mechanical properties of intermetallics in the joint in situ. The Continuous Stiffness Method (CSM) was used during indentation to obtain the instantaneous Young's modulus as a function of depth. The Young's moduli of Cu6Sn5, Cu3Sn, Ag3Sn, Sn-Ag solder, pure Sn, and Cu, were measured by nanoindentation. After indentation, the surface characteristics of each phase were examined using atomic force microscopy (AFM). Significant dislocation pile-up was observed in Sn, Sn-Ag solder, and Cu. The texture of intermetallics, which has a significant effect on the elastic/plastic properties, was evaluated by Orientation Imaging Mapping (OIM). Finite element analysis was conducted to investigate and predict the deformation behavior during indentation and correlated very well with the experimental results.

3:30 PM Break

3:40 PM
Microstructure, Wettability and Mechanical Properties of SnZn Solder with Minor Ag Alloying Addition: *Zhidong Xia*[1]; Yongping Lei[1]; Yaowu Shi[1]; Fu Guo[1]; Ran Lu[1]; [1]Beijing University of Technology, The Key Lab. of Advd. Functional Matls., Ministry of Educ. P.R.C, Coll. of Matls. Sci. & Engrg., 100 Ping Le Yuan, Chaoyang Dist., Beijing 100022 China

In view of the cost and availability of raw materials of lead-free solder, tin-zinc solder is still very attractive to microelectronics application. In many electrical and electronic devices, pads on printed circuit board and leads of components are plated with some alloying materials before soldering. In this study, Sn-8Zn, Sn-9Zn, Sn-10Zn alloys with 0.25%Ag additive were investigated considering the wettability of these three solders on electroless tin, silver, nickel plates and pure copper substrate, respectively. Microstructure of the solders obtained during the wetting process was analyzed by optical microscopy and SEM with EDX. Tensile test was conducted to assess the strength and ductility of the solder. SnZn alloy with 0.25%Ag addition showed good wettability on electroless Ag-plated substrate. The effect of zinc variation on the tensile property of the solders was small. The microstructure of Sn-8Zn-0.25Ag and Sn-10Zn-0.25Ag solders was characterized with special shaped intermetallic compound phase, while the microstructure of Sn-9Zn-0.25Ag was basically the SnZn eutectic phase.

4:00 PM
Three-Dimensional Analysis of the Interface Between a Sn-Zn-Bi Solder and a Substrate by Using the Angle-Lapping Method: *Nobuhiro Ishikawa*[1]; [1]National Institute for Materials Science, High Voltage Electron Microscopy Sta., 3-13, Sakura, Tsukuba, Ibaraki 305-0003 Japan

The microstructure of the interface between a Sn-8wt%Zn-3wt%Bi Pb-free solder and a Cu substrate by packaging of ball grid assembly (BGA) process has been studied. For preparing the specimen, angle-lapping method was applied to analyze the interface between solder and substrate three dimensionally by combination with the analysis from cross-sectional direction. Transmission electron microscope (TEM) was used mainly for analysis. The wettability of the solder and the substrate was not bad because there were few voids on both side of the intermetallic compound(IMC) layer formed at the interface. This result is different from the use of Ni-P electroless plate as the substrate. But many cracks were found inside the IMC layer and their distribution was homogeneous. These cracks may affect the mechanical property of the interface. There also found at least five phases stacked inside the IMC layer.

4:20 PM
Controlling Intermetallic Compound Growth in SnAgCu/Ni-P Solder Joints by Nano-Sized Cu6Sn5 Addition: Szu-Tsung Kao[1]; Yung-Chi Lin[1]; *Jenq-Gong Duh*[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Nanosized Cu_6Sn_5 dispersoids were incorporated into Sn and Ag powders and milled together to form Sn-3Ag-0.5Cu composite solders by mechanical alloying process. In this study, it is aimed to investigate the interfacial reaction between SnAgCu composite solder and electroless Ni-P/Cu UBM after annealing at 240°C for 15 min. The growth of the IMCs formed at composite solder/EN interface was retarded as compared to the commercial Sn3Ag0.5Cu solder joints. With the aid of the elemental distribution by x-ray color mapping in the electron probe microanalysis (EPMA), it is demonstrated that the SnAgCu composite solder exhibited refined structure. It is proposed that the Cu_6Sn_5 additives was pinned on the grain boundary of Sn after annealing and thus retarded the movement of Cu toward the solder/EN interface to form interfacial compounds. In addition, wetting is an essential prerequisite for soldering to ensure good bonding between solder and substrate. It was demonstrated that the contact angles of composite solder paste was less than 25°, indicating good wettability.

4:40 PM
Effect of Electromigration on Intermetallic Compound Growth at the Interfaces Between Ni and Eutectic SnAg(Cu): *Shengquan Ou*[1]; King-Ning Tu[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., 405 Hilgard St., Los Angeles, CA 90095-1595 USA

Formation of intermetallic compound (IMC) at the solder joint interfaces is essential in bonding the parts together. However, excessive IMC formation will cause detrimental effect on the joint strength, e.g., due to Kirkendall void formation. Moreover, under electric current stressing, dramatically different IMC growths at the cathode and anode may become a reliability problem in electronic packaging. We have used eutectic SnAg(Cu) solder lines with Ni wire-electrodes in v-grooves etched on (100) Si surfaces to study the effect of electromigration on IMC growth. The effects of temperature (120 to 180°C), current density (1 to 3E4 A/cm2) and length of the solder line (200 to 800 micron) will be discussed.

5:00 PM
A Study on Tin Whisker Growth on Electroplated Tin in Terms of Substrate, Surface Oxides, and Stress Evolution in Sn-Cu Film: *Dong-Min Jang*[1]; Jin Yu[1]; Taek-Yeong Lee[2]; [1]Korea Advanced Institute of Science and Technology, Matl. Sci. & Engrg., 373-1 Guseong-Dong, Yuseong-gu, Daejon 305-701 S. Korea; [2]Hanbat University, Matl. Sci. & Engrg., San 16-1 Dukmyung-dong, Yuseong-gu, Daejon 305-719 S. Korea

Study of tin whisker growth on pure tin was mostly carried out in 1970's and recently many publications were presented to provide standard acceleration test procedures and growth mechanism. However some disagreement and conflicting results among investigators exist and more systematic understanding is required to resolve tin whiskering. In this study tin whisker growth on electroplated Sn/Cu was investigated. The effect of mechanical polishing of Cu layer before tin plating was presented. Tin whiskers were found only after 12 hours in case of the polished Cu layer. The maximum length of whisker is about 40§- after 3 days. Both compressive stress and surface oxides on tin film are necessary conditions for tin whisker growth, which has not yet been quantitatively measured. The type and amount of surface oxides on tin

film were characterized and stress evolution in Sn/Cu film was measured in-situ at two conditions, air and vacuum at 60C.

5:20 PM
Impact and Thermomechanical Reliability of Sn-Ag-In Solder Joint: *Tatsuya Shoji*[1]; Masayoshi Date[1]; Masaru Fujiyoshi[1]; Koji Sato[2]; [1]Hitachi Metals, Ltd., Metallurgl. Rsch. Lab., 2107-2, Yasugi-cho, Yasugi, Shimane Japan; [2]Hitachi Metals, Ltd., Yasugi Works

Sn-Ag-In ternary alloys were evaluated regarding microstructures and mechanical properties. In the case of Sn-0.3Ag-2In solder bumps, a small number of particles of intermetallic compounds (IMCs) were dispersed within the bulk, and a thin IMC layer was formed at the bond interface with an electroplated Ni/Au pad, compared with Sn-3Ag-0.5Cu solder bumps. Impact reliability of the daisy-chained BGA solder joints was evaluated by using a micro-Charpy impact tester. Impact was applied directly to a substrate whose bond-pads were an electroplated Ni/Au or an organic solder preservative (OSP) coated Cu. The magnitude of the applied impact was changed to investigate its effect on failure lives. We found Sn-Ag-In alloys had longer failure lives than the Sn-3Ag-0.5Cu or the Sn-Pb solder. A thermal cycle test for modules mounted BGA packages was also performed. Sn-Ag-In alloys showed low probability of initial failure compared with that of Sn-3Ag-0.5Cu, although its median life was shorter than that of Sn-3Ag-0.5Cu.

Magnesium Technology 2005: Magnesium and Alloys - Refining, Recycling and Fundamentals

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Monday PM — Room: 2004
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Ramana G. Reddy, University of Alabama, Tuscaloosa, AL 35487-0202 USA; Ralph Harris, McGill University, Mining, Montreal, Quebec H3A 2B2 Canada

2:00 PM
Castability of Magnesium Alloys: *Amanda L. Bowles*[1]; Qingyou Han[1]; Joe A. Horton[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA

There is intense research effort into the development of high pressure die cast-able creep resistant magnesium alloys. One of the difficulties encountered in magnesium alloy development for creep resistance is that many additions made to improve the creep properties have reportedly resulted in alloys that are difficult to cast. It is therefore important to have an understanding of the effect of alloying elements on the castability. This paper gives a review of the state of the knowledge of the castability of magnesium alloys.

2:20 PM
Microstructure and Phase Transformations in Quasicrystal-Containing Mg-Zn-Y Alloys: *Alok Singh*[1]; Masaki Watanabe[2]; Akira Kato[2]; An-Pang Tsai[3]; [1]National Institute for Materials Science, Matls. Engrg. Lab., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Toyota Motor Corp., Matls. Engrg. Div. II, Toyota-cho, Toyota, Aichi 471-8572 Japan; [3]Tohoku University, Inst. of Multiplinary Rsch. for Advd. Matls., 2-1-1 Katahira, Sendai, Miyagi 980-8577 Japan

The strength and deformation behavior of quasicrystal-containing dilute Mg-Zn-Y alloys after hot-rolling or extrusion at 400C have recently been reported. These alloys exhibit high ductility with moderate strength and a highly stable microstructure at elevated temperatures. We report here the effect of heat treatment on two extruded alloys Mg95Zn4.2Y0.8 and Mg92.5Zn6.5Y. The quasicrystal particles, facetted and showing a definite orientation relationship with the matrix, become rounded on heating to 400C. In the matrix, fine rods of MgZn2 precursor phase occur parallel to the hexagonal axis. A ternary phase tau1 related to Mg4Zn7 precipitates in the matrix on annealing of alloy Mg95Zn4.2Y0.8, to be replaced by precipitates of quasicrystal phase on further annealing. In the alloy Mg92.5Zn6.5Y, the quasicrystal phase transforms to a hexagonal Mg25Zn58Y17 phase at 400C. The quasicrystal then reprecipitates on its interface, forming a nano-composite. Effects of microstructural changes on the deformation behavior are described.

2:40 PM
Phase Transformation and Creep of Die-Cast Mg-Al-Ca Based Alloys: *Akane Suzuki*[1]; Nicholas D. Saddock[1]; J. Wayne Jones[1]; Tresa M. Pollock[1]; [1]University of Michigan, Matls. Sci. & Engrg., 3062 H.H. Dow, 2300 Hayward St., Ann Arbor, MI 48109 USA

The stability of microstructure and phases in die-cast AC53 and AXJ530 was investigated in detail by TEM. In both alloys, crystal structure of the eutectic intermetallic compound $(Mg, Al)_2Ca$ at grain boundaries was identified as dihexagonal C36 structure. The C36 phase is not stable and transforms into Al_2Ca (C15 structure, cubic) with aging at 573 K. Since this transition proceeds by shearing of the closed-packed planes with crystallographic orientation relationship of $(0001)_{C36}\|\{111\}_{C15}$ and $[2\text{-}1\text{-}10]_{C36}\|[01\text{-}1]_{C15}$, the network structure of intermetallic compound surrounding α-Mg grains is fairly stable after prolonged exposure at elevated temperature. In addition, precipitation of Al_2Ca in α-Mg was observed after 360 ks aging at 573 K. The precipitates have disc-shape with habit plane of $\{111\}_{C15}\|\{0001\}_{\alpha}$. These transformations are caused by ejection of supersaturated Al and Ca from α-Mg phase. Dislocation substructure evolved during creep deformation will be presented, and relationship between creep properties and microstructure will be discussed.

3:00 PM
Grain Refinement of Mg Alloys by Nanoscaled TiN Particles: *Gabriele Vidrich*[1]; Oliver Moll[1]; Hans Ferkel[1]; [1]TU Clausthal, Inst. for Matls. Sci. & Tech., Agricolastr. 6, Clausthal-Zellerfeld 38678 Germany

A new method was developed which allows a grain refinement of Mg alloys via casting from Mg melts containing nanoscaled TiN particles. The dispersed particles in the melt act as nucleation sites of Mg grains during solidification. Grain refinement was observed by light-microscopy of ingots which were cast from AZ91 melts containing dispersed TiN nanoparticles of particle volume fraction in the melt below 0.2 %. Structure investigations were also carried out by electron microscopy and X-ray diffraction. It was shown that TiN nanoparticles are stable in AZ91 melt for several hours at 700°C. This is a requirement to gain a heterogeneous nucleation of Mg grains on TiN seeds. The results are discussed.

3:20 PM
Inspection of Grain Boundary Sliding in Die-Cast Mg Alloys During Creep Deformation Via Strain Mapping Techniques: *Nicholas David Saddock*[1]; Tresa M. Pollock[1]; Akane Suzuki[1]; Samuel Charles Wildy[2]; J. Wayne Jones[1]; [1]University of Michigan, Matl. Sci. & Engrg., 2300 Hayward Rd., 3062 H.H. Dow, Ann Arbor, MI 48109 USA; [2]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX 13Ph UK

Numerous aluminum containing, magnesium-based die-cast alloys (e.g. AZ91, AS21, AE42 and AC53) exhibit a transition from power law creep at intermediate stresses to a stress dependence of 1-2 at low stresses, indicating the possibility of change in creep mechanism from grain boundary sliding (GBS) to dislocation controlled processes. However, direct experimental evidence of GBS is seldom reported, especially at lower strains. In the present study a high-resolution strain mapping technique developed in previous studies is employed to examine the accumulation of strain during creep of AM50 and AXJ530, the latter containing 3 wt. % calcium. The influence of Ca on the morphology and stability of grain boundary microstructure and its corresponding influence on the development of strain localization in these alloys is explored. Microstructural evolution and the development of dislocation structure, as ascertained by TEM will be described, along with the mechanisms for creep deformation in these alloys at low stresses.

3:40 PM Break

3:55 PM
The Development of New Additions for Liquid Magnesium: Kang Sun[1]; *Stavros A. Argyropoulos*[1]; Tim Kosto[2]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Toronto, Ontario M5S3E4 Canada; [2]Milward Alloys, Lockport, NY 14094-1712 USA

The exothermic behavior of the intermetallic reactions inside various metallic powder compact and its assimilation fundamentals in molten magnesium were examined. The work was focused on two types of compacts. Specifically Mn-Al and V-Al compacts were examined. In this research work cylindrical compacts were manufactured and tested. The experimental work involved simultaneous temperature measurements of three thermocouples as well as detection of the apparent weight of the cylindrical specimen during its assimilation in liquid magnesium. The details of the intermetallic reactions prior to the compact complete disintegration were investigated by microscopically studying the morphology and local composition in a partially

reacted compact. The exothermic reaction increases the compact's temperature above that of the liquid magnesium. In tandem with this exothermic reaction, the compact is suffering a swelling action. This swelling action disintegrates the cylindrical specimen, which is assimilated into the liquid magnesium.

4:15 PM
Numerical Simulations of Radiative Heat Transfer Between High-Temperature Fluidized Beds and Magnesium Castings: *Saytavur Ispandiyar Bakhtiyarov*[1]; [1]Auburn University, Mech. Engrg., 202 Ross Hall, Auburn, AL 36849-5341 USA

Fluidized beds are considered as an efficient alternative to conventional heat treatment operations. The advantages of fluidized beds heat treatment processes are (1) a very high heat treatment rates, (2) temperature uniformity, flexibility of the fluidized bed furnaces (they can operate at any temperature and with any atmosphere, (4) the process is simple to operate and untrained persons can operate furnace and set process parameters. However, the radiative heat transfer in fluidized bed furnaces was not studied systematically in the literature. Existing theoretical models for predicting radiative heat transfer between fluidized beds and castings usually use the Stefan-Boltzman equation. These models require the total effective radiative coefficient to be obtained experimentally. This paper presents the results of numerical simulations, where the absorption coefficient is defined as a function of bed characteristics.

4:35 PM
Scale-Up of Magnesium New Rheocasting from a Laboratory Level to an Industrial Process: *Werner Fragner*[1]; Matthias Gruber[2]; Gernot Macher[2]; [1]ARC Light Metals Competence Center Ranshofen GmbH, Casting Tech., Postfach 26, Ranshofen, Upper Austria 5282 Austria; [2]Non Ferrum Metallpulver Ges.m.b.H. & Co. KG, Austria, PO Box 11, St. Georgen bei Salzburg, Salzburg 5113 Austria

Sludge, dross and black dross are among the most demanding by-products of Magnesium handling and recycling. They are reactive when coming into contact with water including hydrogen and ammonia formation. Especially black dross is a recycling problem being very reactive with its fine magnesium particles and high salt and magnesium oxide content. Together with LKR Non-Ferrum developed a way of treating these by-products either in a dry or a wet way, depending on the incoming material and recovering its components that are magnesium, magnesium oxide and salt. The products from both processes can be reused and are not reactive. With the recovery of metallic Mg the treatment is an economical and ecological feasible way to process sludge, dross and black dross.

4:55 PM
Economic Analysis of the Carbothermal Production of Magnesium: *Robert R. Odle*[1]; Andrew W. McClaine[2]; Jens Frederiksen[3]; [1]Metallurgical Viability, Inc., 60 Blue Hen Dr., Ste. 3000, Newark, DE 19713 USA; [2]Safe Hydrogen, LLC, 30 York St., Lexington, MA 02420-2009 USA; [3]PF&U Mineral Development ApS, Kullinggade 31 5700 Svendborg DK

A techno-economic model of a novel carbothermal process for the production of magnesium using Magnesium Technologies Limited (MTL) patented process has been carried out. Feedstock for the process is a relatively pure MgO recycled from Safe Hydrogen's Magnesium Hydride Slurry Process for hydrogen production, transportation, and storage. The model allows the user to alter design assumptions on most unit operations, composition and cost of selected raw materials, some project conditions including metal prices, labor costs, staffing, and energy costs. The model demonstrates the power of Visual Basic for Applications (VBA) coupled with an Excel Spreadsheet interface for producing steady state material and energy balances, income statements, and an estimate of gross profits for the conditions selected. The model facilitates "what-if" analysis and allows the user to intuitively establish the critical assumptions/conditions for economic viability. The carbothermal process for magnesium promises a relatively simple, automated process with low capital costs and competitive operating costs compared to electrolytic and silicothermic processes. The application of this technology has been limited by the lack of a method to rapidly cool the Mg(vapor) and CO to prevent the back reaction. MTL is developing a technology that promises to solve this problem. Safe Hydrogen is interested in evaluating this technology since their hydrogen storage technology used with a fuel cell technology becomes competitive with gasoline powered internal combustion engines as the price of magnesium drops.

5:15 PM
Co-Existing Phases in the Al-Ca-Mg System: Rai Raz[1]; Shaul Avraham[1]; *Menahem Bamberger*[1]; [1]Technion, Dept. of Matls, Engrg., Technion City, Haifa 32000 Israel

The use of Mg alloys at elevated temperature application can contribute to weight reduction both in critical components and overall weight, that will lead to economical and environmental benefits. The elevated temperature mechanical properties of Mg-Al-Ca alloys is comparable to those of RE alloys. This behavior is attributed to the formation of Al_2Ca precipitates and the fine microstructure of these alloys. The Al-Ca-Mg system is yet to be thoroughly studied. This work will present recent results related to the ternary Al-Ca-Mg phase diagram, which are essential for further thermodynamical simulations.

Magnesium Technology 2005: Wrought Magnesium Alloys II

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Monday PM — Room: 2006
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Sean R. Agnew, University of Virginia, Dept. of Matl. Sci. & Engrg., Charlottesville, VA 22904-4745 USA; Peter Pinfold, Fluor Corp, Alcoa, TN 37701 USA

2:00 PM
Microstructural Evolution During Severe Deformation of AZ-31 Magnesium Alloy Under Non-Isothermal Process Condition: *Yi Liu*[1]; *Amit K. Ghosh*[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109 USA

Large plastic deformation was imparted to Mg alloy plate AZ-31B by compressing along the in-plane direction, in progressive stages below 300C, via non-isothermal processing method and under superimposed hydrostatic pressure. It was shown that by progressively lowering the deformation temperature, microstructures of ultrafine grain size, approaching nanocrystalline structure could be obtained. Starting from a bimodal grain structure, the volume fraction of fine grains increased significantly as grain subdivision by twinning mechanism was utilized to achieve grain refinement. X-ray diffraction results indicated that a strong basal fiber texture emerged normal to the original plate rolling direction, as a result of the new deformation mode, and a very high strength level for such alloys resulted due to reduced dynamic recovery effects.

2:20 PM
Bendability and Microstructure of Magnesium Alloy Tubes at Room and Elevated Temperatures: *Alan A. Luo*[1]; Anil K. Sachdev[1]; Raja K. Mishra[1]; [1]General Motors Research & Development Center, Matls. & Processes Lab., 30500 Mound Rd., MC 480-106-212, Warren, MI 48090 USA

This paper investigates the bendability and microstructure of magnesium alloy AZ31 tubes at room and elevated temperatures. The results suggest limited bendability for magnesium alloy tubes at room temperature. Microstructural analysis indicates that strain localization caused by twinning initiates cracks at the surface which consequently lead to fracture upon room-temperature bending. A moderate temperature (150-200°C) bending process has been developed for magnesium alloy tubes. Microstructural evaluation indicates that twinning is an important deformation mechanism in moderate temperature bending. A significant amount of localized dynamic recrystallization is evident at prior grain boundaries when bending at above 200°C.

2:40 PM
An Internal Variable Approach to the Superplastic Deformation of AZ31 Magnesium Alloy: *Hyunseok Lee*[1]; Won Kyu Bang[2]; Hwan Jin Sung[2]; Young Won Chang[1]; [1]Pohang University of Science & Technology, Matl. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Kyung-buk 790-784 S. Korea; [2]Research Institute of Industrial Science & Technology, San 32, Hyoja-dong, Nam-gu, Pohang, Kyung-buk 790-600 S. Korea

Internal variable theory, proposed by Chang et al., has manifested as an inelastic deformation model based on the dislocation dynamics. The constitutive model is formulated with internal variables corresponding to the deformation state of the material, accordingly has more solid physical basis. In the previous studies, the theory can be demonstrated as a useful method for the interpretation of the structural superplasticity and has been applied successfully for various metallic materials. In the present study, a series of tensile tests of AZ31

magnesium alloy has been carried out to investigate superplastic deformation behavior with temperature variation. Analysis of load relaxation test results based on internal variable theory gave further information about superplastic deformation and its accommodation mechanism.

3:00 PM
Infrared Processing and Hot Rolling of Magnesium Alloys: *Joe A. Horton*[1]; Amanda L. Bowles[1]; Craig A. Blue[2]; Sean R. Agnew[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S-MS6115, PO Box 2008, Oak Ridge, TN 37830 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg 4508-MS6083, PO Box 2008, Oak Ridge, TN 37831-6083 USA; [3]University of Virginia, Matls. Sci. & Engrg., 116 Engr.'s Way, Charlottesville, VA 22904-4745 USA

It is of interest to produce magnesium wrought products more efficiently in order to make them more attractive for applications. Use of radiant heating by infrared or plasma arc lamps have the potential to reduce processing steps and thereby save time and cost. Previous studies at a commercial rolling facility equipped with infrared lamps have shown that this process is feasible with AZ31B. Comparisons of microstructures, mechanical properties, and crystallographic textures for various hot rolling temperatures attained by pack rolling will be presented and correlated with the infrared processing results. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Strength Weight Reduction Materials Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

3:20 PM
The Types and Roles of Deformation Twinning in AZ31 Mg Alloys: *Junichi Koike*[1]; Takeo Miyamura[1]; Akira Nishinoiri[2]; Manabu Enoki[2]; [1]Tohoku University, Dept. of Matls. Sci., 02 Aoba, Aramaki, Aoba-ku, Sendai 980-8579 Japan; [2]University of Tokyo, Dept. of Matls. Sci., Hongo, Bunkyo-ku, Tokyo Japan

Rolled sheets of AZ31 magnesium alloys were tensile tested at room temperature. Twinning behavior was observed at various strain levels up to a pint of fracture, using an optical microscope, an orientation imaging microscope and an acoustic emission system. Twinned area was increased in two separate stages with strain. Initial rapid increase was observed near the yield point, due to the formation of wide lenticular twins. This was followed by another rapid increase close to the failure point, due to the formation of narrow banded twins. Acoustic emission (AE) signals also exhibited two separate stages with strain, which enabled us to relate each twin type with FFTed AE signals. The each type of twin will be discussed in light of its role on deformation and fracture mechanisms.

3:40 PM Break

3:55 PM
Adapted Extrusion Technology for Magnesium Alloys: *Jan-F. Lass*[1]; Friedrich-W. Bach[1]; Mirko Schaper[1]; [1]University of Hannover, Inst. of Matls. Sci., Schönebecker Allee 2, Hannover D-30823 Germany

The rising efforts for an ecologically harmless capacity increase of technical products and processes not only in the automobile industry result in the renaissance of magnesium within the last years. Therefore a variety of magnesium components are already applied in automobiles. But this is nearly restricted to diecasting components. One reason for this is the extremely limited spectrum of adequate magnesium wrought alloys and above all the extrusion technology for the production of adequate profiles which is not explored very well. Especially in the construction of automobiles, increased attempts are made to apply magnesium in bearing structures. Therefore it is necessary to develop an adequate and economical materials processing for the extrusion of magnesium alloys. Moreover also a process method must exist which specifically influences the material properties of extruded magnesium profiles. This article gives a general view about the problems and difficulties which are included in the extrusion process as well as in the resulting material properties of extruded magnesium profiles. Furthermore several possibilities will be described to solve these problems in different ways. Here the examination of the initial materials and their properties, the whole extrusion process as well as the subsequent treatment of the extruded magnesium profiles is very important.

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Constitutive Behavior of AZ31B Mg Sheet: Development and Implementation of Constitutive Model: *Min Li*[1]; XiaoYuan Lou[1]; Frederic Barlat[2]; Robert H. Wagoner[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Alcoa Inc., Alcoa Techl. Ctr., Matls. Sci. Div., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

The mechanical response of basal-textured AZ31B magnesium sheet has been measured in uniaxial tension, in-plane compression, and simple shear using recently developed tests. Reversed paths have been imposed using the same tests. Twinning occurs in compression and untwinning occurs in subsequent tension, with consequently lowered yield stress and rapid work hardening. The yielding asymmetry and unusual evolution and reversal of hardness are difficult to model with standard plasticity formulations. A new formulation has been developed to reproduce the key features of the path dependence of hardening with a view toward implementation in commercial finite element programs. Using such a capability, it may be possible to devise novel forming techniques to take advantage of the unusual room-temperature behavior of AZ31B.

4:35 PM
Microstructure Development of Extruded Mg Alloys: *Soeren Mueller*[1]; Klaus Mueller[2]; Walter Reimers[1]; [1]Technical University, Inst. for Matls. Sci. & Tech., Ernst-Reuter-Platz 1, Sekr. BH 18, Berlin 10587 Germany; [2]Technical University, Extrusion R&D Ctr., Gustav-Meyer-Allee 25, Sekr. TIB 4/1-2, Berlin 13355 Germany

In order to obtain light-weight constructions in the automobile and aircraft industry the extrusion of magnesium profiles is an important factor. Magnesium profiles of the AZ alloy family have been extruded in the direct, indirect and hydrostatic extrusion method at the extrusion research and development center of the Technical Universtity of Berlin. These profiles were then tested at the institute for materials science and technology of the Technical Universtity of Berlin for their microstructure. Thereby the different samples exhibited a different microstructure as well as different mechanical properties. Some of these samples were taken to another deformation after the extrusion process and then also tested for their microstructure and mechanical properties. The following paper will discuss the difference in the extrudates coming from the direct, indirect and hydrostatic extrusion as well as the extrudates which were further deformed in order to optimize the mechanical properties.

4:55 PM
Constitutive Behavior of AZ31B Mg Sheet: Measurement and Analysis of Mechanical Properties: *Xiaoyuan Lou*[1]; Richard K. Boger[1]; Frederic Barlat[2]; Robert H. Wagoner[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Alcoa Technical Center, Matls. Sci. Div., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

HCP alloys have a limited number of non-basal slip systems available. For basal-textured AZ31B magnesium sheet, the activation energy for twinning is asymmetric, leading to a strong difference in the tensile and compressive flow curves. To provide information on the competition of deformation mechanisms of slip, twinning, and untwinning, the mechanical behavior of AZ31B was examined under non-proportional load paths. Techniques have been developed to deform magnesium sheet under in-plane tension/compression and reversed, simple shear. Acoustic emission techniques were used in conjunction with the mechanical tests to detect and quantify the twinning and untwinning mechanism. The results of the mechanical testing are used in combination with the initial and deformed texture to inform the development of a new constitutive model for this alloy.

5:15 PM
In-Situ Investigation of Twinning Behaviour in Mg-3Al-1Zn: *Zohreh Keshavarz*[1]; Matthew R. Barnett[1]; [1]Deakin University, Sch. of Engrg. & Tech., Pigdons Rd., Geelong, VIC 3217 Australia

Magnesium alloys are attractive for automotive and aerospace industries, due to their low density. One problem with these alloys is their limited formability at room temperature. Twinning plays a dominant role in deformation behaviour and it can be expected that an increased understanding of twinning will help improve formability. In the present work, the behaviour of different twinning systems in as-cast Mg-3Al-1Zn is investigated using in-situ tensile tests in a scanning electron microscope. Electron backscatter diffraction and back scatter electron imaging were carried out during the tests. The results show both tension and compression twinning are active at room temperature and that twinning and untwinning occur both during loading and unloading.

Materials Processing Fundamentals: Liquid Metal Processing

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Monday PM Room: 3001
February 14, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

2:00 PM
Flow Conditions at the Meniscus of a Continuous Casting Mold Through a Physical Model Approach: Carlos Antonio Silva[1]; *Varadajan Seshadri*[2]; Itavahn Alves Silva[1]; Versiane Albis Leão[1]; Dimas Bahiense Moreira[3]; [1]Universidade Federal de Ouro Preto, Escola de Minas/Engrg. Metalúrgica, Campus do Morro do Cruzeiro S/N, Ouro Preto, Minas Gerais 35400-000 Brazil; [2]Universidade Federal de Minas Gerais, Metallurgl. Engrg., Rua Espirito Santo, 35, Belo Horizonte, Minas Gerais Brazil; [3]Cia Siderurgica Tubarao, Av. Brigadeiro Eduardo Gomes 930, Serra, Espirito Santo 29163-970 Brazil

Internal and sub-superficial defects of continuously cast steel plates have been associated with flow conditions at the metal-slag interface. Turbulence plays a main role in regard to emulsification of top slag and entrapment of inclusions. The objective of this study was to evaluate the flow pattern as a function of SEN geometry and operating conditions. A 1:1 physical model was used in order to allow testing of actual nozzles. A custom made sensor was developed for measuring sub-surface velocity midway. Experimental data as well as flow visualization indicate a high degree of superficial turbulence for some combinations of geometry and nozzle penetration. These combinations are indicated as detrimental to steel quality.

2:25 PM
Thermodynamics on the Solidificational Refining of Si with Si-Al Melts: *Takeshi Yoshikawa*[1]; Kazuki Morita[1]; [1]University of Tokyo, Dept. of Matls. Engrg., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

Recently, the amount of solar cell production, especially that of polycrystalline silicon, increases significantly with a growing demand for clean energy. Solar grade silicon (SOG-Si), however, would be short of supply with increasing demand because of its dependence on a semiconductor industry. Therefore the development of an innovative low-cost mass production system for polycrystalline silicon is required. The authors have been trying to develop the low temperature silicon refining process, "solidification refining of silicon with Si-Al melts at 1173-1273K". In this study, segregation ratios of impurities such as B, P, Fe, etc., between solid Si and Si-Al melt were experimentally or theoretically determined to evaluate the potency for purification in this process. For almost all impurities, segregation ratio at 1273K was obtained much smaller than segregation coefficient between solid/liquid Si, therefore low temperature solidification process is found to be more effective for purification compared with ordinal solidification refining.

2:50 PM
Measurement of Natural Convection in Ga Melts With and Without Applied Magnetic Fields: *B. Xu*[1]; *Ben Q. Li*[1]; [1]Washington State University, Sch. of Mechl. & Matls. Engrg., Pullman, WA 99164 USA

Results from an experimental study on natural convection induced by temperature gradient in molten gallium contained inside a rectangular container are presented. The damping effect of the convective flow from an externally applied static magnetic field is investigated. Induced velocity and temperature fields are mapped using a standard constant temperature hot-film anemometry and a thermocouple with and without the applied magnetic field, respectively. Detailed experimental setup and procedure are depicted in the present paper. Obtained velocity and temperature profiles are examined against previous numerical simulations and reasonably good agreement has been achieved. Obvious damping effect can be observed in both the temperature and the velocity profiles when the external magnetic field presents.

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Numerical Simulation of Molten Steel Flow Under FC Mold Magnetic Field: *Hwasoo Park*[1]; Misun Im[1]; [1]Kookmin University, Advd. Matls. Engrg., Jung-Nung 3Dong,Sung-Buk Ku, Seoul 136-702 Korea

Numerical simulation has been performed to analyze the characteristics of a molten steel flow in the continuous casting process. Momentum and energy equation has been coupled considering the solidification of steel along with turbulence ê-å model. The vertical bending mold has bifurcated nozzle with 15 degrees of downward angle and FC mold supplies DC induced magnetic fields. The discrete phase model is employed to calculate the molten metal-argon gas flow with Rosin-Rammler distribution law and cloud particle tracking method. Magnetic fields not only change the magnitude of flow vectors but also the direction of flow especially near corner area . They suppress the velocity vectors at port outlet and increase the steel temperature at free surface and inside the mold.

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Evaluation of Y2O3 as Front Layer of Ceramic Crucibles for Vacuum Induction Melting of TiAl Based Alloys: *Jose Joaquim Barbosa*[1]; Carlos Silva Ribeiro[2]; Antonio Caetano Monteiro[1]; [1]University of Minho, Mechl. Engrg., Campus de Azurem, Guimaraes 4800-058 Portugal; [2]University of Porto, FEUP, Rua Roberto Frias, Porto 4200-465 Portugal

During the last decades titanium alloys were found to be valuable engineering materials for many different applications. Formerly used in critical applications like aerospace, aeronautic and military equipment, where the factor cost is not relevant, titanium alloys are finding now new and different markets. However, the development of such new markets will depend on an effective cost reduction of titanium parts, in order to achieve a selling cost suitable with its application in consumer goods. A possible solution to decrease production costs might be the use of traditional casting techniques to produce near net shape functional parts. During the last years, the authors have developed extensive research work on this field, and a new technique both for melting and moulding, using ceramic multi-layered crucibles and investment casting shells was developed. This paper presents some of the results obtained during that research work: a Ti-48Al alloy was melted and cooled inside CaO, MgO and Y2O3 stabilized ZrO2 crucibles with inside layer of Y2O3. The chemical composition, hardness and microstructure at the metal-crucible interface, studied by secondary ion mass spectrometry, SEM/EDS and XRS are presented. On a second step, the same alloy was melted on the same crucibles, and poured into graphite moulds, and the crucibles wall was characterized by SEM/EDS and XRS.

4:20 PM
Simulation of Decarburization of a High Alloyed Liquid Steel Using the Reactor Module of Thermo-Calc: *Lina Kjellqvist*[1]; Bo Sundman[1]; [1]Royal Institute of Technology, Dept. of Matl. Sci. & Engrg., SE-10044, Stockholm Sweden

Thermodynamic software and databases are now powerful and accurate enough to give reliable results when applied to complex processes like the decarburization of liquid steels. The reactor module in Thermo-Calc makes it possible to simulate the reduction of carbon in a liquid high alloyed steel by a mixture with oxygen and argon gases. The process is divided into a small number of local equilibria and the gas, liquid and slag phases are distributed between them based on experimental results and fluid flow simulations. A test case using a stainless steel will be compared to experimental data demonstrating the powerful user interface. Future work will lead to an integration of the thermodynamic software in the fluid flow simulations and the additions of S and P in the thermodynamic simulations.

4:45 PM
Modeling of Scrap Melting in an EAF: Diancai Guo[1]; *Gordon A. Irons*[1]; [1]McMaster University, Steel Rsch. Ctr., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

Scrap melting in an EAF is modeled numerically to gain some insight into the heat utilization during a heat. Scrap movement, gas flow and heat exchange are simulated, heat loss and burner heating efficiency discussed. The model enables to determine how much heat the charge can gain from burners before it is melted, the temperature distribution of the scrap pile during melting process, and how much heat lost through radiation and convection to furnace wall and roof.

5:10 PM
Preliminary Investigation of Fluid Mixing Characteristics in Bath During Side and Top Combined Blowing AOD Refining Process of Stainless Steel: *Ji He Wei*[1]; Hong Li Zhu[1]; Guo Min Shi[2]; Qing Yan Jiang[2]; Sen Long Yang[1]; Xin Chao Wang[1]; Jin Chang Ma[1]; He Bing Chi[2]; Li Bing Che[2]; Kai Zhang[2]; [1]Shanghai University, Dept. of Metallic Matls., 149 Yan Chang Rd., Shanghai 200072 China; [2]The Shanghai No. 1 Iron and Steel Company Ltd, Bao Steel (Group) Corporation, W. Changjiang Rd., Shanghai China

The mixing characteristics of the fluid in the bath of 120 t AOD converter during the refining process of stainless steel with side and top combined blowing have been preliminarily examined on a water model unit. The geometric similarity ratio between the model and its prototype (including the side tuyeres and the top lance) was 1:4. Based on the theoretical calculations for the parameters of the gas stream in the tuyeres and the lance, the gas blowing rates used for the model have been more reasonably determined. The influence of the tuyere number and position, and the gas flow rates for side and top blowing on the mixing characteristics has been considered. The results demonstrated that the fluid in the bath underwent vigorous circulatory motion during the blowing, without obvious dead zone in the bath, resulting a well mixing effectiveness. the gas flow rate of the main-tuyere had a governing role on the characteristics, and the gas jet of sub-tuyere can increase mixing efficiency, and the gas jet from the top lance can prolong the mixing time. Corresponding to the specified oxygen top blowing flow rate by the technology, six side tuyeres with an angle of 27 degree between each tuyere, five side tuyeres with an angle of 22.5 or 27 degree can all reach an essentially equivalent and good mixing effectiveness. The relationships of the mixing time with the gas blowing rates of the main-tuyeres and sub-tuyeres and top lance, the angle between each tuyere, and the tuyere number have been obtained.

Mechanical Behavior of Thin Films and Small Structures: Plasticity and Deformation Mechanisms at Small Length Scale

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

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Session Chairs: Evan Ma, Johns Hopkins University, Dept. of Matls. Sci. & Engrg., Baltimore, MD 21218 USA; David J. Srolovitz, Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton, NJ 08544 USA

2:00 PM Invited
Deformation at the Nanometer and Micrometer Length Scales: Effects of Strain Gradients and Dislocation Starvation: *William D. Nix*[1]; Gang Feng[1]; Julia R. Greer[1]; [1]Stanford University, Dept. Matls. Sci. & Engrg., 416 Escondido Mall, Stanford, CA 94305-2205 USA

Size effects in plasticity are now well known. Plastic deformation in small volumes requires higher stresses than are needed for plastic flow of bulk materials. Here we review the various effects that appear to be responsible for this. The size dependence of the hardness of metals at the micron scale can be described in terms of the geometrically necessary dislocations or, correspondingly, the strain gradients, created in small indentations. But such accounts break down when the size of the deformation volume begins to approach the spacing of individual dislocations or when the crystal becomes dislocation starved. Nanoindentation of epitaxial films at the nanometer depth scale reveals irregular load-displacement curves. In this domain the nucleation of dislocations and plasticity under dislocation-starved conditions appears to be more important than strain gradients. Recent uniaxial compression experiments on tiny samples of gold made by focused ion beam machining and integrated circuit fabrication methods show strong size effects on plasticity, with sub-micron sized crystals showing remarkable strengths after plastic deformation. These experiments involve small deformation volumes and minimal strain gradients. These size effects may be explained by considering a process of strain hardening by dislocation starvation, wherein existing dislocations leave the crystal more frequently than they reproduce themselves by multiplication.

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Contact-Induced Plasticity of Rough Surfaces Under Nanoindentation: E. Buchovecky[1]; *F. Sansoz*[1]; [1]University of Vermont, Mechl. Engrg., 33 Colchester Ave., 201 Votey Bldg., Burlington, VT 05405 USA

The contact mechanics between two nanoscale surfaces has drawn considerable interest in recent years because of its importance to MEMS tribology, nanoindentation, scanning probe microscopy, and coating deformation. Despite current knowledge that nanostructured film surfaces are not perfectly flat at the nanoscale, the influence of surface asperities on contact mechanics during nanoindentation is not fully understood. In addition, it is unclear how the presence of surface asperities, which create highly-stressed points of contact, significantly alters the incipient plasticity of indented films. In this study, we measure and analyze though atomic force microscopy the topography of nanocrystalline Ni surfaces of different grain size. The quasicontinuum method is then used to model the nanoindentation of rough Ni surfaces. The response of atomically-flat, stepped, and experimentally observed surface profiles is modeled under either cylindrical or prismatic indenter geometries. This study demonstrates the key role of surface roughness on the onset of plasticity in indented films and coatings.

2:40 PM
Critical Shear Stress for Onset of Plasticity in Nanocrystalline Cu Films Determined by Using Nanoindentation: *Ji Chen*[1]; Wei Wang[1]; Ke Lu[1]; [1]Institute of Metal Research, Shenyang Natl. Lab. for Matls. Scis., Shenyang 110016 China

Molecular-dynamics simulations have indicated that with the reduction of grain size, the plastic deformation in nc materials gradually changed from a mixture of intergranular and intragranular processes to GB activities dominated mechanisms.[1,2] However, experimental results to verify this argument are still scarce. The critical shear stress (τ_c), which is directly related to the microscopic deformation mechanisms, can be measured by means of nanoindentation.[3] The present work is to investigate the relationship of τ_c with grain sizes of nc Cu ranging from 5 nm to 60 nm. For all nc Cu films, an obvious deviation from the theoretically elastic reponse was detected at approximately the same critical load and depth about 0.0037 mN and 2.7 nm, respectively, which is much smaller compared with that of single crystal Cu. The measured τ_c for the nc Cu was about 7.3 GPa, which is four magnitudes of orders larger than Peierls-Nabarro barrier for the movement of preexsiting lattice dislocations, and very close to the theoretical shear strength for Cu. The high τ_c of the nc-Cu samples may originate from the suppressed lattice dislocation activities by the ultrafine crystallites, and at the same time, the inactive GB accommodation mechanisms at room temperature under the quasi-static indentation. [1]Van Swygenhoven H, Spaczer M, Caro A. Acta Mater 1999;47:3117. [2]Van Swygenhoven H. Science 2002;296:66. [3]Chen J, Wang W, Qian LH, Lu K. Scripta Mater 2003;49:645.

2:55 PM
Simulations of Nanoindentation in Thin Amorphous Metal Films: Yunfeng Shi[1]; *Michael L. Falk*[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

A series of molecular dynamics simulations of nanoindentation in a two-dimensional model of a thin metallic glass film exhibit varying degrees of shear localization below the indenter depending on the means of glass processing. The indenter is circular, and all tests were performed at 9.2% of the glass transition temperature (Tg). The glasses were created from a melt 7.7% above Tg by: (I) quenching gradually over a period of approximately 0.5 μs, (II) quenching quickly over a period of approximately 10 ns, and (III) quenching instantaneously from a well-equilibrated liquid state. Indentation was performed at approximately 0.3 m/s to a depth of 8nm. During nanoindentation shear bands nucleated at an indentation depth of approximately 15 Å in the gradually quenched samples. In the shear band region material that previously exhibited local quasi-crystal-like structural ordering transformed into purely amorphous material under deformation. This structural transition appears to be related to the softening mechanism that results in strain localization in the material. Substantially less

localization occurred in samples produced by higher rates of quenching.

3:10 PM Invited
Evolution of Asperity Contacts: *David J. Srolovitz*[1]; Pil-Ryung Cha[1]; Ji-Hee Kim[1]; Jun Song[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton, NJ 08544 USA

Contacts between opposing surfaces are key aspects of many MEMS switching devices. At low forces, contact is incomplete and is dominated by bumps or asperities on the surface. In this presentation we examine the evolution of asperities under mechanical, capillarity and electromigration driving forces. In the first study, we perform a series of molecular dynamics studies of single asperity contact under constant displacement rate conditions. The dislocation nucleation, migration and annihilation mechanisms are determined in loading and unloading and correlated with the measured force-displacement curve and the change in the contact area. From this, we determine how contact resistance evolves. The force-displacement behavior and the evolution of the contact resistance are shown to be in excellent agreement with cantilever-based experiments. In the second study, we employ phase field methods to examine the evolution of an asperity contact via surface diffusion, driven by capillarity and electromigration. At zero applied field, surface tension quickly rounds the sharp corners at the contact. If the contact is initially narrow, capillarity can lead to the breaking of the contact, but if it is wide, capillarity broadens the contact. With increasing electric field, surface tension still dominates at short times, followed by the electromigration induced broadening of the contact. Based upon the morphology evolution results, we monitor the concommitant change in the contact resistance. Implications for the evolution of contact resistance in devices will be discussed.

3:35 PM Break

3:50 PM
On the Mechanism of Creep-Controlled Electromigration-Induced Drift: *Joris Proost*[1]; Jan D'Haen[2]; [1]University of Louvain, Div. of Matls. & Process Engrg., Place Sainte-Barbe 2, Louvain-la-Neuve B-1348 Belgium; [2]IMEC, Inst. for Matls. Rsch. in Microelect., Wetenschapspark 1, Diepenbeek B-3590 Belgium

Electromigration drift studies on polycrystalline Blech-type test structures have been performed inside a scanning electron microscope. Based on the observed morphological evolution of the hillocking zone at the anodic end, a new analytical expression is presented for the length-dependence of the drift velocity by considering diffusional creep as the plastic flow mode involved in electromigration-induced hillocking. This expression is validated to the observed isothermal drift kinetics at the cathodic end. From the latter, a grain boundary grooving mechanism is proposed to account for the atomistic details of creep-controlled electromigration-induced motion, and the role of grain boundary structure in determining the local direction of groove propagation is illustrated.

4:05 PM
Work Hardening and High Ductility in High-Strength Electroplated Cu with Nanoscale Growth Twins: Y. Morris Wang[1]; *Evan Ma*[1]; Q. H. Lu[2]; M. L. Sui[2]; L. Lu[2]; K. Lu[2]; [1]Johns Hopkins University, Matls. Sci. & Engrg., Baltimore, MD 21218 USA; [2]Chinese Academy of Sciences, Inst. of Metal Rsch., Shenyang China

A high density of growth twins in pure Cu imparts high strength while preserving the room for efficient dislocation storage upon plastic deformation, leading to high strain hardening rates. Tensile test results at 77 K are used to illustrate these effects. In addition to exhibiting high yield strength, the material sustains significant work hardening rates to large strains and at high stress levels, reaching an ultrahigh (GPa) tensile strength combined with impressive uniform elongation and a tensile ductility ~30%. The nano-twinned microstructure is an interesting addition to the repertoire of nanostructured metals that offer a combination of both high strength and ductility.

4:20 PM
Investigation of Maximum Accumulated Plastic Strain Distribution During Equal-Channel Angular Pressing (ECAP), Using 2D and 3D FEM Modeling: *Vladimir S. Zhernakov*[1]; Igor N. Budilov[1]; Salavat T. Kusimov[1]; Igor V. Alexandrov[1]; [1]Ufa State Aviation Technical University, 12, K. Marx St., Ufa 450000 Russia

The current work presents the results of modeling of ECAP of pure copper by means of finite element method (FEM). The comparison of 2D and 3D modeling results was conducted to determine the accumulated plastic strain and its distribution along a billet volume. Stress-strain state of a copper billet after the first pass with constant strain rate was considered. It was established that the type of accumulated plastic deformation distribution in the center of the billet is practically similar in 2D and 3D models. However, 3D modeling allowed to estimate the distribution of deformation along the whole bulk billet and to establish the features of plastic flow heterogeneity during ECAP. The effect of both die design factors and friction on the accumulated plastic deformation was studied during 2D and 3D modeling. The obtained results are used for optimization of die-set parameters of ECAP.

4:35 PM Invited
Dislocation Behavior in Copper and Gold Thin Films: *John Balk*[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Engrg., 177 F. Paul Anderson Tower, Lexington, KY 40506 USA

The geometric constraints imposed by a metal thin film force dislocations to move differently than in a bulk metal. For film thicknesses on the order of one micron, threading dislocations channel between the substrate and film surface as they glide through a film. However, as the thickness of unpassivated films decreases below 400 nm, dislocations glide parallel to the film-substrate interface. This talk will cover various aspects of dislocation behavior in thin film systems, with an emphasis on unpassivated Cu and Au films. In-situ transmission electron microscopy observations will be used to explain the dislocation processes involved and will be related to models for thin film deformation. Parallel glide of dislocations, for instance, is a consequence of constrained diffusional creep. Although this creep mechanism dominates at high temperatures, TEM observations suggest that it persists down to room temperature. These and other aspects of thin film plasticity will be discussed.

5:00 PM
Micron-Scale Plasticity of Gold in the Absence of Strain Gradients: *Julia R. Greer*[1]; William D. Nix[1]; [1]Stanford University, Matls. Sci. & Engrg. Dept., 416 Escondido Mall, Bldg. Peterson 550, Stanford, CA 94305 USA

Classical laws of materials science dictate that mechanical behavior is independent of sample size, however recent experimental results display strong size effect at the microscale. During nanoindentation, non-uniformity of strains is responsible for indentation size effect. ISEs manifest themselves as apparent hardness increase at shallower depths and are explained by strain gradient plasticity theory. While this theory describes hardness variation for depths above ~100nm, it cannot predict discrete strain bursts characteristic of dislocation nucleation at shallower depths. Recently, atomistic behavior during mechanical deformation was studied via MD simulations and indicated that yield strength depended on sample size even without strain gradients. Experimental results testing plasticity of gold at nanoscale without strain gradients are presented. Methodology consists of two distinct fabrication processes of Au cylinders and their uniaxial compression. Test results indicate significant increase in flow stress to several GPa, near theoretical shear strength. Dislocation starvation model is proposed and discussed.

5:15 PM
Surfaces as Membranes: An Approach to Understanding Size Effects: *Danxu Du*[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., D214 E-Quad, Olden St., Princeton, NJ 08540 USA

The concept of a surface as an elastic membrane has been used to describe a wide range of mechanical effects, including interactions between steps on surfaces, growth stresses, etc. Such surface elastic membranes can induce significant stresses within small structures. On the other hand, descriptions of the deformation of small scale structures often require the introduction of strain gradient theories. In the present work, we attempt to connect these two, rather disparate concepts. The connection between the two comes from Stokes theorem that represents integrals over internal variables as lower dimension integrals over surfaces. We show how to rigorously go from a strain gradient theory description to a surface membrane description. The resultant formalism is much easier to implement numerically than the strain gradient formalism. This approach can be used for elasticity and plasticity problems. Applications to available experimental results will be discussed.

5:30 PM
Deformation Mechanisms of Microcrystalline and Nanocrystalline Ni Using Geometrically Different Indenters: *Reza A. Mirshams*[1]; Michael J. Kaufman[1]; [1]University of North Texas, Matls. Sci. & Engrg. Dept., PO Box 310440, Denton, TX 76203 USA

Hardness of electrodeposited nanocrystalline and commercially produced microcrystalline nickel were determined using three geometrically different indenter tips. The results indicate that the dislocation based model describes the deformation mechanism in microcrystalline nickel using the Berkovich tip. However, observations differ with other tips and for the nanocrystalline nickel. Microstructural

examination reveals different deformation mechanisms in the two materials with the different tips.

5:45 PM
Local Strain Development in Near Stoichiometric RuAl Alloys: *Aomin Wu*[1]; Tresa M. Pollock[1]; Qiang Feng[1]; Marc De Graef[2]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15213-3890 USA

RuAl intermetallics display unusual deformation characteristics compared to all other B2 intermetallics. Investigations on the evolution of strains within individual grains have been studied in near stoichiometric RuAl alloys by surface displacement mapping. Strain development is influenced by the amount and distribution of secondary ?Ô-Ru phase. Comparisons between Von Mises effective strain maps and Schmid factors suggest that enhanced straining coincides with a large Schmid factor for {110}<011> slip system, but not for {110}<001>, which is another available slip system in single phase RuAl. Results of dislocation studies using TEM foils prepared by Focus Ion Beam (FIB) on specific grains with representative strain concentrations will be presented. The varying influence of grain orientations, presence of multiple phases and the availability of different dislocation substructure on the overall strain development of RuAl will be evaluated.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Impression and Indentation Testing

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Monday PM Room: 3000
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: M. A. Imam, Naval Research Laboratory, Washington, DC 20375-5000 USA; T. G. Nieh, Livermore National Laboratory, Livermore, CA 94551-9900 USA

2:00 PM Invited
On Some Investigations With the Impression Creep Technique: *D. H. Sastry*[1]; [1]Indian Institute of Science, Metall., Bangalore India

The impression creep technique is a modified indentation creep test wherein the conical or ball indenter is replaced by a cylindrical, flat bottomed punch. It offers several advantages such as simplicity, small quantity of the sample, constant applied load resulting in a constant stress, stable deformation because of the absence of the tertiary creep stage, multiple testing on a single sample, suitability for application to brittle materials etc. This test, pioneered by Prof. Li, is extensively applied in this laboratory to investigate a variety of problems. This novel test technique has been first validated by conducting parallel tensile creep tests on a number of materials. The stress exponent for steady state creep rate, the activation area and the activation energy as determined from the impression creep data are found to concur with those estimated from tensile creep testing. The extent of deformation under the punch as well as the effect of punch diameter have been investigated. The test has been applied to identify the rate controlling mechanisms of creep at high temperatures in a number of metals and alloys. The technique has also been exploited to assess the "single crystal" creep behavior from a polycrystalline sample. Utilizing the impression creep test, the creep behavior of individual zones in stainless steel weldments has been established. The usefulness and the simplicity of the impression creep test have been further demonstrated by its application to the study of superplastic behavior in alloys. This paper presents a cross section of the results obtained in the above investigations. It is concluded that the impression creep test technique is capable of yielding much of the information that can be obtained from tensile creep testing, and furthermore, can provide data which are impossible or extremely difficult to obtain with conventional creep testing.

2:25 PM Invited
Indentation Test Methods: Progress and Accomplishments: *M. Ashraf Imam*[1]; Fahmy M. Haggag[2]; Heshmat A. Aglan[3]; Robert L. Bridges[4]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6320, Washington, DC 20375-5343 USA; [2]Advanced Technology Corporation, 1066 Commerce Park Dr., Oak Ridge, TN 37830-8026 USA; [3]Tuskegee University, Tuskegee, AL USA; [4]Oak Ridge National Laboratory, 4BWXT Y-12, Oak Ridge, TN USA

Traditionally, investigations of elastic and plastic behavior of structural materials have been carried out by uniaxial loading of bars and by multiaxial loading of plates or thin-wall tubes. An alternative method is to use indentation testing techniques and determining the resulting geometric features of the indentation region for specific loadings. This approach offers significant advantages over the conventional method of testing. One method was first developed and demonstrated by J. C. M. Li and coworkers to study high temperature creep. Over the years, different shape and size of the indenters have been used and their applications have been extended to study elastic modulus, yield strength, strain-hardening exponent, strain-rate sensitivity and superplasticity of a wide range of materials including fatigue, stress relaxation, mechanical anisotropy and fracture toughness. In this report emphasis is given to Automated Ball Indentation (ABI) test techniques, invented in 1989, to measure key mechanical properties and its applications to aerospace, transportation, pipelines, shipyards, military, and nuclear.

2:50 PM Invited
Creep Characterization of Microelectronic Solder Balls Based on Miniaturized Impression Creep Testing: *Indranath Dutta*[1]; Deng Pan[1]; Susheel G. Jadhav[2]; [1]Naval Postgraduate School, Mechl. & Astronautl. Engrg., 700 Dyer Rd., Monterey, CA 93943 USA; [2]Intel Corporation, 5000 W. Chandler Blvd., MS CH5-165, Chandler, AZ 85226 USA

The technique of impression creep, first proposed by Professor Li in the 1970s, offers the ability to test specimens of very small material volumes within a relatively short time, and with minimal sample preparation. Here we report on the successful miniaturization and implementation of the impression creep approach to study the creep behavior of 750mm diameter ball grid array (BGA) solder balls attached to a microelectronics packaging substrate. The work was prompted by the strong interest in generating creep databases on lead-free solders based on life-sized joints, in response to the ongoing worldwide transition to lead-free solders in the microelectronics industry. In this paper, experimental creep results on Sn-4.0wt%Ag-0.5wt%Cu BGA balls will be presented, with emphasis on the role of microstructure on creep. Despite the many advantages of impression creep, its widespread utilization has been inhibited by the difficulties in converting impression creep data into a conventional creep equation. Here we report on a detailed finite element (FE) study, undertaken to establish an approach to determine the requisite conversion constants C and k (to translate impression velocity and punch stress to creep rate and uniaxial stress, respectively) as functions of material properties. By defining an appropriate gauge volume based on the crept zone under the impression punch, and by correlating the mean equivalent creep strain with the mean effective stress within this volume, one can arrive at suitable values of C and k, and obtain correlations between key material properties and these constants. Results of these ongoing efforts will be reported.

3:15 PM Invited
Deep Penetration Micro-Indentation Testing of Oriented Polypropylene: *D. M. Shinozaki*[1]; *J. Lo*[1]; [1]University of Western Ontario, Mechl. & Matls. Engrg., London, Ontario N6A 5B9 Canada

Displacement controlled deep penetration microindentation testing has been applied to uniaxially oriented polypropylene. The load-displacement measurements recorded showed differences from those reported in earlier work on isotropic polymers. Examination of the microstructure ahead of the tip face showed that the deformation mechanisms varied with orientation of the tip axis relative to the molecular axis of the material. In the case of penetration parallel to the orientation direction, distinct kink bands were observed ahead of the tip face, localized along the axis of penetration. The end-on indentation of uniaxially oriented polypropylene is shown to be reasonably modeled using a modified version of the Slaughter analysis, originally developed to describe compression of unidirectional fiber com-

posites. As expected, the constraint involved in microindentation testing is found to affect the measured load displacement curve.

3:40 PM Break

3:45 PM
Impression Creep of Sn-3.5 Ag Eutectic Alloy: Fuqian Yang[1]; *Lingling Peng*[1]; [1]University of Kentucky, Chem. & Matls. Engrg., Lexington, KY 40506 USA

The creep behavior of Sn-3.5Ag eutectic alloy was investigated by using impression test technique in the temperatures range 333-453 K and under a punching stress between 3.4 MPa at 453 K and 67.1 MPa at 333 K. The alloy was annealed for 10 hours at 473 K before the impression test. Using a power law between the steady-state impression velocity and the punching stress, the activation energy increases with the punching stress from 44.7 kJ/mole at 6.7 MPa to 79.2 kJ/mole at 46.9 MPa and the stress exponent changes from 1.03 (3.4-13.4 MPa) to 5.9 (20.1-40.2 MPa). However, by using a hyperbolic sine function between the steady-state impression velocity and the punching stress, a single activation energy is found to be 51.0 kJ/mole, which is close to the activation energy for grain boundary diffusion in pure Sn.[1] This suggests that a single mechanism such as grain boundary fluid flow could control the time-dependent plastic flow of Sn-3.5Ag eutectic alloy under the testing conditions. This research is s upported by NSF through a grant DMR-0211706 monitored by Drs. Guebre Tessema and Bruce A. MacDonald. [1]B. Okkerse, Acta. Metall. 2 (1954) 551.

4:10 PM Invited
Nanoindentation Tests for the Investigation of the Mechanical Properties of Bismuth Implanted Layered Amorphous Ge Film: Csilla Kadar[1]; Peter Tasnádi[1]; Gábor Petõ[2]; *András Juhász*[1]; [1]Eötvös University, Dept. for Gen. Physics, 1117 Pázmány Péter st.1/A, Budapest Hungary; [2]Research Institute for Technical Physics and Materials Science, Budapest Hungary

In an earlier paper (Juhász at al. Nucl. Instr. Methods in Phis. Res. B. 148. 1999 355-359.) preliminary results of microhardness measurements taken from bismuth implanted amorphous Ge film were presented. In this paper an account is given about the resumption of that work. The mechanical investigations were carried out by nanoindentation tester which provides more sensitive method than that used previously. The effect of the annealing applied after the implantation was also investigated. The hardness, the elastic modulus and the brittleness of the implanted samples were found significantly lower than those of the unimplanted material. Due to annealing the mechanical properties of the implanted samples were changed toward the unimplanted state. Mechanical measurements were completed by revealing the microstructure of the specimens by electron microscopy which showed the bubbled structure of the implanted zones. The results obtained for the implanted and annealed state are interpreted by a composite model concerning a layered structure consisting of weak (bubbled) and strong bands.

4:35 PM Invited
Relationships Between the Work of Indentation, Hardness, Elastic Modulus, and the Friction and Wear Behavior of Materials: Wangyang Ni[1]; Che-Min Cheng[2]; *Yang-Tse Cheng*[1]; [1]General Motors Research and Development Center, Matls. & Processes Lab., MS 480-106-224, 30500 Mound Rd., Warren, MI 48090 USA; [2]Institute of Mechanics, State Key Lab. for Nonlinear Mech., Beijing 100080 China

The work of indentation is examined using dimensional analysis and finite element calculations for conical and spherical indentation in homogeneous solids and for conical indentation in thin films on substrates. For conical indentation in elastic-plastic solids with work-hardening, we established a relationship between the ratio of hardness to elastic modulus and the ratio of irreversible work to total work of indentation.[1,2] Using this relationship, the ratio of hardness to elastic modulus can be obtained directly from measuring the work of indentation for homogeneous solids. For spherical indentation in elastic-plastic solids with work hardening, we found simple relationships between hardness, reduced modulus, indentation depth, indenter radius, and work of indentation.[3] We have also uncovered similar relationships for conical indentation in hard films on soft substrates. These relationships, together with the relationship between initial unloading stiffness and reduced modulus, provide an energy-based method for determining contact area, reduced modulus, and hardness of materials from instrumented indentation measurements using conical, pyramidal, and spherical indenters. Finally, we show that the ratio of irreversible work to total work plays an important role in determining the friction coefficient and wear behavior of materials and suggest a strategy for reducing friction and enhancing wear resistance of materials. [1]Y.-T. Cheng and C.-M. Cheng, Appl. Phys. Lett. 73, 614 (1998). [2]Y.-T. Cheng, Z. Li, and C.-M. Cheng, Phil. Mag A 82, 1821 (2002). [3]W. Ni, Y.-T. Cheng, C.-M. Cheng, and D. S. Grummon, J. Mat. Res. 19, 149 (2004).

5:00 PM Invited
Elastic Deformation Due to Oscillating Indentations: B. B. Rath[2]; *H. Y. Yu*[1]; [1]US Army Asian Research Office, Tokyo Japan; [2]Naval Research Laboratory, Washington, DC USA

Recent developments in instrumented indentation include the use of dynamic oscillation for improved sensitivity and testing capabilities. Instrumented indentation, also known as depth-sensing indentation or nano-indentation, is increasingly being used to probe the mechanical response of metals, ceramics, polymers and biological materials. Dynamic contact problems have also received considerable attention recently in the mathematical literature. This work is to study the elastic field in an isotropic half space due to dynamic frictionless indentation. The rigid indenter tip is assumed to be axisymmetric with a smooth convex profile such that the contact area is a circular region. A point load method will be used to obtain the analytical solutions for the time-harmonic normal indentation.

5:25 PM Invited
Plasticity of Single Crystals Under a Sliding Indenter: *Bhaskar S. Majumdar*[1]; Bing Ye[1]; [1]New Mexico Tech, Dept. of Matls. & Metallurgl. Engrg., Socorro, NM 87801 USA

While the literature on the friction and wear of metallic materials is voluminous, there are still considerable questions regarding the deformation and damage mechanisms. The near surface behavior is dominated by plasticity and microstructural evolution, and these in turn dictate frictional forces and further wear. Such near surface phenomenon have taken on added importance in modern micro electro mechanical systems that employ textured metallic films. In this work, we have focused attention on single crystal substrates, with a view to understanding fundamental dislocation processes under a sliding indenter. The results indicate a very strong influence of the orientation of loading on the deformation response, and this appears to be manifested mainly through the hardening response. The geometry of multiple slip and the morphology of deformation will be illustrated and compared with available data in the literature on highly textured fcc alloys. The effects of friction coefficients are analyzed, and we comment on how the morphology of slip might in turn influence frictional coefficients. The first author had the privilege of learning plasticity and dislocation theory from Professor Li, and provides his respects on the occasion of his 80th Birthday.

5:50 PM
Indentation Studies of Self Adhesion of Poly(dimethylsiloxane): *Xinzhong Zhang*[1]; Fuqian Yang[1]; J. C.M. Li[1]; [1]University of Rochester, Mechl. Engrg., Rochester, NY 14627 USA

The self-adhesion of poly(dimethylsiloxane) (PDMS) was studied using a cylindrical indenter. The indenter had a flat end coated with a thin layer of PDMS. The flat end was contacting the planar surface of a PDMS block. Unlike spherical indenters, there was no hysteresis in loading and unloading in the elastic range since the contact area did not vary. The pull-off force is a measure of the energy of self adhesion at the instant of separation. The self-adhesion energy between PDMS surfaces was found to increase with the square root of contact time indicating molecular diffusion as the dominant mechanism for self-adhesion. The activation energy for self diffusion of PDMS was obtained from the temperature effect and was compared with that of viscous flow of PDMS measured by an impression creep test. Work supported by NSF through DMR-9623808 monitored by Bruce MacDonald.

Microstructural Processes in Irradiated Materials: Modelling Defect Evolution and Oxide Dispersion Strengthened Alloys

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Monday PM Room: 3011
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Christophe Domain, EDF R&D, Dept. MMC, Les Renardieres, Moret sur Loing F-77250 France; Brian Wirth, University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

2:00 PM Invited
Temperature Accelerated Dynamics Study of Radiation Damage Annealing in MgO: *Arthur F. Voter*[1]; [1]Los Alamos National Laboratory, Theoretl. Div., T-12, MS B268, Los Alamos, NM 87545 USA

We present results of a collaborative[1] atomistic study of radiation damage annealing in MgO following cascades in the 1 keV energy range. Using a Buckingham potential, knock-on events were simulated with molecular dynamics. Longer times were probed using temperature accelerated dynamics, enhanced by minimum escape barriers from dimer searches (dimer-TAD). Configurations with a small number of defects, representative of the post-cascade results, were evolved using dimer-TAD for times up to seconds and beyond. Results were complimented with molecular statics and some verified with density functional theory. An interesting picture emerges for the diffusion and coalescence of defects produced in this energy range, involving time scales from ns to years. Also, diffusion of interstitial clusters shows a surprising nonmonotonicity. [1]B.P. Uberuaga, R. Smith, A.R. Cleave, F. Montalenti, G. Henkelman, R.W. Grimes, A.F. Voter, and K.E. Sickafus, Phys. Rev. Lett. 92, 115505 (2004).

2:40 PM Invited
Kinetic Monte Carlo Calculations to Study Defect Evolution in Irradiated Metals: *Maria J. Caturla*[1]; [1]Universidad de Alicante, Fisica Aplicada, Facultad de Ciencias, Fase II, Alicante E-03690 Spain

Currently kinetic Monte Carlo (kMC) models are widely used to study the accumulation and evolution of damage during irradiation. In this talk we will give a brief overview on the calculations done for pure metals, both f.c.c. and b.c.c. The influence of different parameters in the final conclusions of the simulations will be discussed. In particular we will describe the use of kMC models to understand the relevant parameters that control microstructure evolution during irradiation. Emphasis will be made in those parameters where little or no information exists form either experiments or simulations, such as the interaction of defects with impurities. One important part of the presentation will be devoted to He effects in b.c.c. Fe comparing it to f.c.c. metals and to experiments both on He desorption and neutron irradiation.

3:20 PM
Displacement Cascade Influence on Void and Precipitate Nucleation: *Kenneth Calvin Russell*[1]; Byungkon Kim[1]; [1]Massachusetts Institute of Technology, Dept. Matl. Sci. & Engrg. & Nucl. Engrg., Rm. 13-5050, 77 Mass. Ave., Cambridge, MA 02139-4307 USA

Displacement cascades are the primary damage event in most irradiation of metals. The cascades are typically vacancy-rich regions surrounded by a halo of self-interstitial atoms. At reactor operating temperatures the vacancy is mobile and the cascades dissipate to create a more or less uniform sea of vacancies and self-interstitial atoms. This excess of point defects gives greatly enhanced diffusion rates and in addition provides a driving force for nucleation, in particular of voids, dislocation loops, and precipitate particles. The cascades may also have direct effects on nucleation by either creating on destroying embryos of nucleating aggregates. A partial differential equation for nucleation is derived which includes such effects. The equation is normalized to give dimensionless coefficients of the derivatives, which in turn predict experimental conditions when the various cascade effects will and will not be significant. More complete solutions of the differential equation will also be presented.

3:40 PM Break

4:10 PM Invited
Development of Advanced Nanostructured Ferritic Alloys for Nuclear Fission and Fusion Applications: *David T. Hoelzer*[1]; Matt J. Allinger[2]; Michael K. Miller[1]; G. R. Odette[2]; Jim Bentley[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [2]University of California, Santa Barbara, CA 93106 USA

Mechanically alloyed (MA) oxide-dispersion strengthened 12-14Cr ferritic alloys, such as the Japanese 12YWT and INCO MA957, contain a high number density of <5nm sized particles enriched in Y, Ti, and O. The superior high-temperature creep properties of these so-called nanostructured ferritic alloys (NFAs) are attributed to this nano-particle dispersion. In addition, since their unique microstructure may also provide enhanced resistance to radiation damage and high levels of transmutation product helium, both the fusion and advanced fission reactor materials programs have considerable interest in NFAs. This paper highlights our current experimental and theoretical understanding of the nano-particle structure and formation and strengthening mechanisms, including the effects of processing variables, as well as their high temperature stability. Potential issues such as costs, joining methods, their stability under irradiation and low fracture toughness will also be discussed briefly.

4:50 PM
Microstructural Analysis on Tensile-Deformation Behavior of Oxide-Dispersion Strengthened Ferritic Steels: *Ryuta Kasada*[1]; Naoki Toda[2]; Akihiko Kimura[1]; [1]Kyoto University, Inst. of Advd. Energy, Gokasho, Uji, Kyoto 611-0011 Japan; [2]Kyoto University, Grad. Sch. of Energy Sci., Gokasho, Uji, Kyoto 611-0011 Japan

For high burn-up operation of light water reactor and super critical water reactor, our research group has been developing a series of oxide dispersion strengthening (ODS) ferritic steels as fuel cladding because of the excellent high-temperature strength and resistance to irradiation hardening.[1] These excellent performances are considered to be due to the ultra-fine nano-scale yttria particles dispersed in the matrix. Although the strengthening mechanism due to the yttria dispersoids has been considered to be the Orowan bypassing mechanism, no obvious evidence was shown for the ODS steels containing such ultra-fine dispesoids. In this study, deformed microstructure of the ODS steels was investigated and discussed. The materials used were ODS ferritic steels which contain various chromium contents of 13~22wt%.[1] TEM bservations were carried out with post- and in-situ straining techniques. [1]A. Kimura, et al., "R&D of Advanced Ferritic Steel for High Burn-up Fuel Cladding", ICAPP'04, in press.

5:10 PM
Tensile Properties and Fracture Mode of a Wrought ODS Molybdenum Sheet Following Fast Neutron Irradiation at Temperatures Ranging from 300C to 1000C: *Brian V. Cockeram*[1]; Richard W. Smith[1]; Lance L. Snead[2]; [1]Bechtel Bettis Laboratory, PO Box 79, ZAP 05R/MT, W. Mifflin, PA 15122-0079 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA

A commercially available Oxide Dispersion Strengthened (ODS) Molybdenum alloy that has been rolled into 0.76 mm thick sheet possesses a fine dispersion of La-oxide particles, fine grain size (about 1.2 um), and a high recrystallization temperature (1800C). These microstructural features result in excellent creep resistance and strength at high temperatures, and high levels of tensile elongation at low temperatures. Since wrought processing results in alignment of oxide particles in the working direction, the mechanical properties of ODS molybdenum are anisotropic, but are generally an improvement over many molybdenum-base alloys. The microstructural features of ODS moylbdenum that improve the non-irradiated mechanical properties may also have an influence on the irradiated properties. The change in the mechanical properties of ODS molybdenum after irradiation are not known, and are investigated herein following irradiation in the High Flux Isotope Reactor (HFIR) at 300C, 600C, and 870C to 1000C to neutron fluences between 10.5 to 200 X 10^20 n/cm^2 (E > 0.1 MeV). Irradiation of ODS molybdenum at 600C resulted in a large increase in strength (137% to 56%), but the Ductile to Brittle Transition Temperature (DBTT) was room-temperature. The room-temperature DBTT for ODS is below the range of DBTT values (100C to 710C) reported in literature for molybdenum alloys irradiated at 600C, which demonstrates that ODS molybdenum has an improved level of resistance to radiation embrittlement. The thin sheet toughening mechanism, where ODS molybdenum splits along grain boundaries during

fracture to leave thin ligaments that are stretched under plane stress conditions to large amounts of elongation, is shown to produce the ductile behavior at room-temperature. However, irradiation of ODS molybdenum at 300C resulted in a large amount of strengthening (150% to 130%) and the DBTT (800C) that is the same as observed for unalloyed molybdenum. Irradiation of ODS at 870C to 1000C resulted in small increases in yield strength (29% to 10%) with a post-irradiated DBTT (-100C) that is lower than values reported in literature for molybdenum alloys. Fractography and microstructure examinations are used to show that a fine grain size and fine oxide particle size provide the observed improvements in the post-irradiated tensile properties of ODS molybdenum.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Analysis of Interdiffusion Microstructures: Session II

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Monday PM Room: 3007
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: John H. Perepezko, University of Wisconsin, Matls. Sci. & Engrg., Madison, WI 53706 USA; Harold D. Brody, University of Connecticut, Matls. Sci. & Engrg., Storrs, CT 06169-3136 USA

2:00 PM Invited
A Model for the Lateral Deformation of Diffusion Couples: *W. J. Boettinger*[1]; G. B. McFadden[1]; S. R. Coriell[1]; J. A. Warren[1]; R. F. Sekerka[2]; [1]NIST, Gaithersburg, MD 20899 USA; [2]Carnegie-Mellon University, Pittsburgh, PA 15213 USA

A model is proposed to describe the shape change of a binary diffusion couple when the intrinsic (lattice) diffusivities of the two substitutional species differ. The classical uniaxial Kirkendall shift is obtained only if the displacements are artificially constrained to be in the diffusion direction. In the usual experimental case when the lateral surfaces of the diffusion couple are traction-free, a more general displacement field is obtained that accounts for the lateral shape change data of Voight and Ruth. In the interdiffusion zone, near the free surfaces of the diffusion couple, the sample deforms inward and outward respectively on opposite sides of the couple. The model uses an isotropic stress-free strain rate proportional to the vacancy creation/annihilation rate and an elastic/plastic constitutive model. In addition to numerical simulations with the model, the limiting behaviors for couples with lateral dimensions large and small compared to the diffusion distance are reported.

2:30 PM Invited
Teaching the Square-Root Diffusivity Method: *Martin Eden Glicksman*[1]; Afina Lupulescu[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., Troy, NY 12180 USA

The interdiffusion of atomic species in multicomponent alloys is a subject of considerable complexity, with broad applications throughout materials engineering. Specifically, multicomponent diffusion concepts provide novel design strategies to improve the performance of alloys and coatings that must resist degradation and aging at elevated temperatures. Engineers who conceive and design structures for high-temperature use are challenged by these problems. Consequently, during their undergraduate or graduate programs, materials engineering students should be exposed to fundamental aspects of multicomponent diffusion. Professor John Morral demonstrated the utility of a related transport matrix, the "square-root diffusivity matrix." His integrative methodology to understand many aspects of multicomponent diffusion, now taught at Rensselaer, employs his computer program, Profiler. Through this approach, students are now easily acquainted with one-dimensional, single-phase, multicomponent diffusion problems as a tractable class of numerical solutions, including the formation of zero-flux planes, up-hill diffusion, S-shaped diffusion paths, etc.

3:00 PM
Implementing the Square-Root Diffusivity Method: *Afina Lupulescu*[1]; Martin Eden Glicksman[1]; Srividya Kailasam[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., Troy, NY 12180 USA; [2]National Exposure Research Laboratory, Las Vegas, NV 89119 USA

Applying the theory of multicomponent diffusion and solving Fick's second law is a challenging task. Fortunately, most of the tedious mathematical labor can be eliminated by employing programs that efficiently handle the required linear algebra steps. In 1986, Morral and Thompson developed a systematic methodology for constructing one-dimensional single-phase multicomponent diffusion solutions, referred to as the "square-root diffusivity method." Implementing the Morral-Thompson method for multicomponent alloys is now made convenient by using the public domain computer program, Profiler, developed by Morral. Quantitative predictions of multicomponent diffusion effects, particularly the penetration curves and diffusion paths, were checked against experiments using several programs. Generally, good results were obtained. To study multicomponent diffusion behavior for different alloys and investigate the kinetics of zero-flux planes (ZFP's), the authors developed a new MatLab© code that closely follows a combination of Morral and Thompson's approach as well as that by Glicksman and Lupulescu.

3:25 PM Break

3:40 PM
Test of Darken's Mobility Assumption in the Cu-Ni-Zn System: *Robert Thomas DeHoff*[1]; Nagraj S. Kulkarni[2]; [1]University of Florida, Matls. Sci. & Engrg., Gainesville, FL 32611 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4515, #111, Oak Ridge, TN 37831-6063 USA

Darken's assumption that the mobility of a component in a homogenous system is the same as that in a system subjected to a composition gradient was central in establishing relations between the tracer and intrinsic or interdiffusion coefficients. These relations have been examined primarily in binary systems with mixed results. In this presentation, the Darken relations are examined for the ternary Cu-Ni-Zn system for which adequate tracer diffusion data and CALPHAD-based thermodynamic models are available. With the aid of an intrinsic diffusion simulation developed by the authors, the composition paths and Kirkendall shifts predicted from the Darken and Manning theories are compared with experimental data. Important issues concerning the development of multicomponent diffusion databases are addressed.

4:05 PM
Flux-Independent Theory of Nonlinear Diffusion for Vegard's Law Solutions: *J. S. Kirkaldy*[1]; [1]McMaster University, Brockhouse Inst. for Matls. Rsch., 1280 Main St. W., Hamilton, Ontario L8S 4M1 Canada

While the phenomenological Boltzmann-Matano (B-M) analysis argues that a concentration-dependent diffusion coefficient D(X) relates to infinite diffusion couple experiments by the solute-conserving, flux-formulated non-linear parabolic equation in terms of mole fraction X, $-(1/2)(dX/d\lambda)=d/(d\lambda)\ D(X)\ (dX/d\lambda)$, this contribution proves that for stable substitutional Vegard's Law solutions, automatically subsuming a mechanistic vacancy-dominated contribution to a Kirkendall creep process at local equilibrium, both a crystalline volume and a molar mass conserving condition follow. Concomitantly, the usual Onsager generalization from irreversible thermodynamics leading to this equations has to be replaced by a Helmholz free energy based on a Ginzburg-Laudau variational equation in which a solute-conserving flux does not and cannot enter under threat of over-determination. Consequently, conservation is relegated to the processing and symmetry of the free energy density whereby D(X) proves to reside outside the differentials on the right. As further validation of the flux-free construction it is demonstrated that in contrast to the above B-M formulation the variable D's extracted from diffusion couples is approximate to those generated by a set of incremental couples where consistently D also resides outside the differentials. The simplification of the B-M analysis and the methodology for a correctly framed coefficient evaluation are then established. Since in the ternary generalization solute fluxes are likewise not defined, the concept of "zero flux plane" must be re-examined. It is remarkable that in this representation the commonly observed single clustering maxim act as Kirkendall drift markers. Furthermore, in this Vegard's Law formulation volume and mole numbers remain conserved as in the binary case, their neutral

planes are not coincident. A quantitative verification of the flux-free disposition is given for the representative cases.

Neutron Diffraction Characterization of Mechanical Behavior: Deformation I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Monday PM Room: 3004
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Camden R. Hubbard, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

2:00 PM Invited
Using Polycrystal Models and Neutron Diffraction to Advance Our Understanding of Materials: *Carlos N. Tomé*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-8, MS G755, Los Alamos, NM 87545 USA

Twinning is an important deformation mode in HCP materials, which strongly influences texture and hardening evolution during plastic forming. As a consequence, twinning contribution to texture and hardening has to be accounted for in constitutive descriptions of HCP aggregates. We use a Composite-Grain Polycrystal Model for describing twinning-related constitutive response in a general manner. This model accounts for directional barriers to dislocation motion posed by the twin lamellae in the grains, and also for the evolution of twin fraction with deformation in the grains. As an application, we simulate in-plane and through-thickness compression in rolled Zr, Be and Mg. Development of polycrystal models hinges strongly on the experimental capability for correlating modeling parameters with measurements. The neutron diffractometers SMARTS and HIPPO (LANSCE-LANL) have in situ testing capabilities, and provide us with an important tool for following evolution of texture, hardening and internal stresses as a function of deformation. We will discuss the results of our modeling efforts, related experimental results, and also how the experimental information can be used to infer information about microscopic deformation mechanisms.

2:20 PM
Coupling of Twinning, Texture, and Hardening During Deformation of Zirconium: *George C. Kaschner*[1]; Carlos N. Tomé[1]; Sven C. Vogel[2]; Don W. Brown[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, LANSCE-12, MS H805, Los Alamos, NM 87545 USA

We present experimental observations of high purity zirconium loaded to small strains at quasi-static strain rates in an equilibrium liquid nitrogen bath then reloaded at room temperature. Neutron texture measurements were performed on each sample at three stages: as-annealed, after cryo-loading, and after room temperature deformation. Material loaded first in an in-plane orientation was found to harden compared to a reference test performed at room temperature only. Zirconium loaded first in the through-thickness orientation was found to soften when reloaded at room temperature compared to a room temperature reference test of the same orientation.

2:40 PM
Neutron Diffraction Study of Deformation Mechanisms of Magnesium Alloy AZ31: *Sean R. Agnew*[1]; D. W. Brown[2]; Ozgur Duygulu[1]; C. N. Tomé[2]; [1]University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; [2]Los Alamos National Laboratory, MTS-8, Los Alamos, NM USA

In-situ neutron diffraction experiments are performed on magnesium alloy AZ31 with a number of distinct initial textures resulting from prior hot-rolling, extrusion and equal channel angular extrusion. Diffraction is used to probe the development of internal stresses during loading and unloading. The anisotropy and asymmetry resulting from the individual textures is probed by testing in tension and compression along multiple sample directions. Not only are there striking distinctions in the mechanical behaviors of the variously textured samples, the internal stress development is quite distinct between the samples. The differences in the internal stress (and texture) development can be used to determine the active deformation mechanisms, in particular for deformation twinning. The determination of active mechanisms may be done in a more quantitative fashion for cases of slip dominated deformation through computer simulation of the internal stress development using the elastoplastic self-consistent (EPSC) code. Recently, a single set of critical resolved shear stress and hardening parameters have been used to simulate the observed mechanical behavior and internal stress developments of all the variously textured samples. Finally, samples are tested in-situ at elevated temperatures in order to determine if the internal stress development (i.e., deformation mechanisms) change with temperature.

3:00 PM
Three Dimensional Measurement of Type 2 Strains in Zircaloy-2: *Richard Alan Holt*[1]; Ronald Rogge[2]; Feng Xu[1]; Brian W. Leitch[3]; [1]Queen's University, Mechl. & Matls. Engrg., Kingston, Ontario K7L 3N6 Canada; [2]National Reserach Council of Canada, Neutron Prog. for Matls. Sci., Chalk River, Ontario K0J 1J0 Canada; [3]Atomic Energy of Canada Ltd., Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada

The development of Type 2 residual strains has been measured in three dimensions in tensile specimens machined in the normal, transverse and rolling directions of hot-rolled Zircaloy-2 slab. The slab was about 25 cm wide by 3.8 cm thick by 200cm long. The texture of the slab and its spacial distribution were measured on the E3 spectrometer at the NRU reactor and the type two strains were measured in-situ during testing at room temperature in the L3 spectrometer using several crystallographic reflections. The (0002) plane normals were oriented predominently in the normal direction of the slab and was fairly uniform over about 2 cm at mid-thickness over most of the width of the slab. The tensile specimens were centered at mid-wall of the slab. Specimens tested in the transverse and rolling directions deformed by slip, and specimens tested in the normal direction eventually deformed by twinning. Strain measurements were made with and without load in the elastic regime and at intervals of 0.2-2% plastic strain after yield had occurred. The results are compared with the predictions of published self-consistant polycrystalline models.

3:20 PM
In-Situ Neutron Study of Deformation Mechanisms in Zirconium Alloys at Cryogenic and Room Temperatures: *T. A. Sisneros*[1]; M. A.M. Bourke[1]; D. W. Brown[1]; T. M. Holden[2]; S. C. Vogel[1]; T. R. Woodruff[3]; [1]Los Alamos National Laboratory, Los Alamos, NM USA; [2]Northern Stress Technologies, Deep River Canada; [3]University of Central Florida, Orlando, FL 32816 USA

In hexagonal metals, multiple slip and twinning systems compete as active deformation mechanisms. In particular, the formation of twins, plays an important role in the evolution of hardening by creating barriers to the propagation of dislocations for slip and other twin systems. The relative propensity of each deformation mechanism depends on parameters such as temperature, strain rate, purity, grain size, and texture. We report the effect of temperature variation (300K, 250K, and 216K) on twinning during a quasi-static uniaxial compression test in a zirconium alloy (Zr-1. 4Sn-0.1Cr-0Fe-0.1O-0.05Ni wt. %). The relative activities of the deformation mechanisms are determined by complementing in-situ neutron diffraction measurements performed at Los Alamos National Laboratory with polycrystalline plasticity modeling.

3:40 PM Break

4:00 PM Invited
Recents Developments in Diffraction Techniques for the Biomaterials: *Lodini Alain*[1]; [1]Universite de Reims, Moulin de la Housse, 51100 Reims France

The preferred orientation of hydoxyapatite crystallites at the interface bone-implant in sheep tibia bones has been measured with the neutron 2 axis diffractometer at the Institut Max Von Laue Paul langevin, extracted 60 days after implantation. The implant has two faces, one coated and one non-coated with plasma-sprayed HAp (80 microns). We probed the samples with a spatial resolution of 0.5 mm started from the interface in order to inspect the reorganisation of the HAp crystallite distribution after implantation.

4:20 PM Invited
Neutron Texture Analyses in Sauropod Bones: *A. Pyzalla*[1]; R. Ferreyro[1]; M. Stempniewicz[1]; A. Gunther[2]; H.-G. Brokmeier[2]; [1]TU Wien, Inst. of Matl. Sci. & Tech., Wien Austria; [2]GKSS, Geesthacht Germany

Sauropod dinosaurs are the largest animals that ever walked on earth. Their only remnants are fossilized bones. These fossilized bones due to their original complex hierarchical structure and due to diagenesis during burial have a complex multiphase microstructure. Within this microstructure part of the original features of the bone are preserved. The aim of our investigations is a comparison between the fossilized sauropod bones and contemporary animal bone. By this comparison we expect to find out how the extraordinary weight of the sauropods (up to 100 tons) was accommodated by the skeleton and specifically if the sauropod bone microstructure shows adaptations to the huge loads the bones had to carry. On of the strengthening mechanisms of bone is the development of a preferred orientation of the hydroxylapatite crystals. In order to verify the hypotheses that sauropod bones adapted to high loads by developing texture, neutron pole figure analyses were carried out at the diffractometer TEX-2 at GKSS, Geesthacht for femur and humerus of different sauropod species and animals of different ontogenetical age. The results of the analyses revealed that the fossilized bones still contain a fibre texture whose intensity varies across the bone diameter and differs for animals of different ontogenetical age. Even for animals of the same species and similar ontogenetical age the pole figures, in accordance with bone histology, imply different growth strategies for the long bones.

4:40 PM
Measurements and Predictions of Strain Pole Figures for Uniaxially Compressed Stainless Steel: *Cecilia Larsson*[1]; Bjorn Clausen[2]; Tom M. Holden[2]; Mark A.M. Bourke[2]; [1]Linkoping University, Dept. of Mechl. Engrg., Linkoping Sweden; [2]Los Alamos National Laboratory, Los Alamos, NM USA

Strain pole figures representative of residual intergranular strains were determined from an -2.98% uniaxially compressed austenitic stainless steel sample. The measurements were made using neutron diffraction on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory, using an Euler cradle to obtain spectra over a range of sample orientations. The time-of-flight technique used in SMARTS facilitated the simultaneous recording of eight hkl lattice plane reflections in individual spectra. The measurements were compared with predictions from an elasto-plastic self-consistent model and found to be in good agreement. The average of the strain differences between measurement and model for the angles and reflections considered was 17 x 10^{-6}, well within the error of 150 x 10^{-6} attributed to the measurement technique. The model was used to assess the sensitivity of the strain distribution in the deformed sample to the initial texture. While weak textures (such as the sample's 1.6 x random) did not show significant effects, stronger textures (e.g., 6 x random) reduced the inherent rotational symmetry of the strain field with strain variations of up to 900 x 10^{-6}.

5:00 PM
Crystallographic Texture and High Temperature Deformation Mechanisms in Zr-2.5Nb: *Richard Alan Holt*[1]; Pingshun Zhao[1]; Yue Li[1]; [1]Queen's University, Mechl. & Matls. Engrg., Kingston, Ontario K7L 3N6 Canada

The bulk crystallographic texture and through its wall variations in Zr-2.5Nb tubes extruded in the temperature range 650-975°C have been measured by neutron diffraction. The micro-texture and microstructure have also been characterized by scanning electron microscopy with electron backscattering diffraction and by transmission electron microscopy with selected area diffraction. At low extrusion temperature (650°C), where the majority phase is the hcp alpha-phase, the texture development is dominated by slip within the hcp phase, and in the courser grains the (0002) texture is predominantly radial - more so on the inside third of the tube wall where the strain ratio, Q, (the ratio of the radial to tangential strain during extrusion) is highest. At intermediate extrusion temperature (815°C), the texture development appears to be dominated by the interaction between the soft bcc beta-phase and the harder hcp phase which are present in roughly equal proportions. Boundary sliding mechanisms may be dominant. Here the (0002) texture is predominantly transverse, more so on the inside third of the tube wall. This clearly illustrates the change in deformation mechanisms from the lower temperature. Tubes made from billets pre-heated to 975°C, exhibited a large front to back variation, indicating substantial chilling during extrusion. The fronts of these tubes (the end exiting the extrusion press first) exhibited a beta-to-alpha transformation texture, with a dominant component of (0002) along the tube axis. The back ends exhibited a texture similar to that produced at 815°C because of chilling of the billet in the extrusion chamber. In the former case, the through wall variations corresponded to the temperature profile through the wall during extrusion, with the outer third of the wall (chilled by the container) exhibiting less of the transformation texture. A variety of microstructures are observed by SEM and TEM which correlate with the starting microstructures of the billets and with the observed textures.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Thin Film Stability and Reactions, Electro- and Thermomigration Phenomena

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Monday PM — Room: 3016
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Engrg., Hsin-chu 300 Taiwan; Srinivas Chada, Jabil Circuit Inc., FAR Lab/Advd. Mfg. Tech., St. Petersburg, FL 33716 USA

2:00 PM Invited
Thermal Stability Enhancement in Sputtered Cu Films Containing Insoluble Tungsten Carbides: *Yung-Yen Hsieh*[1]; Jinn Chu[1]; C. H. Lin[1]; T. Mahalingam[2]; [1]National Taiwan Ocean University, Inst. of Matls. Engrg., Keelung Taiwan; [2]Alagappa University, Dept. of Physics India

In this paper, we report enhanced thermal stabilities of nanostructured Cu films containing insoluble tungsten carbides prepared by sputtering. Tungsten carbides in the form of W_2C are present in the supersaturated solid solution of Cu, as confirmed by X-ray photoelectron spectroscopy and X-ray diffraction. Focused ion beam analysis reveals that the films are thermally stable during annealing when they are in contact with Si and the copper silicide is not formed up to an annealing temperature 530°C. The films exhibit good thermal stability at high temperatures and it can be rationalized as a consequence of a refined grain structure together with the strengthening effect of W_2C.

2:30 PM
Thermal Stability Improvement Study of Sputtered Cu Films with Insoluble Substances: *Chon-Hsin Lin*[1]; Jinn Chu[1]; Yung-Yen Hsieh[1]; T. Mahalingam[2]; *Chun-Hui Lin*[1]; S.F. Wang[3]; [1]National Taiwan Ocean University, Inst. of Matl. Engrg., Keelung Taiwan; [2]Alagappa University, Physics, India; [3]National Taipei University of Technology, Depts. of Matls. & Minerals Resources Engrg.

Copper is an attractive material for metallization in microelectronics, because of its low resistivity and high reliability against electromigration compared with Al and its alloys. However, Cu diffuses readily into Si and SiO_2, resulting in the formation of copper silicide compounds at low temperatures. Our prior studies confirmed the better thermal stability of Cu-W and Cu-Mo and showed that a low film resistivity could be achieved by adding small amounts of insoluble elements with the fine-grained film microstructure after annealing. In present study, dilute amounts of insoluble substances were introduced in Cu film during sputtering. A newly developed Cu metallization process is proposed with a self-forming barrier between Cu film and Si by vacuum thermal annealing. The process can improve the thermal stability of Cu and suppress Cu diffusion into the Si substrates, retaining a low resistivity of Cu. The thermal stability of Cu film produced by this process was characterized by various techniques such as X-ray diffraction, focus ion beam (FIB), secondary ion mass spectroscopy (SIMS), transmission electron microscope (TEM), X-ray photoelec-

tron spectroscopy (XPS), electrical resistivity and current-voltage (I-V) curve measurements, and the results are discussed.

2:50 PM
Crystallization and Failure Behaviors of Ta-TM (TM=Fe, Co) Nanostructured/Amorphous Diffusion Barriers for Copper Metallization: *Jau Shiung Fang*[1]; C. P. Hsu[2]; G. S. Chen[3]; [1]National Huwei University of Science and Technology, Dept. of Matls. Sci. & Engrg., 64 Wunhua Rd., Huiwei, Yunlin 632 Taiwan; [2]National Huwei University of Science and Technology, Grad. Inst. of Electro-Optical & Matl. Sci., 64 Wunhua Rd., Huiwei, Yunlin 632 Taiwan; [3]Feng Chia University, Dept. of Matl. Sci., Taichung 410 Taiwan

This work examines the thin-film properties and diffusion barrier behaviors of sputtered Ta-TM (TM=Fe, Co) films, aiming at depositing a highly crystallization-resistant and conductive diffusion barriers for Cu metallization. Four-point probe measurements, X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to examine the barrier properties. Structural examination indicted that intermetallic-compound-free amorphous Ta-TM films were obtained by magnetron sputtering, and thus giving a resistivity about 146.82 ìÙ-cm .and 247.01ìÙ-cm for Ta50Fe50 and Ta50Co50 films, respectively. The Si/Ta50Fe50/Cu and Si/Ta50Co50/Cu stacked samples were observed to fail completely at temperature above 600°C and 650°C because of the formation of Cu3Si protrusions between silicon and Ta-TM barrier layer. Highly thermal stabilized amorphous Ta-TM thin film can thus be potentially adopted as a diffusion barrier for Cu metallization.

3:10 PM
Interfacial Reactions in the Al/Ta Couples: *Chin-Yi Chou*[2]; Sinn-Wen Chen[1]; [1]National Tsing-Hua University, Chem. Engrg. Dept., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan; [2]National Tsing-Hua University, Matls. Sci. & Engrg. Dept., Hsin-Chu 300 Taiwan

Thermal inkjet technology is in widespread use for color printers, and the market is still growing. Tantalum has low contact resistance, good adhesion and chemical resistance and is used as the diffusion barrier between the gold and aluminum layers. Understanding of the Al/Ta contact stability is fundamentally important for the inkjet technology. This study investigated the Al/Ta contact stability using the reaction couple technique. A Ta foil and an Al chunk were polished, placed together in a graphite mold, and sealed in a quartz tube under vacuum. The quartz capsule was annealed in a furnace at 750 and 900°C for various lengths of time. After heat treatment, the couple was examined metallographically using SEM and optical microscopy. Reaction layers were formed at the interfaces. The composition of the reaction layer was determined using EDS, and a compositional homogeneity range was observed for the Al3Ta phase.

3:30 PM
Solid State Reaction in the Au-Cu Wire Connection with Al-Cu Pad During Aging: *Guh-Yaw Jang*[1]; Jenq-Gong Duh[1]; Hideyuki Takahashi[2]; David Su[3]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec. 2 Kuang-Fu Rd., Hsinchu 300 Taiwan; [2]JEOL Ltd., Application & Rsch. Ctr., Tokyo Japan; [3]TSMC Ltd., Failure Analy. Div., 9, Creation Rd., 1, Sci.-Based Industl. Park, Hsinchu 300 Taiwan

In integrated-circuit packages, wire bonding technique is the preferred method for making electrical connections between the chip and lead frame. The influence of aging at 150°C from 0 h to 3000 h for interfacial reaction of the Au-Cu wire bonded with Al-Cu pad was investigated in this study. The wire-bond were first cold mounted in epoxy and then sectioned by using a slow speed diamond saw. Cross-sectional samples were ground and polished. To observe various intermetallic compounds (IMCs) and fine voids with field-emission scanning electron microscope, polished samples were ion milled with precision etching and coating system. The $Au_5(Al,Cu)_2$ IMC was found between Au-Cu wire and Al-Cu pad in the as-assembled wire-bond. After aging for 168 h, $Au_2(Al,Cu)$ IMC formed in the edge of wire-bond. The Al-Cu pad was consumed completely after aging for 500 h, and $Au_4(Al,Cu)$ IMC was observed at the Au-Cu wire/$Au_5(Al,Cu)_2$ interface. It was revealed that $Au_5(Al,Cu)_2$ IMC would be entirely transformed into $Au_4(Al,Cu)$ IMC in the wire-bond aged more than 1000 h. With the aid of microstructure evolution, quantitative analysis, the interfacial phase transformation between Au-Cu wire and Al-Cu pad could be probed and revealed.

3:50 PM Break

4:10 PM
Thermomigration of SnPb Solder Bump and Effect on Coarsening of Microstructure: *Y. C. Chuang*[1]; *Cheng-Yi Liu*[1]; [1]National Central University, Chemi. & Matls. Engrg., No.300, Jungda Rd., Jhongli, Taoyuan 320 Taiwan

Thermomigration(TM) is the net atomic flux driven by a thermal gradient. It has been reported that the TM-induced atomic transportation could be a serious reliability issue in C4 solder bump. Therefore, the study of solder thermomigration deserves a serious attention. In this study, stable thermal gradient (1000°/cm) has been created along a SnPb solder joint. The TM atomic flux was determined to be J=9.96 atom/cm2.s and we also obtain the important TM parameter, molar heat flux, Q??= 0.904 kJ/mole. Coarsening in SnPb solder has been studied by numerous researchers. Interestingly, we found that TM will affect coarsening behavior of SnPb solder bump. The detail kinetics of coarsening in the microstructure of SnPb solder under the effect of TM will be presented in this talk.

4:30 PM
Electromigration Effects at Bimetallic Interfaces: *Helen Theresa Orchard*[1]; Lindsay Greer[1]; [1]University of Cambridge, Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Electromigration is important to consider in integrated-circuit design as current densities in interconnects can approach 10^10 A m-2. At bimetallic interfaces, however, even lower current densities can strongly affect the growth of intermetallic compounds. Such diffusion and intermetallic compound formation are relevant, for example, for the reliability of wire bonds and vias on integrated circuits, and continue to be studied as operating conditions, including temperature, become more extreme. Existing analyses of electromigration at bimetallic interfaces are for cases in which compound growth is diffusion-controlled. For thin films, however, interfacial reaction barriers can be important and even dominate over diffusion control [Gösele and Tu, J. Appl. Phys. 53 (1982) 3252]. In the current paper, earlier analyses of electromigration effects are extended to include both diffusion and reaction kinetics. Wire bonds for high-temperature electronics are used as a case study, with interpretation of microstructural observations and discussion of further implications.

4:50 PM
Electric Current Effects on Metal-Metal Reactions: *Javier E. Garay*[1]; Umberto Anselmi-Tamburini[2]; Zuhair A. Munir[2]; [1]University of California, Dept. of Mechl. Engrg., Bourns Hall, Riverside, CA 92521 USA; [2]University of California, Chemi. Engrg. & Matls. Sci., Davis, CA USA

The effect of high-density direct current upon metallic systems was studied with the aim of decoupling Joule heat effects from other intrinsic effect. Investigations on interfacial reactions in the Ni-Ti and Cu-Ni system were carried out. Current densities of up to 2546 A.cm-2 were used in the temperature range of 625-850ºC. All of the intermetallic compounds (NiTi, Ni3Ti and NiTi2) present in the equilibrium phase diagram were identified in the product layer. In addition, b-Ti solid solutions formed in samples annealed above the a ® b temperature, 765ºC. The growth of all product layers was found to be parabolic and the applied current was found to significantly increase the growth rate of the intermetallic layers. In the Cu-Ni system, the effct of DC current was directional as the current was found to increase the diffusivity of Ni atoms in the direction that electrons were traveling in. The results on currents are explained in terms of current induced changes in the growth mechanism arising from changes in point defect mobility.

5:10 PM
Copper Hole Filling by Pulse Reverse Electroplating Method in 3-D SIP: Jin-Soo Bae[1]; Gun-Ho Jang[1]; *Jae-Ho Lee*[1]; [1]Hongik University, Matls. Sci. & Engrg., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 USA

Copper hole filling is the important factor in 3-D stacking interconnection of SIP(system in package). As the packing density is getting higher, the hole size is getting smaller. When DC electroplating is applied, defect-free hole cannot be obtained in small size hole. To prevent the defects in holes, pulse and pulse reverse current was appiled in copper hole filling. The size of holes are 50, 75, 100 micron in diameter and 100 micron in height. The holes were prepared by DRIE method. TaN and Ta was sputtered for copper diffusion barrier. Hole specimen were filled by DC, pulse and pulse/reverse current electroplating methods. The effects of current types on copper deposits were investigated. Cross section of holes were observed by SEM to find the defects in holes. When pulse reverse plating method was used, successful hole filling was obtained.

Phase Transformations Within Small-Size Systems: Order-Disorder Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee

Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Monday PM Room: 3002
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Zhong-Lin Wang, Georgia Institute of Technology, Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA; David E. Laughlin, Carnegie Mellon University, Matls. Sci. & Engrg., Pittsburgh, PA 15213 USA

2:00 PM Invited
Phase Equilibria in Nanocrystalline Magnetic Recording Materials: *David E. Laughlin*[1]; Mihaela Tanase[2]; Yingguo Peng[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Carnegie Mellon University, Physics, 5000 Forbes Ave, Pittsburgh, PA 15213 USA

It is well known that the size of grains or particles plays an important role in their thermodynamics and hence phase equilibria. For example, small particles are known to have different transition temperatures than their bulk counterparts. In this paper we will look at the phase equilibrium that exists in two important materials that are currently of interest as magnetic recording media, namely thin film Co based hcp alloys and nanoparticles of FePt. The role of the size of grains or particles on surface segregation will be discussed. For the case of FePt nanoparticles the role of particle size on the atomic ordering transition will be presented. Atomic ordering in particles may proceed either heterogeneously or homogeneously. Both these cases will be discussed. This research has been sponsored by the DSSC of CMU and SEAGATE Research of Pittsburgh, PA.

2:35 PM
The A1-L10 Phase Transition in the Alloy Nanoparticle: *Bo Yang*[1]; Mark D. Asta[1]; O. N. Mryasov[2]; T. Klemmer[2]; R. Chantrell[2]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., 2220 Campus Dr., Evanston, IL 60201 USA; [2]Seagate Research, 1251 Waterfront Place, Pittsburgh, PA 15222 USA

Monte-Carlo simulations are applied to investigate ordering processes in L10 alloy nanoparticles. We employ parametric, model-system studies to examine effects on nanoparticle ordering associated with reduced coordination and interfacial segregation, factors that become increasingly relevant as the surface to volume ratio increases with decreasing particle size. We also explore the kinetic pathways for nanoparticle ordering under the isothermal annealing conditions. On the basis of calculated long-range-order parameters defined to characterize chemical order in the context of magnetic properties, we find reduction in the thermodynamic ordering temperatures with decreasing nanoparticle size. This reduction of the ordering temperatures is found to be enhanced with an increasing driving force for the surface/interface segregation. We find a tendency toward the formation of the metastable multidomain configurations under the condition of isothermal annealing from disordered fcc nanoparticles state.

3:00 PM
Size Dependence of $L1_0$ Ordering in FePt Nanoparticles: Y. K. Takahashi[1]; T. Koyama[1]; T. Ohkubo[1]; *K. Hono*[1]; [1]National Institute for Materials Science, 1-2-1 Sengen, Tsukuba 305-0047 Japan

As the recording density approached to 1 Tbit/in2, the size of recording bit will be smaller than 10 nm. To overcome the thermal instability due to the reduction of the size, L10-FePt with a large magnetocrystalline anisotropy is thought to be the most promising material for ultrahigh density magnetic recording media appliations. Since as sputtered films have disordered fcc strcutrue, they must be ordered to the L10 structure either by in-situ annealing or post annealing. In the course of the studies of ordering from fcc to L10 FePt, we found that there is a size dependence of the ordering when the particle size become smaller than approximately 5 nm. Simple Bragg-Williams calculation supports that the ordering becomes unstable when the particle size become smaller than a critical size. The critical size depends on the interfacial energy. This finding indicates that we must consider both the superparamagnetic limit and the ordering limit when we design the FePt recording media.

3:25 PM Break

3:40 PM Invited
Structure, Phase Transformation and Twinning of Magnetic Nanocrystals: *Zhong Lin Wang*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Rm. 163, Love Mfg. Bldg., Atlanta, GA 30332-0245 USA

Depending on the Fe to Pt elemental ratio, the Fe-Pt alloys can display chemically disordered face centered cubic phase or chemically ordered phases. We report detailed microscopic studies on phase transformation, coalescence and twin structure formation of thermally annealed 6 nm FePt nanocrystals under high vacuum on an amorphous carbon surface. Our high-resolution transmission electron microscopy (HRTEM) studies show that A1 to $L1_0$ phase transformation occurs at 530°C. The multilayered nanocrystal assemblies coalesce to form larger grains at 600°C. Shape and surface atomic arrangement of the monodisperse FePt magnetic nanocrystals and their evolution induced by annealing have been studied. Truncated octahedron enclosed by flat {100}, stepped {111} and zig-zag {110} facets is the dominant shape adopted by the as-synthesized FePt nanocrystals. The Marks decahedron shaped FePt nanocrystals and icosahedron related multiply twined FePt nanocrystals are also identified in the as-synthesized nanocrystals. A new structural model the multiply twined nanocrystals related to icosahedron has been proposed. After annealing, the {110} facet disappears; the regular cuboctahedron becomes a dominant shape for the chemically ordered FePt nanocrystals. Atomic arrangement on the FePt nanocrystals surfaces is the same as that in the volume although some defects such as atomic steps and kinks exist. Iron atoms are preferably the terminating layer of the {100} surface after annealing. Thanks to the contribution made by Drs. S.H. Sun, H. Zeng, J.P. Liu, J. Li and Z.R. Dai.

4:15 PM
Ordering Reaction and Sintering Behavior in [FePt]100-xCrx Nanoparticles: *Chandan Srivastava*[1]; Gregory B. Thompson[1]; James W. Harrell[2]; David E. Nikles[3]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 34587 USA; [2]University of Alabama, Dept. of Physics, 206 Gallalee Hall, Box 870324, Tuscaloosa, AL 35487 USA; [3]University of Alabama, Dept. of Chmst., Box 870336, Tuscaloosa, AL 35487-0336 USA

We report the effect of incorporating Cr into FePt nanoparticles as a mechanism to reduce the ordering temperature while magnetically decoupling sintered nanoparticles. Self-assembled arrays of FePt nanoparticles are potential systems for next-generation magnetic recording. STEM/EDX measurements and XRD confirmed that Cr was successfully incorporated into the FePt lattice in the as-synthesized state. Upon increasing the temperature, the Cr does not precipitate out of the particle, even after L10 ordering. XRD indicates a modest increase of the lattice spacing of the [FePt]100-xCrx at temperatures prior to the L10 phase transformation. This lattice expansion behavior is not observed in FePt nanoparticles. Scherrer analysis of the XRD peak widths indicate that Cr hinders grain growth in the sintered particles at elevated temperatures. A series of time and temperature coercivity measurements for the [FePt]100-xCrx will be presented and discussed in terms of the phase transformation and sintered morphology of the nanoparticles.

Shape Casting — The John Campbell Symposium: Filling and Feeding

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday PM Room: 2008
February 14, 2005 Location: Moscone West Convention Center

Session Chair: John T. Berry, Mississippi State University, Mississippi State, MS 39762-5925 USA

2:00 PM
Gating Design After Campbell - A Practical Experience of the Daily Struggle With Porosity: *Salvador Valtierra*[1]; [1]Nemak, R&D, Arco Vial Km 3.8, Garcia, Nuevo Leon 66000 Mexico

Gating design can be considered as an Art, Religion, Science or sorcery (actually we are not sure about any of them at all!) In the 60s (Ancient history BC) gating considered mainly the effect of shape and size of gating systems on the speed of flow. And some deviations to the hydraulic model to refine the systems, introducing heat losses into the gating systems. Despite the huge amount of information coupled with computer simulation the successful design of a casting system was not always achievable. History can be rewritten after Campbell contributions. The present work shares the experience accumulated during 12 years of a love-hate relationship with oxides generated by bad gating designs. Also a description of the resulting collection of oxides, bubbles trails and accompanying friends, observed in castings poured in semi-permanent molds using traditional gating designs. These are compared with the successful results obtained when using Campbell's rules.

2:25 PM
Filling Mold Cavities at High Flow Velocities Without Turbulence: *John L. Jorstad*[1]; Mike Thieman[2]; [1]JLJ Technologies, Inc., 9112 Donora Dr., Richmond, VA 23229 USA; [2]THT Presses, Inc., 7475 Webster St., Dayton, OH 45414 USA

Professor Campbell has long taught that molten aluminum that exceeds 0.5 meters/second flow velocity during casting is increasingly prone to turbulence-related defects such as oxide folds that significantly reduce component performance. Semi solid aluminum melts behave quite differently. Partially solidified melts having a significant fractions of solid (approximately 50%) can flow at velocities exceeding 2 meters/second while still maintaining a stable flow front. The viscosity of molten aluminum increases as the fraction solid becomes greater, and the more viscous a melt, the faster it can flow without turbulence. Semi solid melts are thus better-able than liquid to fill thin and very detailed mold cavities while still producing high integrity parts. This paper will describe the results of set of casting trials wherein the effects of in-gate velocity on casting integrity was determined while casting liquid as well as semi solid A356 alloy.

2:50 PM
Designing Reliable Castings: *Malcolm Blair*[1]; Raymond W. Monroe[1]; Christoph Beckermann[2]; Richard Hardin[2]; Kent Carlson[2]; John Griffin[3]; Charles Monroe[2]; [1]Steel Founders' Society of America, 780 McArdle Dr., Unit G, Crystal Lake, IL 60014 USA; [2]University of Iowa, Mechl. Engrg., 2212 Engrg. Bldg., Iowa City, IA 52242-1527 USA; [3]University of Alabama, Matls. Sci. & Engrg., 1016 15th St. S., Birmingham, AL 35294-4552 USA

Castings offer flexible and efficient design solutions for many products. However, designs are generally based on strengths of materials calculations and the experience of the designer. Industrial experience with current products tends to dominate in the development of future designs. This process leads to an incremental development of design utilizing factors of safety, which lead to increasing weights of components and inevitably inefficient uses of materials. Factors of safety are a way of developing cushions to avoid the unforeseen failures because of unexpected loads on the part or reductions in expected properties due to their manufacture. All product forms are subject to this paradigm. In castings the unquantifiable factors such as shrinkage, microshrinkage and surface indications lead to more and more conservative design rules. Non-destructive testing such as radiography and surface inspection do not give the designer any way of assessing the effect of shrinkage or surface indications. Programs to design for these casting features and develop new non-destructive standards and techniques are essential to the efficient use of castings. Work to develop more quantitative design rules are addressing the issue of shrinkage and surface indications. The development of new quantitative design rules for castings is progressing for steel castings through an integrated approach between producers, users and researchers.

3:15 PM
Vortex-Gate Design for Gravity Casting: *Fu-Yuan Hsu*[1]; Mark R. Jolly[2]; John Campbell[2]; [1]Auspicium Co., Ltd., 2F-3, No. 4, Sec. 1, Jen-Ai Rd., Taipei Taiwan; [2]University of Birmingham, IRC in Matls. for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

A novel runner system design, named a Vortex-Gate, has been explored for aluminum gravity casting. Using this design the velocity of flow of the liquid metal flow was controlled below the critical value and, at the same time, a high flow rate was maintained and the flow behavior does not appear to generate "bi-film" defects. The "virtual" experiment using a computational modeling package, and the "physical" experiment, a real-time X-ray radiography study, were found to be in reasonable agreement.

3:40 PM Break

3:50 PM
Characterization of Metal Filling Behavior of Lost Foam Castings Using Real Time X-Ray Technology: *Wanliang Sun*[1]; Harry E. Littleton[1]; Charles E. Bates[1]; [1]University of Alabama, Matls. Sci. & Engrg., 917 Bldg., 1530 3rd Ave. S., Birmingham, AL 35294-4480 USA

Characterization of metal filling behavior of lost foam castings is very important to understand, and in turn control the foam pyrolysis products related casting defects. In this study, metal filling of lost foam castings at various conditions was characterized using non-invasive real time X-Ray visualizations. A simple rectangular plate was used as casting sample. Parameters such as metal filling velocity, number of metal converging lines, metal front profile and metal progressing angle were used to characterize the metal filling behavior. Metal filling behaviors under different process variables such as foam pattern fusion level, coating permeability, casting thickness, glue joint types and gating types were investigated. It is observed that under different conditions, the metal filling of lost foam castings changed significantly. Correlation between process parameters and metal filling behavior will assist the process control of the lost foam casting process.

4:10 PM
The Five Feeding Mechanisms: *Christopher Malcolm Gourlay*[1]; Arne Kristian Dahle[1]; [1]University of Queensland, CRC for Cast Metals Mfg. (CAST), Sch. of Engrg., St. Lucia, Queensland 4072 Australia

This paper summarises a number of studies on the rheology of solidifying alloys and discusses these results with reference to the five feeding mechanisms proposed by Campbell. Distinct changes in rheological response at the coherency and maximum packing solid fractions are correlated with transitions between feeding mechanisms. The effect of alloy and solidification conditions on the development of mush strength during solidification is discussed with respect to the operating feeding mechanisms and particular attention is given to the role of burst feeding. A similar mechanism is also shown to operate in the formation of banded defects. The distribution of solid within a casting can affect where different feeding mechanisms operate. Results are presented showing that when filling occurs with partially solid material, the crystals migrate towards the centre of the cross-section. This leads to uneven solid fraction distributions in the cavity and therefore local areas of lower solid fraction which can operate as preferential feeding paths.

4:30 PM
Experimental Quantification of Interdendritic Permeability: *Øyvind Nielsen*[1]; Svein Ove Olsen[1]; [1]SINTEF, Matls. & Chmst., PB 124 Blindern, Oslo 0314 Norway

The interdendritic permeability is a key parameter for the understanding of feedability and porosity formation in shaped castings. In the present work, a permeameter has been developed in which a melt is cast into a vertical steel pipe encompassed by two independent tube furnaces (top and bottom), used to set up a thermal gradient that promotes directional solidification from the bottom. The sample temperature is measured by a series of thermocouples along the sample length, and the pipe bottom is opened when the temperature close to the bottom reaches a predefined value in the solidification interval. Thus, interdendritic liquid is allowed to exude through the bottom sample surface, and the flow rate is measured with a graphite float placed at the top surface. The advantages with the design are: (i) the special flux alloys used in previous permeameters are not needed because the top surface remains liquid throughout the experiment, (ii)

the cooling rate can be controlled, and (iii) the temperature at which interdendritic feeding stops can be measured. Experiments have been carried out for A356, and the measured interdendritic flow rates have been used to quantify the differences in feedability due to the addition of grain refiner and modification by strontium. The results can be used e.g., to shed light on the well-known increase in microporosity with increasing strontium content. The main challenge with the experiment is the fact that the solid fraction, and thus the permeability, varies along the sample length. A mathematical model, taking into account the macrosegregation formation due to shrinkage and exudation, that can be used to extract permeability data from the measurements is being developed.

4:50 PM
The Critical Gate Velocities for Magnesium Casting Alloys (ZK51A): *Farhad Bahreinian*[1]; [1]University of Toronto, Matls. & Sci. Engrg., 184 College St., Rm. #140, C/O Prof. S. Argyropulos, Toronto, Ontario M5S 3E4 Canada

The effects of gate entry velocity on the soundness of the ZK51A Alloy were investigated by considering a new theory, The Critical Gate Velocities. The surface turbulence and oxide layer formation were evaluated using an accurate video camera. The results show that in this alloy, the critical gate velocity is about 0.5 m/s. Surface turbulence phenomenon occurs at velocities more than 0.5 m/s, which decreases the mechanical properties. The critical gate velocity decreases to 0.25 m/s when the casting thickness is 10 mm. The results are well correlated to the critical gate velocities values observed in aluminum alloys.

5:10 PM
The Connection Between Filling, Feeding and Applied Pressure in Cast Aluminum Alloys: *Rogelio Luck*[1]; Robert P. Taylor[1]; John T. Berry[1]; [1]Mississippi State University, Dept. of Mechl. Engrg., PO Box ME, Mississippi State, MS 39762 USA

Oxide bifilms often appear to be linked with the dispersed type of porosity observed in longer freezing range aluminum alloys. Although measures exist to help prevent the inadvertent incorporation and the subsequent unfurling of these bifilms into potential pores, little information would seem to be available specific to feeder design for this class of alloys. This is in contrast to the information available concerning the design of feeding systems for short freezing range alloys, in particular low-carbon steels, where macroscale shrinkage is liable to occur. The various measures required regarding solidification order, required feeder volume, range of feeding are plentiful for this second class of alloys and are more than adequately covered by Campbell's feeding rules. The paper attempts to draw together facts which might help fully quantify the needed guidelines for feeding long freezing range aluminum alloys.

Superalloys and Coatings for High Temperature Applications: Bond-Coat Technologies - II

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3
Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Monday PM — Room: Nob Hill A/B
February 14, 2005 — Location: San Francisco Marriott

Session Chairs: Richard S. Bellows, Solar Turbines Inc., Matls. & Process Engrg., San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Dept. of Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA

2:00 PM Invited
Integrated Approach to Superalloy and Coatings Technology Development: *David A. Litton*[1]; [1]Pratt & Whitney, Matls. & Processes Engrg., M/S 114-41, 400 Main St., E. Hartford, CT 06108 USA

To provide customers with higher performance and reduced operating costs, noise, and emissions, hot section engine component designs demand materials systems that can perform predictably in more hostile environments for longer exposures. To reduce maintenance costs, materials technologies that allow for refurbishment to extend service lives of parts over multiple engine overhauls are critical. For the same reasons, part count reduction is driving the design of parts with multiple airfoils such as integrally-bladed rotors and vane clusters. Integrated designs to monitor the condition of hot section components are under study to maximize uninterrupted service. These considerations multiply the constraints on the development of superalloy and coating materials and processing technology. This paper will correlate requirements for materials properties to these design constraints and summarize current approaches in materials and processing technology research and development to maximize customer value.

2:30 PM Invited
Secondary Reaction Zones in Coated Single Crystal Superalloys: *Pierre Caron*[1]; Odile Lavigne[1]; Catherine Ramusat[1]; [1]ONERA, DMMP/MHT, 29, Ave. de la Div. Leclerc, Châtillon 92322 France

In addition to the usual interdiffusion zone existing between nickel-based superalloys and their overlay or aluminide protective coatings, some secondary reaction zones (SRZ) may form on certain conditions. SRZ have a deleterious effect on the mechanical strength of the alloy substrate when extending over several tens of micrometers. Occurrence of SRZ beneath a Pt-modified aluminide bond coat has been checked in a number of superalloys in order to determine the influence of the substrate chemistry. More specifically, minor and major compositional modifications as well as stress relaxing heat treatments have been tested in a new generation Re and Ru bearing single crystal superalloy in order to avoid the formation of SRZ. The microstructure of the specimens has been investigated in the as-coated state and after long-term high temperature ageing treatments.

3:00 PM
The Effect of Alloy Composition on the Precipitation of Topological Close Packed Phases at Aluminide Coatings on Superalloys: *Catherine Mary Rae*[1]; Matthew Simon Hook[1]; Roger C. Reed[2]; [1]Cambridge University, Dept. of Matls. Sci., Pembroke St., Cambridge CB2 3QZ UK; [2]University of British Columbia, Dept. of Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Aluminide coatings provide considerable protection for turbine components subjected to hostile environments. However, inter-diffusion between the coating and the substrate causes changes in the phase composition and morphology which destroys the creep resistance of the alloy in the affected area and which, in a thin walled section, can considerably reduce the ability of the component to support load. We have compared the evolution of low temperature, high activity Aluminide and Platinum-modified Aluminide coatings on three very different alloys; RR3000, CMSX-4 and TMS75. We discuss the factors determining the distribution of the refractory elements in these coatings we observe that the precipitation of TCP phases in the interdiffusion zone varies considerably with the alloy composition. The morphology determines the speed with which the precipitates form, and we suggest that this may control the rate of ingress of the coating into the substrate and also the formation of the secondary reaction zone.

3:25 PM
Microstructure and Mechanical Properties of Al-Ni-Ru Alloys: *Fang Cao*[1]; Qiang Feng[1]; Tresa M. Pollock[1]; [1]University of Michigan, 3062 H. H. Dow Bld., 2300 Hayward St., Ann Arbor, MI 48109 USA

Systems based on RuAl have the potential to perform as bond coat materials in superalloy-Thermal Barrier Coating (TBC) systems for advanced gas turbine engines. For coating applications, an understanding of the ternary Ni-Al-Ru system is essential since diffusion of Ni from the Ni-based superalloy substrate is unavoidable during manufacture and service of the TBC systems. In order to address this subject, alloys with selected compositions in the Al-Ni-Ru system were melted by Crystalox induction cold crucible melting and the microstructure and present phases were examined. Compression tests were performed at room temperature up to 973 K for these alloys. Creep behavior was investigated by compression creep tests at 1273K in vacuum condition. Finally, the deformation mechanism was studied by dislocation substructure analysis and identification.

3:50 PM Break

4:10 PM Invited
Cost Reductions in the Aerospace Coatings Industry: *Ken S. Murphy*[1]; [1]Howmet Research Center, 1500 S. Warner St., Whitehall, MI 49461 USA

Cost reductions come from many places in the manufacturing process. Some are from the systems used, the empowerment of workers to effect change, and relationships with suppliers in addition to the traditional process/equipment improvements and raw material costs. This paper describes how the various management techniques and alternative material selection are integrated into the aerospace coating shop floor. It will discuss how the Alcoa Business System has impacted cost at Howmet - Thermatech Coatings and how Palladium is being assessed as an alternative to Platinum as the noble metal used in high temperature diffusion aluminide coatings.

4:40 PM
Superalloy-Dependent Stability of β-NiAl Phase in MCrAlY Coatings: *Emmanuel Perez*[1]; Travis Patterson[1]; Yong-Ho Sohn[1]; [1]University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. & Dept. of Mechl., Matls. & Aeros. Engrg., Orlando, FL 32816-2455 USA

Hot section components in gas turbines can be MCrAlY-coated to provide the component with an Al reservoir that maintains a protective oxide later on the coating's surface. Over the service life of the component, the coatings degrade by composition and phase changes due to oxidation/hot-corrosion on the coating's surface, and due to multicomponent interdiffusion from and into the superalloy substrate. This interdiffusion process leads to Al depletion from the coating, and in many cases is the life-limiting factor. In this study, the rate of degradation for MCrAlY coatings on several Ni-base superalloys was examined with an emphasis on the composition-dependence of Al interdiffusion. Several diffusion couples have been prepared to examine the composition-dependence of Al with several Ni-base superalloys. Concentration profiles obtained from EDS and EPMA were analyzed to determine the effective interdiffusion coefficient. These results will be discussed in terms of composition-dependence of the Al interdiffusion and the phase stability of β-NiAl in MCrAlY coatings.

5:05 PM
Inhomogeneous TGO Formation in Thermal Barrier Coatings with Rough Top-Coat/Bond-Coat Interfaces: *Feng Tang*[1]; Leonardo Ajdelsztajn[1]; Julie M. Schoenung[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

The thermal cycle lifetime of thermal barrier coatings (TBCs) is affected greatly by the features of the thermally grown oxides (TGOs). This paper presents our observation of inhomogeneous TGO formation as a result of the roughness profile at the top-coat/bond-coat interface. NiCrAlY bond coats were thermally sprayed with the LPPS and HVOF processes, and the YSZ top coats were sprayed with the APS process. These processes resulted in a rough bond coat/top coat interface. The TBC specimens were thermal cycled for more than 300 one-hour cycles, in air at 1121°C. It was found that the TGO that formed at the peaks in the roughness profile were notably thicker than the TGO that formed at other areas. The mechanisms for the inhomogeneous TGO formation and its effect on the TBC lifetime are discussed. Characterization methods included SEM, XRD, and EDS.

5:30 PM
The Development of the Virtual Coating Program: *Rudder Wu*[1]; Makoto Osawa[1]; Hiroshi Harada[1]; [1]National Institute for Materials Science, High Temp. Matls. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

A thermal barrier coating (TBC) simulation program based on the energy release rate model has been developed to evaluate delamination along the TGO/bond coat interface. The program accepts three input categories including engine parameters (overhaul/cruising TIT, thin-film cooling), aviation parameters (flight time/cycles, taking-off/thrust-reversal time) and material properties. Based on these parameters, real-time cyclic temperature profile across the thickness of a TBC system is generated. Consequently, the empirically-determined parabolic functions of top coat sintering and TGO growth were also ascertained in the calculation. Upon correlating the calculated energy release rate with the published mode II interface toughness ($50Jm^{-2} \leq \Gamma \leq 80Jm^{-2}$), a critical TGO thickness of ~ 3-4 microns depending on the extent of top coat sintering was obtained, comparable to that observed in a commercially used nozzle-guided vane. Therefore, this program serves as a platform for TBC design and enables lifetime prediction and lifetime-remaining assessments.

Surface Engineering in Materials Science - III: Laser Processing for Surface Modification

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Monday PM — Room: 2022
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Narendra B. Dahotre, University of Tennessee, Dept. of Matl. Sci. & Engrg., Knoxville, TN 37932 USA; Roger Narayan, Georgia Tech, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

2:00 PM
Laser Deposition of Titanium Boride Reinforced Titanium Alloy Composites: *Davion Hill*[1]; Rajarshi Banerjee[1]; Jaimie Tiley[2]; Peter C. Collins[1]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

Due to their wide applicability, there is considerable interest in the development of metal-matrix composites consisting of hard precipitates, such as transition-metal borides, dispersed in a metallic/alloy matrix. One such system that has generated considerable interest in recent years is the titanium boride in titanium alloy matrix system. Laser engineered net-shaping (LENS™) is a directed laser deposition process which uses a powder feedstock and appears to be a promising technology for the processing of these metal-matrix composites. Two types of TiB reinforced composites have been studied, one with an a+b matrix based on the alloy Ti-6Al-4V and the other with a primarily b matrix based on the alloy Ti-5Al-5V-5Mo-3Cr-0.5Fe (TIMETAL 5553). Using a feedstock consisting of a blend of pre-alloyed Ti-6Al-4V (or TIMETAL 5553) and elemental boron powders, these composites have been deposited in a single step via the LENS™ process. These as-deposited composites exhibit a refined homogeneous distribution of TiB precipitates within the alloy matrix, a consequence of the rapid solidification rates inherent to the LENS™ process. The microstructure of the LENS™ deposited composites has been investigated in detail using SEM and TEM based techniques. The room temperature tensile properties and wear resistance of these composites is currently being investigated and will be presented in this paper.

2:20 PM
Laser Processing of Bulk Crystalline Alloys for Improved Corrosion Resistance: *J. G. Hoekstra*[1]; G. J. Shiflet[1]; S. J. Poon[2]; J. R. Scully[1]; J. M. Fitz-Gerald[1]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA USA; [2]University of Virginia, Dept. of Physics, Charlottesville, VA USA

In general it has been shown that amorphous metals have several attractive properties, particularly in areas of wear and corrosion resistance. Advances in metallic glass chemistries with enhanced solidification characteristics have furthered the development of bulk metallic glasses. In this study laser surface treatments were conducted in two alloy systems with glass forming abilities: Al and Fe based alloys. Cross-sectional analysis showed melt depths ranging from 0.5 to 3 microns largely dependent on the total surface dose. The resulting microstructures were correlated with electrochemical analysis and devitrification behavior. Potentiodynamic polarization results and impedance measurements show significant improvement over native materials, exhibiting several characteristics of amorphous surface layers. Characterization was performed by electron microscopy (SEM), energy dispersion (EDS), diffraction (XRD), and electrochemical analysis.

2:40 PM
Pulsed Laser Deposition of Tetrahedral Amorphous Carbon Nanocomposites: *Roger Jagdish Narayan*[1]; [1]Georgia Institute of

Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr. NW, Rm. 361, Atlanta, GA 30332-0245 USA

Tetrahedral amorphous carbon-metal nanocomposite films of >1 micron thickness were deposited on Ti-Al-V alloy substrates using a novel multicomponent target design during pulsed laser deposition. We have created novel nanostructured tetrahedral amorphous carbon-silver and tetrahedral amorphous carbon-titanium nanocomposite thin films. These nanocomposite films were characterized using Z-contrast scanning transmission electron microscopy (STEM-Z) and electron energy loss spectroscopy (EELS). Scratch testing demonstrated good adhesion of tetrahedral amorphous carbon-metal nanocomposite films to Ti-Al-V alloy. Nanoindentation testing of the tetrahedral amorphous carbon-metal films demonstrated excellent hardness and modulus values, within the range of 35 GPa and 350 GPa, respectively. Wear testing demonstrated coating lifetimes in excess of 300,000 cycles for tests conducted in Ringer's USP solution under ~ 1 GPa initial maximum Hertzian pressure.

3:00 PM

Heat Absorbed by 410 Stainless Steel During Laser Surface Modification Processes: *Jaimin P. Rao*[1]; [1]Illinois Institute of Technology, 3001 S. Michigan Ave., Apt. # 1804, Chicago, IL 60616 USA

Energy absorption and temperature distribution within the substrate material plays a very important role in laser surface modification processes. This study is to investigate the amount of energy absorbed by 410 stainless steel, when it is irradiated by High Power Direct Diode laser. Both experimental and computational techniques are employed in this study. Power absorbed by the substrate is determined by using the measured temperature history in an inverse heat transfer analysis by FEM based HOTPOINT system. The spatial distribution of actual power intensity on the irradiated surface is obtained as a function of incident power level. The results show that the coefficient of net heat absorption is in the range of 50-70%, which agrees with the direct simulations and data available from literature. It is also found that power intensity distribution along long axis is not top hat as claimed by the manufacturer.

3:20 PM

Studies on Laser Composite Surfacing of Aluminium with Silicon Carbide and Alumina: *Jyotsna Dutta Majumdar*[1]; B. Ramesh Chandra[1]; A. K. Nath[2]; Indranil Manna[1]; [1]Indian Institute of Technology, Metallurgl. & Matls. Engrg., Kharagpur, W. Bengal 721302 India; [2]Centre for Advanced Technology, Laser R&D, Block B, Indore, Madhya Pradesh 452 013 India

The present study aims at improving the wear resistance of aluminium by forming a thin layer of SiC and Al2O3 dispersed metal matrix composite by laser surface engineering. Laser processing was carried out with a 2 kW continuous wave CO2 laser by pre-placing a thin layer of SiC and Al2O3 on the surface of Al substrate and subsequently melting the surface with high power laser. The main process variables for the present study were laser power, scan speed, particle chemistry and thickness of pre-placed layer. A detailed characterization of the modified zone was undertaken to study the the morphology and distribution of the particles. The average microhardness and wear resistance of the composite surfaced layer was studied in details to compare the influence of process parameters on the kinetics and mechanism of wear. Finally, the optimum processing zone for laser composite surfacing of Al with SiC and Al2O3 was derived.

3:40 PM Break

3:55 PM

Laser Cladding of W-Cu Composite on Bronze Substrate: *Sheng-Hui Wang*[1]; Lijue Xue[1]; [1]National Research Council of Canada, Integrated Mfg. Tech. Inst., 800 Collip Cir., London, ON N6G 4X8 Canada

The feasibility of depositing W-Cu composite overlays by laser cladding technique was explored. Two types of tungsten powders, with an average size of 10 microns and 32 microns, were used to deposit coatings with various thicknesses. It is shown that tungsten particles tend to separate from the copper molten pool or coalesce within it, which occurs more easily for the fine powders than for the coarse ones, making it simpler to deposit dense coatings with coarse tungsten powders. Laser cladding of uniform thick coatings was proven to be difficult, due to the separation of tungsten from copper. Nickel addition enhances the possibility of depositing uniform W-Cu composite coatings, preventing or limiting the segregation and separation of the constituents. The preliminary results indicate that, by adjusting processing parameters and powder-blend compositions, it is feasible to fabricate uniform W-Cu overlays with various thicknesses that are metallurgically bonded to the substrates.

4:15 PM

The Effects of Glass-Forming Coatings on Fatigue Behavior of 316L Stainless Steel: *Fengxiao Liu*[1]; W. Yuan[1]; C. L. Chiang[2]; J. P. Chu[2]; P. K. Liaw[1]; R. A. Buchanan[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Dougherty Engrg. Bldg., Knoxville, TN 37996 USA; [2]National Taiwan Ocean University, Inst. of Matls. Engrg., Keelung 202 Taiwan

The Zr-31Cu-13Al-9Ni (atomic percent) glass-forming coating was deposited onto the 316L stainless steel by magnetron sputtering, followed by subsequent annealing. The effects of the glass-forming coating on the fatigue behavior of the 316L stainless steel were investigated. The application of the coating gave rise to the significant increases in both the fatigue life and the fatigue limit. Depending on the stress applied to the steel, the fatigue life can be increased by 30 times, and the fatigue limit can be elevated by 30%. Compared with the annealed steel, the cold-rolled steel substrate increased the fatigue life due to the higher strength of the steel. The post heat treatment of the glass-forming coating lowered the fatigue life because of the partial crystallization of the coating. High compressive residual stresses introduced when depositing were determined by the curvature measurement. The fractography showed that the coating remained well adhered to the steel. The surface-roughness measurements also indicated the improvement of the surface conditions. Therefore, the improved fatigue behavior indicated that the excellent adherence of the coating to the steel, the improved surface condition, together with the high compressive residual stresses, were the main reasons for the improvement of the fatigue behavior of the coated steel system.

4:35 PM

Laser Melting of Graded Coatings (Fe-SiC-Ni-Cr) on AISI 316L Stainless Steel: *Dillibabu Sastikumar*[1]; Alagan Viswanathan[1]; M. Jamal Mohamed Jaffar[2]; A. K. Nath[3]; [1]National Institute of Technology, Dept. of Physics, Tiruchirappalli, Tamilnadu 620 015 India; [2]Jamal Mohamed College, Dept. of Physics, Tiruchirappalli, Tamilnadu 620 020 India; [3]Center for Advanced Technology, Industrial CO2 Laser Sec., Indore 452 013 India

AISI 316L Stainless steel is widely used in the aggressive corrosive environments because of its high corrosion resistance. However, its wear resistance is poor due to relatively low hardness (about 200 HV). It could not be hardened by heat treatment technique due to its austenitic phase. Different routes are being attempted using laser surface modification technique to improve its surface hardness. In this study, AISI 316L SS was preplaced with graded coatings of Fe-SiC-Ni-Cr and laser melted for improving its surface hardness. Three different compositions of Fe-SiC-Ni-Cr were prepared and preplaced one over another (thickness of each composition layer was around 300im) on the surface of AISI 316L SS. The preplaced graded coatings were melted with laser radiation with different powers (3.3 & 2.0 kW) and scan speeds (1.0, 2.0 & 3.0 m/min.) This study showed the formation of two distinct layers (with and without microstructure) when the laser power was 2 kW and scan speeds were 1.0 & 2.0 m/min. When the scan speed was 3.0 m/min., intermixing of layers was observed. Mixing of layers was prominent when the power was 3.3 kW (scan speeds 3.0 & 2.0 m/min). When the scan speed was 1.0 m/min, complete mixing of layers (homogeneous alloying) was observed. The layers without and with microstructure exhibited microhardness in the range of 1400-1500 HV and 170-250 HV, respectively. The microhardness near the surface of the complete alloyed region was about 250 HV up to a depth of 50 microns and after that 700-900HV up to the alloy-substrate interface (depth 650 microns). The hardness obtained in the complete alloyed region is about 4 times of the AISI 316L SS substrate. The paper also discusses different phases formed during laser melting and distribution of elements in the laser melted regions.

Texture and Microstructure in Thin Films and Coatings: Techniques and Coatings

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Monday PM Room: 3010
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Hualong Li, ResMat Corp., Montreal, Quebec H3A 2B3 Canada; Masashi Watanabe, Lehigh University, Bethlehem, PA 18015 USA

2:00 PM Invited
In-Fab Control of Texture and Microstructure in Microelectronic Devices: *Kris Jan Kozaczek*[1]; [1]Hypernex, Inc., 3006 Rsch. Dr., State College, PA 16801 USA

Microstructural features such as phase composition, crystallographic texture, and grain size distribution affect the adhesion, stress voiding, electromigration resistance, and electrical resistivity of thin films and interconnects used in 90 nm and 65 nm technologies. The off- and in-line R&D efforts allow one to optimize the microstructure for film and interface properties, and ultimately for improved performance. One of the tools used in-line for microstructure tailoring is an automated x-ray diffraction (XRD). We will show the examples of in-fab applications of XRD tools capable of mapping 200 and 300 mm wafers for phase, grain size, and texture with throughout rates up to 40 wafers per hour. Current deposition and processing techniques such as CVD, PVD, electroplating, annealing, and electro-mechanical polishing provide numerous opportunities for microstructure control throughout the fabrication process. Adversely, tool instability or process excursions are reflected in microstructural deviations across one wafer or from wafer to wafer. XRD has been successfully applied in the leading semiconductor fabs to establish the processing-microstructure-property relationships, and as a process monitor sensitive to deposition conditions. The examples will focus on a typical processing route in fabrication of damascene copper interconnects. Additional applications in metal gate processing and in manufacture of ferroelectric memory devices will be presented.

2:30 PM
Practical Comparison of Orientation Determination Techniques in Transmission Electron Microscopy: *Masashi Watanabe*[1]; David B. Williams[1]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Electron backscatter diffraction (EBSD) techniques in scanning electron microscopy (SEM) can automatically measure and visualize individual crystallographic orientations of grains in polycrystalline materials. This approach is more efficient than conventional electron diffraction in transmission electron microscopy (TEM) because sample preparation for SEM-EBSD is much easier (no need for electron-transparent thin films) and much larger areas can be examined relatively quickly. However, TEM approaches are still useful because the spatial resolution is higher than SEM-EBSD and crystallographic relationships between grains can be correlated directly with chemistry around the grain boundaries. Recently, several automated orientation-determination techniques have become available in TEM: e.g. automatic crystallography for TEM (ACT) and orientation determination based on a convergent beam electron diffraction method. In this study, the TEM-based orientation-determination techniques will be compared practically in terms of accuracy and usefulness in misorientation measurements between grains in thin-film specimens.

2:50 PM
Depth-Resolved Internal Strain and Texture in Coatings Using Micro-Focused High-Energy X-Rays: *Jonathan Almer*[1]; Magnus Oden[2]; [1]Argonne National Laboratory, Advd. Photon Source, 9700 S. Cass Ave., Bldg. 431, Argonne, IL 60439 USA; [2]Lulea University of Technology, Engrg. Matls., Sweden

We investigate internal strain, microstructure and texture in physical-vapor-deposited (PVD) metal nitride coatings TiN and CrN. The non equilibrium nature of the PVD process leads to high defect densities and internal stresses in these coatings. We describe a new technique, using high-energy synchrotron x-rays, focusing optics and an area detector, which allows strain and texture for multiple crystallographic planes to be measured by a single x-ray exposure. The coatings are measured in a cross-sectional transmission geometry, rather than the (traditional) reflection geometry. The depth resolution is therefore direct and given by the x-ray beam size (down to one micron), rather than cumulative as in reflection-based methods. Unique texture states, nonlinear strain-orientation distributions and strain gradients are observed for different coatings. These observations are compared with existing micromechanical models and correlated with deposition conditions. This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

3:10 PM
Grain Boundary Energy as a Function of Misorientation in <111> Fiber-Textured Al Films: Experiment and Computation: *Katayun Barmak*[1]; Jihwan Kim[1]; Chang-Soo Kim[1]; Gregory S. Rohrer[1]; Anthony D. Rollett[1]; Hao Zhang[2]; David J. Srolovitz[2]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton Inst. for the Sci. & Tech. of Matls., Princeton, NJ 08540 USA

Electron back scatter diffraction and a statistical multiscale method have been used to determine the relative boundary energy as a function of misorientation at more than ten-thousand triple junctions in <111> textured Al films. The films are 1.7 microns thick and are sputter deposited onto oxidized silicon and annealed at 450°C for 5 hours. The large data set of more than ten thousand junctions has allowed the relative boundary energies to be determined with a 2° resolution in misorientation. The strong texture allied with a nearly columnar grain structure means that almost all of the boundaries have tilt character and a common <111> axis. The experimental energies are compared with energies computed with atomistic (molecular dynamics) simulations of migrating boundaries in bi-crystals. A potential appropriate to Al is used. Good agreement between the experimental and computed energies is found with cusp-shaped minima at the same locations.

3:30 PM Break

4:00 PM
Effect of Heat Treatment on Texture of Zirconium Oxide Thin Film Grown on Zr-2.5Nb Substrates: Jianlong Lin[1]; Hualong Li[1]; *Jerzy A. Szpunar*[1]; [1]McGill University, Dept. of Metals & Matls. Engrg., M.H. Wong Bldg., 3610 Univ. St., Montreal, PQ H3A 2B2 Canada

Zirconium oxide thin film grown on Zr-2.5Nb alloy substrates acts as a barrier against further corrosion and hydrogen permeation in nuclear reactors. The effect of texture of zirconium substrates on the development of the texture of the oxide thin film was investigated. Heat treatment was applied to Zr-2.5Nb substrates in order to modify the microstructure and texture of the substrates. Texture of the substrates and the oxide film was measured and ODF was calculated from the measured pole figures data. The results show that the texture of the oxide film is affected by the texture of substrates. A study was undertaken to investigate the oxidation kinetics in 500°/C air environment on heat treated substrates and as-received Zr-2.5Nb substrate. It is found that the texture of the oxide film is related to the texture of the substrates and oxidation kinetics of the Zr-2.5Nb alloy is affected by the heat treatment history on the substrates and oxide microstructure and texture. SEM was employed to investigate the substrates and oxide morphology. The result obtained indicated that the morphology of the oxide film is related to the amount of ?Ò-phase in the substrates.

4:20 PM
Process-Microstructure Relationship of AlN Layers Produced by Reactive Magnetron Sputtering: Ulises Figueroa[1]; *Olimpia Salas*[1]; Joaquín Oseguera[1]; [1]ITES-CEM, Ingeniería Mecánica y Mecatrónica, Carretera a Lago de Guadalupe km. 3.5, Atizapán, México 52926 México

AlN PVD layers deposited on glass substrates by reactive magnetron sputtering under various conditions were investigated. The main processing variables were: gas mixture working pressure and substrate to target distance. The resulting films were analyzed by Scanning Electron Microscopy + Energy Dispersive x-ray Microanalysis, x-ray diffractometry and Transmission Electron Microscopy. Observations indicate that the characteristics of these films: residual stress, growth morphology and surface roughness are influenced mainly by working pressure and target to substrate distance.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Monday PM Room: 3003
February 14, 2005 Location: Moscone West Convention Center

Session Chairs: Perry H. Leo, University of Minnesota, Aeros. Engrg., Minneapolis, MN 55408 USA; Yu U. Wang, Virginia Tech, MSE, Blacksburg, VA 24061 USA

2:00 PM Opening Remarks

2:05 PM Invited
Nanoscale Phenomena in Synthetic Functional Oxide Heterostructures: *R. Ramesh*[1]; [1]University of California, Dept. of Matls. Sci. & Engrg. & Dept. of Physics, Berkeley, CA 94720 USA

In 1990, I started a basic science program at Bellcore that was aimed at creating model perovskite oxide heterostructures using ferroelectrics as a case study. Our original goal was to study the rather enigmatic problem of polarization fatigue using epitaxial films as model systems. However, in the process of doing so, we discovered a novel solution to this problem, namely the use of conducting oxide electrodes as contacts rather than conventional metals such as Pt. The key here, of course, is the atomic scale microstructure of electrical interfaces in the ferroelectric capacitor. A considerable amount of intellectual property based on this invention has been generated and is currently being licensed for development and manufacture. Over the past several years at Maryland, we have focused considerable effort on understanding the growth and characterization of functional oxide thin films and heterostructures, specifically ferroelectric, dielectric and magnetic perovskites. Using both epitaxial and polycrystalline materials on a variety of substrates as test vehicles, we have been carrying out systematic studies on the effect of composition, point defect chemistry, strain and processing variables on the microstructure and physical properties. One novel aspect of our work is the combined use of focused ion beam milling and scanning force microscopy techniques to understand the influence of film microstructure on the relevant properties at the nanoscale, specifically domains in epitaxial films and mapping of their dynamics at the nanoscale using scanned force microscopy. This, in conjunction with the implementation of a variety of novel probes, including scanning microwave microscopy, femtosecond optical probes of polarization dynamics, as well as a comprehensive theoretical treatment (both first principles and continuum mechanics) has provided us with an interdisciplinary approach to understanding complex dynamical phenomena in these materials. I will focus on some of our recent observations on nanoscale phenomena in ferroelectric thin films using scanned probes. In this presentation, I will describe some possible areas where fundamental measurements in conjunction with theoretical studies and modeling will enable a better understanding of the complex phenomena involved in these materials, especially the role of structural, chemical and functional interfaces (such as domain walls). The work at Maryland is supported by the NSF-MRSEC.

2:30 PM Invited
The Morphology and Directed Self Assembly of Quantum Dots on Surfaces: O. Shklyaev[2]; M. Beck[1]; K. Thornton[3]; M. D. Asta[1]; M. J. Miksis[2]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Engrg., 2220 Campus Dr., Cook Hall, Evanston, IL 60208 USA; [2]Northwestern University, Engrg. Sci. & Appl. Math., Evanston, IL 60208 USA; [3]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI USA

Armen Katchaturyan has had a long interest in the effects of stress on the evolution of interfaces. Here we discuss the important role of elastic stress on the evolution of island morphology, or quantum dots, on surfaces. We first discuss the formation of a Ge island on a Si substrate. We combine first-principles calculations of the stress dependence of the Ge (105) and Si (100) surface energies along with an analytical formulation of the elastic strain energy of pyramidal islands. We find that the dependence of the surface energy on strain plays an unexpectedly large role in the energetics of island formation. Directed self assembly is a promising route to controlling the location of islands on surfaces during heteroepitaxy. The results of three-dimensional phase field calculations of the directed self assembly on templated substrates with various morphologies will be given.

2:55 PM Invited
Modeling Microstructure Formation Using Phase Field Crystals: *Ken R. Elder*[1]; [1]Oakland University, Physics, Rochester, MI 48309 USA

The vast majority of naturally occurring or man-made solids are not in equilibrium and contain complex spatial structures on nanometer, micron or millimeter length scales. This is particularly important since these morphologies often determine the mechanical, electrical and optical properties of the material. Elastic and plastic deformations frequently have a significant impact on the nature of the morphologies, but are difficult to incorporate in theoretical treatments. In this talk I would like to discuss the use of phase field crystals to model elastic and plastic deformations in microstructure formation in pure and binary systems. For illustrative purposes a number of applications will be considered including liquid phase epitaxial growth, spinodal decomposition, eutectic solidification, dendritic growth and material hardness.

3:20 PM
Long-Time Dynamics of Biaxially Stressed Solid Surfaces: *Jerome Paret*[1]; [1]L2MP - CNRS UMR 6137, Faculté des Sciences de St. Jerome, Case 142, Marseille 13397 France

Using a phase-field model including strain fields, we numerically investigate the dynamics of a biaxially stressed solid surface. A multigrid algorithm is used to solve the elastic part of the problem. Its efficiency allows us to explore for the first time the late stages of the full 3D Grinfeld instability. Recent analytical predictions [P. Berger et al., Phys. Rev. Lett. 90, 176103 (2003)] regarding stability and selection of patterns are confirmed. It appears that, in presence of a large scale stabilization mechanism, the system reaches an equilibrium state corresponding to a non-trivial striped pattern.

3:35 PM Break

4:00 PM Invited
Numerical Simulations of Pattern-Directed Phase Decomposition in Stressed Films: *S. M. Wise*[1]; J. S. Lowengrub[1]; J. S. Kim[1]; W. C. Johnson[2]; [1]University of California, Math. Dept., Irvine, CA 92697-3875 USA; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA USA

A Cahn-Hilliard evolution equation possessing a source term is employed to study the morphological evolution of a strained heteroepitaxial thin film, during continuous mass deposition, on a substrate with an embedded coherent island. Both the elastic properties and the surface energy of the film are anisotropic. A sophisticated multigrid method and an implicit time integration scheme are combined to make an efficient numerical method, one which enables numerically tractable computation in both two and three dimensions. Herein we present preliminary two-dimensional results of our work showing feasibility of both the model and numerical method. This work is supported by the U.S. National Science Foundation through the Center for the Design of Nanoscopic Materials.

4:25 PM
Modeling of Self-Assembling Nano-Structures in Thin Constrained Layers.: *Andrei Artemev*[1]; Alexander L. Roytburd[2]; Julia Slutsker[3]; [1]Carleton University, Mech. & Aeros. Engrg., 1125 Colonel By Dr., Ottawa, ON K1S 5B6 Canada; [2]University of Maryland, Matls. Sci. & Engrg., College Park, MD 20742 USA; [3]National Institute of Standards & Technology, Gaithersburg, MD 20899 USA

The formation of patterns produced by misfitting coherent domains in thin constrained layers was studied using the phase-field method based on microelasticity theory. Simulation was performed for (i) multivariant systems in which misfit strain can be accommodated by assembling domains of different orientation variants and (ii) for monovariant systems. Different types of regular patterns were obtained including columnar, striped, labyrinth, and lattice patterns. The effects of thickness of polydomain layer, misfit between domains, and misfit with monodomain constraining layer on the patterns were studied. The structure maps illustrating conditions at which different types of structure can be obtained were produced. The results of computer

simulation were compared with simplified analytical models of domain structures in constrained layer.

4:50 PM
Theory and Modeling of Spinodal Decomposition in Constrained Films: *Julia Slutsker*[1]; Andrei Artemev[2]; Alexander L. Roytburd[3]; [1]National Institute of Standards & Technology, Matls. Sci. & Engrg., 100 Bureau Dr., Gaithersburg, MD 20899 USA; [2]Carleton University, Mech. & Aeros., Ottawa Canada; [3]University of Maryland, Matls. Sci. & Engrg., Coll. Park 20742 USA

The theory and phase field modeling is developed for analysis of the compositional and structure transformations in constrained layer. The equation of TDGL is used for simulation of a structural transformation, and Cahn-Hillard equation is used for simulation of a concentration decomposition. The initial state in both cases is unstable and decomposition of an uniform film results in formation of an equilibrium two- phase mixture consisting of the phases with different values of the order parameter or concentrations. Period and morphology of two-phase structure depend on the thickness of the film and misfit between film and substrate layer. The results of the simulation are compared with experimental observations in multiferroic nano-structures and polydomain structures of ferroelectrics.

5:05 PM
Next-to-Nearest Neighbor Interaction and Equilibrium Properties of Steps on Kossel Crystal Vicinal Surface: Vasiliy B. Korsakov[1]; *Robert A. Suris*[1]; [1]A.F. Ioffe Physico-Technical Institute, Polytekhnicheskaya 26, St. Petersburg 194021 Russia

The simplest discrete model of monoatomic step on crystal surface is the step on (100)-surface of cubic Kossel crystal, where atoms interact only with its nearest and next-to-nearest neighbors (NN and NNN respectively). The complete analytical description of such a step was given by Burton, Cabrera and Frank (BCF) more than 50 years ago. The BCF approach is based on the kink statistics formalism and, in principle, completely describes equilibrium properties of a step without "overhangs". Unfortunately, in general case of arbitrary step direction this description is quite cumbersome and does not allow to "feel" step properties. We investigate steps, inclined relative to [10] direction, in particularly, [11]-step. We show that if inclination is sufficiently large, one can neglect not only overhangs, but also "negative" kinks. The prohibition of negative kinks radically simplify BCF formalism and allow to obtain essential properties of "inclined" steps in simple analytical form. We show that equilibrium structure of such steps is determined only by the interaction between NNN. If this interaction is repulsive, the step has saw-like shape, while attractive interaction leads to straightening of the step. Kink size distribution, step diffusivity and step free energy are obtained as functions of step inclination and NNN interaction energy. Also we extend BCF formalism to the case of arbitrary, non-pairwise interactions, if only these interactions are limited to the nearest and next-to-nearest neighbors.

5:20 PM
Diffusion in Crystalline Composition-Modulated Films: *Alan F. Jankowski*[1]; [1]University of California/Lawrence Livermore National Laboratory, CMS - Matls. Sci., PO Box 808, MS L-352, Livermore, CA 94551-9900 USA

The diffusivity in alloy systems at low temperatures is determined using composition-modulated structures. An artificial concentration wave is produced by alternating a deposition of the alloy elements. A quantification of the interdiffusivity coefficient is determined by analyzing the decay of the composition fluctuation, that is, the static concentration wave using the microscopic theory of diffusion. As it is customary to assume that there is a linear relationship between ln D and 1/T over a wide range of temperature, the bulk diffusion coefficient represents the long wavelength approximation of the interdiffusivity. The dependency of interdiffusivity on structure is found in general expressions that account for the specific periodicity and growth orientation of the multilayer structure. The kinetics are quantified by analyzing changes in the composition fluctuation through x-ray scattering measurements. In addition to the examination of single-phase crystalline systems as Cu-Ni and Cr-Ti, the approach is now developed to assess two-phase layered systems. Specifically, as in Ni-(Cr,Mo) where a face-body centered cubic combination form a pseudo epitaxial system.

The Langdon Symposium: Flow and Forming of Crystalline Materials: High Temperature Deformation Including Superplasticity

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Monday PM — Room: 3024
February 14, 2005 — Location: Moscone West Convention Center

Session Chairs: Eric M. Taleff, University of Texas, Dept. of Mechl. Engrg., Austin, TX 78712-0292 USA; Tadao Watanabe, Tohoku University, Dept. of Nanomech., Sendai, Miyagi 980-8579 Japan; Farghalli A. Mohamed, University of California, Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA; Norio Furushiro, Osaka University, Internatl. Student Ctr. & Dept of Matls. Sci. & Engrg., Grad. Sch. of Engrg., Suita, Osaka 565-9871 Japan

2:00 PM
Effect of Nano-Dispersion Particles on Superplastic Flow in Zn-22% Al: *Farghalli A. Mohamed*[1]; Yuwei Xun[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., 916 Engrg. Tower Bldg., Irvine, CA 92697-2575 USA

A detailed creep investigation was performed on Zn-22%Al in which nano-scale dispersion particles were introduced by cryomilling. The objective of the investigation was to determine the effect of these dispersion particles on creep and microstructure in region I and region II of the sigmoidal plot between stress and strain rate, which was previously reported for the alloy. The results show that while the particles have no significant effect on the sigmoidal plot, their presence influences several characteristics in region I. An examination of creep microstructures in region II and region I reveals that only some grains contain dislocations, many of which are attached to the dispersion particles. On the basis of these results and other available information, it is concluded that region I and region II are controlled by the same deformation process, in which the sliding of groups of grains is accommodated by dislocation motion in the blocking grains.

2:15 PM
Elongation to Failure in the Model of Cooperative Grain Boundary Sliding: *Oscar Akramovich Kaibyshev*[1]; Anatoliy Ivanovich Pshenichnyuk[1]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin str., Ufa, Bashkortostan 450001 Russia

In recent papers[1,2] we presented the model of the superplastic deformation (SPD), which postulates the cooperative grain boundary sliding (CGBS) as a principal mechanism of deformation. The model shows that the SPD occupies the interval of s (or) values between diffusion and dislocation creep. However, the SPD is not a mere combination of these processes but presents a separate phenomenon and is realised by a specific deformation mechanism - the CGBS. The stresses, which set the lower and the upper limits of the SPD stress interval, are not imposed artificially but are derived from the condition of CGBS bands development. The threshold stress depends on the temperature, mean grain size, variance of size distribution and the value of the percolation level. The model adequately describes s- dependencies for the SPD in real materials. On the base of those results we simulated the elongation to failure. The calculation is based on the concept of cooperative grain-boundary sliding and consists of solving the equation: where 2r (z,t) is the cross-sectional width at time t and a space coordinate z (-l(t)<z<l(t)), VB is the rate of shear in a separate band, n (z,t) is a random function describing the spatial distribution of the CGBS bands along the specimen at time t. Nonuniformity of CGBS band spatial distribution, which was created randomly during deformation, leads to flow localization on macro-level. An analysis of the statistical properties of the random function n(z,t) allows us to define deformation to failure and its dependence on the strain-rate, mean grain size

and samples geometry. [1]O.A. Kaibyshev, A.I. Pshenichnyuk, V.V. Astanin. Acta mater., 46(1998)4911. [2]A.I.Pshenichnyuk, O.A. Kaibyshev, V.V. Astanin. Phil. Mag. A79(1999)329.

2:30 PM
Cavitation Failure in Superplasticity: *Atul H. Chokshi*[1]; [1]Indian Institute of Science, Metall. Dept., Bangalore 560012 India

Although most early studies on superplasticity examined failure largely in terms of stability of plastic flow against flow localization, it is now recognized that concurrent cavitation plays a dominant role in the failure of many superplastic alloys. Professor Langdon was amongst the first to recognize the importance of cavitation in the failure of superplastic materials. This paper will briefly review various important aspects on cavity nucleation, growth and interlinkage in a wide range of superplastic materials from conventional microduplex metallic alloys to ceramics, and it will highlight techniques developed to retard cavitation during industrial superplastic forming.

2:45 PM
Material Modelling Data for Superplastic Forming Optimisation: *Norman Ridley*[1]; Pete S. Bate[1]; Baoliang Zhang[1]; [1]University of Manchester, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK

Optimisation of the superplastic forming process involves the determination of the minimum forming time within the constraints of product characteristics such as thickness distribution and cavitation level. However, superplastic forming is a non-linear system, with the material behaviour giving a significant contribution to that non-linearity. For example, dynamic grain growth gives useful strain hardening but can ultimately reduce strain rate sensitivity, and conversely forming with a decreasing strain rate can enhance ductility. Superplastic materials can also exhibit plastic anisotropy. The results of tests on two commercial superplastic alloys, AA5083 and AA7475, have been used to provide data for material modelling. Simple power law descriptions of the mechanical behaviour were not adequate, and constitutive relationships for these materials are presented and discussed.

3:00 PM
Superplasticity in Ceramics with Intergranular Phases: *Martha L. Mecartney*[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92697-2575 USA

Highly deformable, fine grain oxide ceramics can be created using intergranular phases to limit grain growth and promote superplastic behavior. Cubic zirconia (8 mol% Y2O3 stabilized ZrO2, 8Y-CSZ) can be deformed over 500% in tension at 1430°C with the addition of 5 wt.% intergranular silica. A mixture of 5 wt.% SiO2 and 1 wt.% Al2O3 in 8Y-CSZ can achieve strain rates of 0.0002/sec at 1200° C and 30 MPa stress. Alumina based composites can achieve high strain rates of 0.01/sec (1500°C, 50 MPa stress) with the combined addition of intergranular silica and zirconia. Al2O3 composites with 10 vol.% ZrO2 and 0.5 mol% of the intergranular phase CuO/Mn3O4 have demonstrated deformation rates of 0.001 at 1300°C under 30 MPa stress. This talk will summarize how intergranular phases can be used to achieve the goal of high strain rate superplasticity in ceramics.

3:15 PM
A Unified Tensile Test to Define the Superplastic Properties of Materials: *Peter N. Comley*[1]; [1]The Boeing Company, PO Box 3707, m/c 5K-63, Seattle, WA 98390 USA

The testing methods and presentation of SPF data have encompassed a wide spectrum of techniques, and results can differ from one test to another. In particular, distortion of the coupon during testing, the difficulty of measuring strain in the gauge length, the translation of constant crosshead velocity to strain rate, and other factors, have all disguised or distorted the true superplastic properties of materials. This paper proposes a specification to define the correct superplastic properties of any material. It addresses the shape of the coupon to minimize errors of measurement, the application of constant strain rate, the generation of true stress and true strain, the format that data should be presented, and the generation of "m" and "n" values. It is hoped that this could become the basis of a world standard SPF test that both academic and industrial institutions can use.

3:30 PM
A Comparative Study of Cavitation Characteristics in Superplastic 5083 Al and AZ31 Mg Alloys: Yasumasa Chino[1]; *Hajime Iwasaki*[1]; Mamoru Mabuchi[2]; [1]National Institute of Advanced Industrial Science and Technology, Matls. Rsch. Inst. for Sustainable Dvlp., 2266-98 Shimo-Shidami, Moriyama-ku, Nagoya 463-8560 Japan; [2]Kyoto University, Dept. of Energy Sci. & Tech., Kyoto 606-8501 Japan

The plasticity-controlled growth rate of cavities during superplastic deformation was statistically investigated for 5083 Al alloy and AZ31 Mg alloy. When the cavity growth rate was evaluated on the basis of macroscopic strain calculated using the displacement of the specimen, the growth rate for the Al alloy was larger than that for the Mg alloy. However, the growth rate of the Al alloy was in agreement with that of the Mg alloy when the cavity growth rate was evaluated on the basis of the microscopic strain due to grain boundary sliding. The results obtained lead to two conclusions: (1) the rate of cavity growth is not affected by the kind of materials, that is, the nature of the grain boundary and (2) the microscopic strain due to grain boundary sliding should be used to exactly evaluate the rate of cavity growth for superplastic deformation.

3:45 PM
Change in Crystallographic Distribution During Superplastic Deformation in an Al-Zn-Mg-Cu Alloy: Yoshimasa Takayama[2]; *Norio Furushiro*[1]; [1]Osaka University, Internatl. Student Ctr. & Dept. of Matls. Sci. & Engrg., Grad. Sch. of Engrg., 1-1 Yamadaoka, Suita, Osaka 565-9871 Japan; [2]Utsunomiya University, Dept. of Mechl. Sys. Engrg., 7-1-2, Yoto, Utsunomiya, Tochigi 321-8585 Japan

The change in crystallographic orientation distribution during superplastic deformation in an Al-Zn-Mg-Cu alloy has been investigated in order to reveal the deformation mechanism. The well known strain rate dependence of the deformation behavior was examined by SEM/EBSP (scanning electron microscope/electron back scatter diffraction patter) analysis. Fraction of low angle grain boundaries increased after deformation at the high strain rate, while fraction of random boundaries was high in the specimen deformed at the low strain rate. Randomization of the initial texture was also found during deformation at the low strain rate. Further, the intragranular misorientation, grain boundary misorientation and local orientation are analyzed in detail to discuss the accommodation process.

4:00 PM Break

4:15 PM
High Temperature Deformation and Fracture Controlled by Grain Boundaries: *Tadao Watanabe*[1]; Sadahiro Tsurekawa[1]; Shigeaki Kobayashi[2]; Shin-ichi Yamaura[1]; [1]Tohoku University, Dept. of Nanomech., Sendai, Miyagi 980-8579 Japan; [2]Ashikaga Institute of Technology, Dept. of Mechl. Engrg., Faculty of Engrg., Ashikaga, Tochi Japan

Grain boundaries can play important roles in deformation and fracture in bicrystals and polycrystals at high temperatures where grain boundary phenomena become dominant. It has been well established thatgrain boundary phenomena strongly depend on the type and structure of grain boundary.[1] So it is indispensable to understand how different grain boundary phenomena interact to each other to enhance or supress plastic deformation and fracture of polycrytstalline materials at high temperatures. This paper is an overview of recent studies of structure-dependent grain boundary phenomena which are involved in high temperature deformation and fracture in polycrystalline materials. Recent applicationsof grain boundary engineering to the control of intergranular brittleness,[2] oxidation brittleness[3] and also development of superplasticity[4] will be discussed. [1]T.Watanabe, Metall.Trans., A14(1983),531; [2]T.Watanabe, Mater.Sci.Eng., A166(1993), 11; [3]S.Yamaura, S.Tsurekawa and T.Watanabe, Mater.Trans.44(2993), 1497; [4]S.Kobaayashi, T.Yoshimura,S.Tsurekawa and T.Watanabe, Mater.Trans.,44(2003),1469.

4:30 PM
Elevated Temperature Deformation: Hot Working Amplifies Creep: *Hugh J. McQueen*[1]; Michael E. Kassner[2]; [1]Concordia University, Mechl. Engrg., 1455 Maisonneuve W., Montreal, QC H3G 1M8 Canada; [2]University of Southern California, Aeros./Mechl. Engrg., Los Angeles, CA 90089-1453 USA

Because creep research is motivated by restricting high temperature T strain ε, it generally considers rates below 0.0001/s and low ε defined by tensile failure or by minimum rate in compression. Hot working (0.01-100/s) studies aim to reduce hot strength and raise fracture ε in deformation modes extending far into steady state. While dependence of stress σ and substructure character in steady state are similarly linked to T and ε rate through dynamic recovery (DRV), the high ε permits evolution unrecognized in creep, even geometrically refining the grains to the subgrain size and aligning them into intense textures. The higher σ leads to higher misorientations that in multistage processing gives rise to static recrystallization to lower forces, improve ductility and refine structure. Although grain boundary (GB) sliding declines to less than 1% of total ε at high rates, it is the inherent cause of failure; nevertheless, accommodation of sliding at triple junctions by DRV-enhanced grain flow provides torsional ε exceeding 100.

In Al-5Mg, solute drag on dislocations constrains subgrain formation to larger ε with sizes smaller and stresses higher by a factor of 4; yet elongations are similarly multiplied due to augmented strain rate sensitivity. In low stacking fault energy metals, dynamic recystallization refines grains, lowers strength and raises ductility as GB migration lowers σ concentrations at triple junctions and retards cracking. In thermomechanical processing, substuctures can be developed for strength with toughness, creep resistance in superalloys, or conversion to superplastic behavior.

4:45 PM

Effect of Thermo-Mechanically Induced Microstructural Coarsening on the Evolution of Creep Response of SnAg-Based Microelectonic Solders: Robert A. Marks[2]; Deng Pan[1]; Susheel Jadhav[3]; *Indranath Dutta*[1]; [1]Naval Postgraduate School, Ctr. for Matls. Sci. & Engrg., Dept. of Mechl. & Astronautical Engrg., 700 Dyer Rd., Monterey, CA 93943 USA; [2]University of California, Dept. of Matls. Sci. & Engrg., Berkeley, CA 94720 USA; [3]INTEL Corporation, Assembly Tech. Dvlp., 5000 W. Chandler Blvd., CH5-165, Chandler, AZ 85226 USA

Microelectronic solder joints, which serve as both electrical and mechanical connections between a chip and various parts of a package, are subjected to aggressive thermo-mechanical cycling (TMC) during service, with shear strains and homologous temperatures reaching up to 1 and 0.9, respectively. As a result, the creep behavior of the tiny solder joints limits the reliability of the entire microelectronic package. In addition, the microstructures of the new lead-free solders (Sn/Ag and Sn/Ag/Cu) can undergo significant in situ strain-enhanced coarsening during TMC, resulting in in-service evolution of the creep behavior of the joints. In these materials, Ag3Sn and Cu6Sn5 precipitates can serve as barriers to dislocation motion, and thus, influence creep behavior, dependent on the particle size and spacing. In this paper, the coarsening kinetics of Ag3Sn particles in Sn/Ag-based microelectronic interconnects is presented, and the results are correlated with impression creep data from individual microelectronic solder balls subjected to particle-growth anneals. Coarsening influences creep behavior in two ways. At low stresses, the creep rate increases proportionately with precipitate size. At high stresses, as proposed by Langdon, precipitate coarsening influences creep response by altering the threshold stress for particle-limited creep. Based on these observations, a creep model for solder interconnects undergoing in situ coarsening is presented. Supported by NSF, SRC and INTEL Corp.

5:00 PM

Deformation Behavior of Mg-Li Alloys at Elevated Temperatures: *Zuzanka Trojanova*[1]; Zdenek Drozd[1]; Pavel Lukac[1]; Frantisek Chmelik[1]; [1]Charles University, Dept. of Metal Physics, Ke Karlovu 5, Praha 2 12116 Czech Republic

Magnesium alloys are attractive for a large amount of applications. It is important to estimate the deformation mechanisms responsible for the deformation behaviour of Mg alloys at elevated temperatures. Experiments were conducted to evaluate the influence of the testing temperature and strain rates on the deformation behaviour. The tests were performed using Mg-4 wt. % Li (Mg-12 at. % Li) at temperatures between 293 and 473 K and at a constant but different initial strain rate. Stress relaxation tests were carried out in order to estimate parameters of a possible thermally activated process. The results show that the flow stress rapidly decreases with increasing temperature. Softening occurs at higher temperatures and it is attributed to annihilation of dislocations during deformation. The activation volume calculated from the stress relaxation test decreases with strain. This investigation demonstrates that the imposed initial strain rate influences the course of stress-strain curves.

5:15 PM

Analysis of the Effect of Si Content on the Creep Response of a Mg-5Al-Mn Alloy: Stefano Spigarelli[1]; Marcello Cabibbo[1]; Claudia Scalabroni[1]; *Enrico Evangelista*[1]; Pal Ulseth[2]; Otto Lohne[2]; [1]Universita' Politecnica delle Marche, Dept. of Mech., Via Brecce Bianche, Ancona 60131 Italy; [2]NTNU, Dept. of Matls. Tech. & Electrochmst., Trondheim N-7491 Norway

The die-cast AM50 (Mg-5Al-0.5Mn) alloy is used for the production of automotive components and other parts that require good ductility and toughness combined with reasonable tensile strength. However, this material, like many other Mg-alloys including AZ91, has a relatively low creep resistance; on the other hand, the beneficial effect of Si addition in enhancing the creep strength at low stresses, has been clearly observed by comparing the high-temperature behaviour of AZ91, AS21 (Mg-2Al-1Si) and AS41 (Mg-4Al-1Si). In this context the present study aims at investigating the effect of small Si-addition (0.3, 0.5, 0.8 and 1.5%Si) on the creep response of a AM50 alloy. Tensile tests at room temperature and constant load creep tests at 150 and 125°C were carried out on die-cast samples, without any prior-heat treatment. The effect of chemical composition was investigated by testing 5 different alloys with Mg-content increasing from 0 (base AM50 alloy) to 1.5 %. Tensile tests at room temperature indicated that the increase in Si content had a minor effect on strength, but determined a reduction of ductility, a result that was fully consistent with the data available for the conventional AM50 (yield strength 130 MPa, tensile strength 210-225 MPa, elongation 8-10%) and AS41 (yield strength 140 MPa, tensile strength 215 MPa, elongation 6%). Nevertheless, the effect of Si-content was much more pronounced at high temperature. The creep curves, irrespective of the Mg content, consisted in a well defined primary stage, followed by a short secondary region and by a short tertiary stage that led to fracture without extensive necking. A mere 0.3% addition in Mg resulted in an increase of the time to fracture from 23 to 36 h under 100 MPa at 125°C; further Mg-additions produced a substantial increase in creep strength (time to rupture 133 h in the same testing conditions), accompanied by a decrease of strain to fracture. Optical and electron microscopy studies were carried out to investigate the influence of Mg in the creep response; this effect was attributed to the precipitation of Mg2Si particles.

5:30 PM

Effect of Microstructural Variables on Creep Fatigue Resistance in Udimet 720Li: *Philippa Ann Reed*[1]; Hon Tong Pang[1]; Jeff W. Brooks[2]; [1]University of Southampton, Sch. of Engrg. Scis., Highfield, Southampton, Hants SO17 1BJ UK; [2]QinetiQ, Struct. & Matls. Ctr., Cody Tech. Park, Ively Rd., Farnborough, Hants GU14 0LX UK

An assessment of the effects of microstructure on both high temperature crack initiation and propagation of short fatigue and crrepfatigue cracks in a notch stress field in a powder metallurgy turbine disc alloy is presented. The notch was chosen to represent a similar stress concentration feature to that found in the fir-tree root fixing in a turbine disc. The assessment has been carried out on 3 microstructural variants of Udimet 720Li, where grain size and gamma prime size have been varied by sub-solvus heat treatments. Assessment of such behaviour will indicate whether current long crack lifing methodologies (for example based on Paris-type assumptions and long crack growth da/dN versus ?K data) can be extrapolated to such conditions. Comparison of air and vacuum behaviour allows the relative contributions of creep and oxidation in accelerating the fatigue processes to be elucidated.

5:45 PM

Anelastic Creep Behaviour of RR-58 Aluminum Alloy at 180°C: Phenomenological Aspects and Analysis Based on the Unbowing of Dislocation Segments: *Levi de Oliveira Bueno*[1]; [1]Universidade Federal de São Carlos, Dept. de Engenharia de Materiais, Rod. Washington Luiz, km. 235, Caixa Postal 676, São Carlos, SP 13565-905 Brazil

The strain relaxation behaviour after full stress removals during creep of RR-58 Hiduminium alloy has been observed. Creep tests were carried out at 180ºC, with stresses of 120, 170 and 230 MPa. Tests involved a series of unloading cycles after different creep periods involving primary, secondary and tertiary stages. At least two relaxation stages were observed and attempts made to interpret the results according to a model based on thermally activated unbowing of dislocation segments. A suggestion is made for the use of an integrated form of the sine hyperbolic rate equation directly with the readings of anelastic strain and time. Activation parameters are calculated an interpreted considering microstructural details of the material. The appearance of the fast and slow anelastic creep stages may be just a consequence of the high non-linearity relation existing between the line tension and the area swept by the segments during their unbowing movement.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Rapid Prototyping

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday AM — Room: 2005
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: James W. Sears, South Dakota School of Mines & Technology, Rapid City, SD 57701 USA; Jianxin Liu, Extrude Hone Corporation, ProMetal, Irwin, PA 15642 USA

8:30 AM
Supersolidus Liquid Phase Sintering of Tool Steel for Rapid Tooling: *Jianxin Liu*[1]; Howard Kuhn[1]; [1]Extrude Hone Corporation, ProMetal, 1 Industry Blvd., PO Box 1000, Irwin, PA 15642 USA

Three dimensional printing (3DP) is a solid-free form fabrication technique using metal powder as the building material. Green performs made by 3DP usually consist of less than 60 volume percent of metal powder, about 10 volume percent binder to hold metal powder together, and the remainder being pore space. Because of high porosity level a liquid phase must present in the thermal process to attain high density. In this work, supersolidus liquid phase sintering of prealloyed tool steel powder, M4-Co, was investigated. Promising experimental results and successful manufacture practice proved the M4-Co can be used successfully for rapid tooling.

8:50 AM
Development of Laser Ultasonics for Defect Detection During Laser Powder Deposition: Jason Nemeth[1]; Marvin Klien[2]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technology, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]Lasson Technologies, 6059 Bristol Pkwy., 1st Fl., Culver City, CA 90230 USA

Laser ultrasonics shows great promise for on-line monitoring of product integrity in laser powder deposition (LPD). In the LPD process, we wish to detect defects on each layer as it is being deposited. The defect information can be then used to control critical weld parameters or alert the operator of a problem requiring maintenance. In this way, defect scan then be corrected before many defective parts are produced. Laser ultrasonics uses a pulsed laser to generate an ultrasonic wave and a continuous-wave laser interferometer to detect the small surface displacement when this wave arrives at the point of detection. Laser ultrasonics is ideal for in-line measurements because there is no sensor in contact or near-contact with the work piece. In this study, we have used laser-generated surface waves to interrogate LPD parts in both stainless steel and Ti. The processing approach was optimized using simulated pores produced using blind holes.

9:10 AM
Micron Scale Deposition for Rapid Prototyping of Electronic Components: John Preheim[2]; Jacob Colvin[1]; Keith Whites[2]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technolgy, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines & Technology, Electl. & Computer Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Maskless Meso-Scale Material (M3D) is a Direct Write technology that uses nano-particles inks to build micron-scale electronic prototypes. The process can deposit most any nano-particle that can be suspended in a liquid with a viscosity of 1 to 1000 cp. Particle loading can be has high as 60%. A variety of materials have been deposited including; silver, gold, platinum, copper, rhodium, PMA, graphite epoxy, kapton, and barium titanate to name a few. The response of several electronic devices (e.g., capacitors, filters, resistors, inductors) have been characterized and compared to simulations.

9:30 AM
Structure-Property-Process Parameters Correlation in Rapid Prototyped 316L by Direct Laser Deposition: *Jyotsna Dutta Majumdar*[1]; Indranil Manna[1]; Lin Li[2]; [1]Indian Institute of Technology, Metallurgl. & Matls. Engrg., Kharagpur, W. Bengal 721302 India; [2]University of Manchester Institute of Science and Technology, Mech., Mfg. & Aeros. Engrg., Manchester M60 1QD UK

Laser assisted fabrication is a technique that utilizes high-power lasers to melt materials in the form of a powder or wire and controlled deposition of the molten metal in a layer-by layer fashion to induce a pre-determined shape. In the present study, 316L stainless steel has been fabricated by laser assisted direct metal deposition technique with a high power continuous wave Diode laser using Ar as shrouding environment. The main process variables were, applied power, and scan speed. Powder feed rate was maintained constant to 20 mg/s. After fabrication, the surface and cross section of the fabricated components were characterized using optical and scanning electron microscopy. X-ray diffraction study and energy dispersive spectroscopic analysis. The mechanical and electrochemical properties of the fabricated component were evaluated. Finally, a detailed study of the influence of process parameters on the characteristics and mechanical properties of the fabricated component has been undertaken to optimize the processing zone.

9:50 AM
Multi-Material Extrusion for Fabrication of Artificial Teeth: Jiwen Wang[1]; *Leon L. Shaw*[1]; Thomas B. Cameron[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., Inst. of Matls. Sci., 97 N. Eagleville Rd., U3136, Storrs, CT 06269 USA; [2]Dentsply Ceramco, Burlington, NJ 08016 USA

A solid freeform fabrication (SFF) based technique, called multi-material slurry extrusion (M2SE), has been developed to fabricate artificial teeth made of dental porcelains and silver metals. The rheological and extrusion behavior of dental porcelain and silver slurries are controlled by the solids loading, pH value, and drying speed of the slurries after delivery. Dental crowns and solid teeth made of dental porcelains alone and dental porcelains plus silver metals are demonstrated using the M2SE method. Issues related to fabrication of dental units, such as slumping, overhang angles, bridging capability, material overfilling, generation of porosity, resistance of the green body to cracking under the weight of the top layers and dimensional accuracy, are discussed. The effects of the rheological behavior and extrusion parameters on these issues are investigated and the solutions are proposed.

10:10 AM Break

10:25 AM
Microstructure and Mechanical Properties of Spray-Formed H13 Tooling: *Yaojun Lin*[1]; Kevin M. McHugh[2]; Young-Soo Park[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., Idaho Falls, ID 83415 USA

Spray forming is a recently developed technology that has been demonstrated to successfully fabricate molds and dies. During this approach, atomized droplets are directed towards a pre-designed pattern where they accurately capture features of the pattern during solidification to form the desired mold or die. Compared with the conventional techniques for mold/die production, a series of delicate machining, grinding, and polishing steps can be eliminated in spray-formed tooling. Furthermore, rapid solidification associated with spray deposition can produce metastable phases in the deposited tooling material. As a result, die properties such as hardness and toughness can be tailored via subsequent aging (tempering) heat treatment. In contrast, conventional austenitization/quench/temper heat treatment is required in conventional mold/die making techniques. Therefore, spray-formed tooling will save energy, production cost and production time and avoid mold/die distortion during heat treatment. This paper reports the microstructures and mechanical properties of H13 alloy samples processed via spray-formed tooling.

10:45 AM
Fabrication of PEM Fuel Cell Bipolar Plates by SLS: *Ssuwei Chen*[1]; David Bourell[1]; [1]University of Texas, Lab. for Freeform Fabri-

cation, TX Matls. Inst., 1 Univ. Sta., MC C2200, Austin, TX 78712-0292 USA

One of the barriers to commercialization of Polymer Exchange Membrane (PEM) fuel cells is economic fabrication of the bipolar plate. Manufacturing of pure graphite bipolar plates with machined flow fields is both time consuming and costly. The advantage of applying Selective Laser Sintering (SLS) for bipolar plate fabrication is that integrated cooling channel can be simultaneously formed on the central surfaces of the plate rather than by gluing two half plates back-to-back as is used in many other techniques. Requirements such as electrical conductivity, gas impermeability, etc. for bipolar plates are reported as affected by material selection and fabrication process.

11:05 AM
Application of In-Situ Melt Pool Sensor and Z-Height Control System to Laser Engineered Net Shaping (LENS™) Process: *John E. Smugeresky*[1]; Baolong Zheng[2]; Leonardo Ajdelsztajn[2]; Yizhang Zhou[2]; Julie M. Schoenung[2]; Enrique J. Lavernia[2]; [1]Sandia National Laboratories, Dept. 8724, Livermore, CA USA; [2]University of California, Cheml. Engrg. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA

Direct fabrication of metallic parts with LENS™ is achieved by feeding powders to the melt pool created by a high-powered laser beam on a substrate. During LENS™ deposition, both a constant melt pool size and a constant deposit height are of importance, in particular for microstructural homogeneity and dimension accuracy of the parts. Using a melt pool sensor (MPS) to maintain the same melt pool size, and a Z-height control system to retain the same deposit height are seen as positive measures to insure the most uniform features. The objective of this work is to establish the correlation of process parameters to the geometrical features of the deposit microstructure and dimensional accuracy of the deposited parts, with and without MPS and Z-height control system. The microstructure characteristics and micro-hardness of the laser deposited parts, as a function of type of control environment, are reported. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000. Work at UC Davis is supported by the National Science Foundation under grant number DMI-0423695.

11:25 AM
Characterization of Interfaces Between LENS® Deposited and Cast or Wrought 304L Stainless Steel: *J. E. Smugeresky*[1]; C. V. Robino[2]; D. D. Gill[2]; M. F. Harris[2]; M. L. Griffith[2]; [1]Sandia National Laboratories, Livermore, CA 94551 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA

The repair and/or modification of expensive metal hardware using LENS® (Laser Engineered Net Shaping™) instead of complete refabrication has the potential for huge savings in cost and delivery time for transportation industries. As such, the interface between LENS deposited material and the underlying base material must be metallurgically sound and possess mechanical properties equal to/or better than the base material. A metallurgical evaluation was conducted on thirteen different such interface configurations of deposited material and the underlying base material, representing a spectrum of deposition conditions from complete part build, to hybrid substrate-LENS builds, to repair/ modification builds. Tensile bars, with the interface at the center of the gauge length were pulled to failure to determine mechanical properties and characterize the fracture behavior. Good mechanical properties and full density were observed for all configurations. When tested to failure, fracture occurred by ductile micro-void coalescence. Based on microstructure charactereization, the repair and hybrid interfaces showed the same metallurgical integrity and properties as monolithic LENS deposits. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Forming

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday AM — Room: 2009
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: M. R. Stoudt, National Institute of Standards and Technology, Matls. Performance Grp., Gaithersburg, MD 20899-8553 USA; Glenn S. Daehn, Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

8:30 AM Invited
Strategies for Sheet Metal Forming Using Mechanical Impulse: *Glenn S. Daehn*[1]; Manish Kamal[1]; Mala Seth[1]; Jianhui Shang[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

Presently sheet metal forming is almost exclusively thought of, performed and analyzed in a manner where "inertial forces are negligible". The main emphases of this presentation are: 1) There are methods that can reliably, predictably and robustly impart dramatic velocities and acceleration levels in sheet metal forming, 2) High velocity and acceleration can dramatically improve the stability of metal flow improving formability and resistance to wrinkling, 3) Acceleration due to impact can be used emboss textures on the surfaces of metals and strongly modify springback, and 4) Elegant methods exist for combining these high velocity forming technologies with more traditional metal forming methods. When used in a strategic manner, impulsive metal forming can treat many persistent problems in sheet metal forming. This overview will also highlight two focus areas. First, is the design and operation of an electromagnetic coil configuration that is very efficient, robust and gives a very uniform electromagnetic pre ssure distribution. This coil is especially useful for forming nominally flat components with fine surface features. Second, we will highlight how formability can be controlled and improved through impulsive means. Improved formability is a compelling reason to consider high velocity forming in sheet metal working.

8:55 AM
Electrohydraulic Forming of Automotive Panels: *Sergey F. Golovashchenko*[1]; Viacheslav S. Mamutov[2]; [1]Ford Motor Company, Mfg. & Processes, 2101 Village Rd., Ford Rsch. & Advd. Engrg., Dearborn, MI 48124 USA; [2]St. Petersburg Polytechnical University, 29 Politekhnitcheskaya st, St. Petersburg 195251 Russia

In this presentation, results on sheet metal forming using pulsed electrohydraulic technology will be discussed. Pulsed electrohydraulic forming is an electrodynamic process, based upon high-voltage discharge of capacitors between two electrodes positioned in a fluid-filled chamber. This technology combines the advantages of conventional hydroforming and high-rate forming processes. Compared to traditional forming in steel dies, electrohydraulic forming allows more uniform distribution of strains, makes wider the formability window, and reduces springback of parts. Parameters of electrohydraulic discharge and experimental results on formability of outer body skin panel material will be reported.

9:15 AM
Pulsed Electromagnetic Forming of Aluminum Body Panels: *Sergey F. Golovashchenko*[1]; Nickolas M. Bessonov[2]; Richard W. Davies[3]; [1]Ford Motor Company, Mfg. & Processes Dept., 2101 Village Rd., Dearborn, MI 48124 USA; [2]Institute of Problems of Mechanical Engineering, 61 Bolshoy prospekt V.O., St. Petersburg 199178 Russia;

[3]Pacific Northwestern National Laboratory, Energy Matls., 902 Batelle Blvd. K2-03, Richland, WA 99352 USA

Pulsed electromagnetic forming is based on high-voltage discharge of capacitors through a coil. This pulse generates an intense transient magnetic field in the coil that applies pressure to the metal work-piece to do the work. In theoretical analysis, the propagation of electromagnetic field is defined by quasi-stationary Maxwell equations transformed in Lagrangian form. Dynamics of elastic-plastic deformation is modeled using solid mechanics equation of motion and modified theory of elastic-plastic flow. Energy conservation law is employed for simulation of heat transfer, which is important to define the appropriate stamping rate without overheating the coil. Results of numerical simulation and experimental data will be presented. Methodology of testing durability of coils for pulsed electromagnetic forming will be also discussed.

9:35 AM

Product and Process Development with Local Thermal Manipulation of Aluminum: *Børge Iver Bjørneklett*[1]; [1]Hydro Aluminium, Auto. Struct., Fabrikkvei 1, PB 15, Raufoss N-2831 Norway

The present paper describes a concept that introduces a new dimension in design and fabrication of components and structures in aluminum for the transportation sector. By intensified heating in short pulses it is possible to manipulate the mechanical properties of aluminum alloys at accurate positions. The basic principle of local thermal manipulation is recently applied in different areas e.g. crash absorbing members, in new forming techniques and assembly methods. Innovations in this field have spurred new product designs and exiting possibilities for through process simulation. Practical examples and conceptual studies are shown for LTM in fabrication of various automotive aluminum components.

9:55 AM

Material Characterisation for Laser-Assisted Sheet Metal Hydroforming: Heinz Haferkamp[1]; Jens Bunte[1]; *Lars Engelbrecht*[1]; [1]Laser Zentrum Hannover, Matls. & Processes, Hollerithallee 8, 30419 Hannover Germany

Localised laser heating used during sheet metal hydroforming processes should reduce the work pressures necessary for hydroforming presses. By reducing the yield strength and the strain hardening using local heating, small form elements like creases can be formed at very low pressures of 2 MPa, whereas cold forming requires pressures which are 20-50 times higher. Besides the forming temperature and work pressure, temperature distribution is very important and can be modified using a special beam forming optics. This leads to improvements in the plastic deformation distribution. A significant improvement of the material's formability is shown by forming limit curves (FLC) which were generated using the bulge-test. Moreover, the mechanical properties and the grain structure of the form elements generated were determined. All investigations were carried out for a deep drawing steel, a 5182 aluminium alloy and an AZ31 magnesium alloy.

10:15 AM Break

10:30 AM

Evaluation of Deformation-Induced Surface Morphologies Generated in an Aluminum Alloy Sheet: *M. R. Stoudt*[1]; J. B. Hubbard[1]; J. Liu[1]; [1]National Institute of Standards and Technology, Matls. Performance Grp., 100 Bureau Dr., Stop 8553, Gaithersburg, MD 20899-8553 USA

Numeric predictions of mechanical behavior and friction during forming are central to the automotive design process; yet presently, significant discrepancies exist between what is predicted by numeric methods and what is measured on real surfaces. These discrepancies indicate that a better understanding of the fundamental relationships between deformation and the ensuing roughness at the free surface is required for better predictive accuracy. Most of the available surface roughness data are derived from assessments that compress the complex surface information into quantities that are too coarse with respect to the length scale of the relevant features. Furthermore, these assessments frequently contain tacit assumptions about the distributions of the surface features within the roughness data that may further degrade the precision. The results of a rigorous statistical protocol characterizing the roughening of a 6xxx series aluminum sheet will be discussed with emphasis on development of a better approach that should produce a robust generic model of roughening behavior.

10:50 AM

Microstructure Based Modelling of Al-Mg-Si Alloys in Development of Local Heating Processes for Automotive Structures: *Hallvard G. Fjaer*[1]; Børge I. Bjørneklett[2]; Ole R. Myhr[2]; [1]Institute for Energy Technology, Dept. of Process & Fluid Flow Tech., PO Box 40, NO-2027 Kjeller Norway; [2]Hydro Aluminium Structures Raufoss AS, Product & Process Dvlp., PO Box 15, NO-2831 Raufoss Norway

This work addresses the exploitation of local heating on automotive parts of age hardening aluminium alloys. Local heating can be applied for many purposes, e.g. correction of geometrical shapes, reduction of the flow stress in forming operations, and manipulation of the yield strength and the ultimate strain (i.e. local material design). One particularly promising application of local material design is in controlling the buckling behaviour during a crash situation by deliberately imposing local soft zones (i.e. thermally induced triggers). The use of an appropriate modelling tool is essential for a successful industrial implementation of such local heating processes. A FE-model with an integrated numerical model for presipitates has been developed, where constitutive equations incorporate the effect of the evolving microstructure on flow stress and work hardening. The model is applied in simulations of induction heating for creating thermally induced triggers and finally to simulate the effect of these triggers on the initial buckling performance of a crash box.

11:10 AM

The Potential of Bake Hardening in Hot Rolled Multiphase Steels: *Heinz Palkowski*[1]; *Thorsten Anke*[1]; [1]Institut for Metallurgie, Collaborative Rsch. Ctr. 362, Robert-Koch-Str. 42, Clausthal-Zellerfeld 38678 Germany

Hot rolled multiphase steel qualities, e.g. Complex Phase steel or Martensite steel, show a clear Bake-Hardening-Effect rising with the degree of prestrain. Using a complex shaped construction unit (e.g. B-pillar) a locally different strength behaviour results after thermal treatment. Fundamental questions have to be answered: - Maximum for the increase of yield and tensile strength at a given prestrain? - Influence given by the kind of prestrain (one and/or biaxial)? - Rise of yield strength as a function of temperature and holding time to a maximum and when is the maximum reached? - Potential of the Bake-Hardening-Effect concerning light weight optimised body structures? The main aspect of the investigation is to determine the influence of the Bake-Hardening-Effect on mechanical properties of a formed construction unit made of multiphase steels manufactured as hot rolled strip. The mechanical-technological properties are analyzed using the Design of Experiments DoE.

11:30 AM

The Role of Non-Metallic Inclusions on the High Temperature Fracture Behavior of Low C, Al-Si Steels: *Edgar Omar Garcia-Sanchez*[1]; Armando Salinas-Rodriguez[1]; Luis Leduc-Lezama[2]; [1]Centro de Investigacion y de Estudios Avanzados del IPN, Metalurgia, Carretera Saltillo-Monterrey km 13, Ramos Arizpe, Coahuila 25900 Mexico; [2]HYLSA-DAP, Ave. Los Angeles 325 Ote, San Nicolas de los Garza, Nuevo León 66452 Mexico

The effect of temperature on the ductility of hot rolled, low C steel plates alloyed with 0.2%wt Al and 0.5%wt Si was investigated by isothermal tensile tests from 850 to 1150°C at a strain rate 5x10-4s-1. It was found that the temperature dependence of the percent reduction of area at fracture exhibits a minimum at 1000°C. Extensive characterization by SEM of fracture surfaces and deformed microstructures near the fracture surface of test samples showed that at temperatures below and above the ductility minimum, fracture takes place by nucleation, growth and coalescence of microvoids at interfaces between ferrite or austenite and (Al, Si)-rich inclusions. Energy dispersive X-ray microanalysis and electron backscattering diffraction were used to characterize the chemistry and structure of these inclusions. The ductility minimum was attributed to a synergistic effect of microvoid formation and strain-induced formation of ferrite films at austenite grain boundaries. The results obtained are discussed in terms of the hot rolling conditions used to manufacture these steels in a Continuous Strip Processing Plant.

11:50 AM

Study on the Cold Formability of Drawn Dual-Phase Steels: *Kyung Soo Park*[1]; Seong Il Hong[1]; Duk Lak Lee[2]; Chong Soo Lee[1]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Gyeongbuk 790-784 S. Korea; [2]Pohang Steel Company, Techl. Rsch. Labs., Koedong-dong, Nam-gu, Pohang, Gyeongbuk 790-600 S. Korea

Non-heat treated steels are attractive in the steel-wire industry since the spheroidization and quenching-tempering treatment are not involved during the processing, reducing the production costs. Dual-phase steel receives a great interest nowadays as a candidate for the non-heat treated steels due to its high hardening capability. Therefore, in this study, the cold formability of dual-phase steels were investigated with the variation of microstructure through different heat-treatment; intercritical quenching (IcQ), intermediate quenching (ImQ)

and step quenching (SQ). The cold formability was studied by estimating the deformation resistance and the forming limit. The deformation resistance, an important factor in determining die life, was estimated by calculating the deformation energy. Also, the forming limit was estimated by measuring the critical strain revealing crack initiation at the notch tip of the specimens. The IcQ-DP steel was found to represent the superior cold formability than other microstructures.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Inorganic Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Tuesday AM Room: 3018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Jud Ready, Georgia Tech, GTRI-EOEML, Atlanta, GA 30332-0826 USA; Lourdes G. Salamanca-Riba, University of Maryland, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany

8:30 AM Opening Remarks

8:35 AM Invited
Semiconducting and Piezoelectric Nanobelts, Nanosprings and Nanorings: *Zhong Lin Wang*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

Nanowire and nanotube based materials have been demonstrated as building blocks for nanocircuits, nanosystems and nano-optoelectronics. Quasi-one-dimensional nanostructures (so called nanobelts or nanoribbons) have been successfully synthesized for semiconducting oxides of zinc, tin, indium, cadmium and gallium, by simply evaporating the desired commercial metal oxide powders at high temperatures. The belt-like morphology appears to be a unique and common structural characteristic for the family of semiconducting oxides with cations of different valence states and materials of distinct crystallographic structures. Using the technique demonstrated for measuring the mechanical properties of carbon nanotubes based on in-situ transmission electron microscopy, the bending modulus of the oxide nanobelts has been measured, and the nanobelt is shown to be a dual mode nanoresonator for NEMS technology. Field effect transistors and ultra-sensitive nano-size gas sensors, nanoresonators and nanocantilevers have also been fabricated based on individual nanobelts. Thermal transport along the nanobelt has also been measured. Very recently, nanobelts, nanorings and nanosprings that exhibit piezoelectric properties have been synthesized, which are potential candidates for nano-scale traducers, actuators and sensors. The discovery of single-crystal perfect nanorings and its "slinky" growth model will be presented.

9:05 AM Invited
Semiconductor Nanowires as Building Block for Nanoelectronics and Nanophotonics: *Deli Wang*[1]; [1]University of California, ECE Dept., 9500 Gilman Dr., EBU1 3201, La Jolla, CA 92093 USA

Semiconductor nanowires are attractive and versatile building blocks to assemble and electrically interconnect active devices, which enable potential integration of multi-functional components on a single chip. In this presentation, I will first describe LEDs and FET based decoders assembled from bottom-up approach using semiconductor nanowires. The FET based decoders show substantial signal gain enabling signal restoration without external amplification. Second, bistable nanoscale switches based upon core-shell nanowire heterostructures organized in a crossbar architecture have been demonstrated to exhibit well-defined bistability, large ON/OFF ratios and long retention time, and from which the crossed nanowire nonvolatile RAM arrays have been assembled. Furthermore, we have integrated the new decoder with crossed-NW NVRAM arrays. Lastly, epitaxially branched single crystal nanowires were synthesized by a multiple step VLS synthesis with well controlled dimension, density and properties of nanobranches, and demonstrated active nanoscale devices. The implication of these results for nanoelectronics and nanophotonics will be discussed.

9:35 AM Break

10:00 AM Invited
Semiconductor Nanowires and Their Optical Properties: *Peidong Yang*[1]; [1]University of California, Dept. of Chmst., Berkeley, CA 94720 USA

Nanowires are of both fundamental and technological interest. They represent the critical components in the potential nanoscale electronic and photonic device applications. In this talk, we will introduce the vapor-liquid-solid crystal growth mechanism for the general synthesis of nanowires of different compositions, sizes, orientation and doping profile. Particularly, synthesis and organization of different types of heterostructured nanowires will be discussed. Wide band gap semiconductor nanostructures with near-cylindrical geometry and large dielectric constants exhibit two-dimensional ultraviolet and visible photonic confinement (i.e. waveguiding). Combined with optical gain, the waveguiding behavior facilitates highly directional lasing at room temperature in controlled-growth nanowires with suitable resonant feedback. The nanowire optical emission has been studied in detail using high-resolution optical microscopy. The waveguiding behavior of individual zinc oxide (ZnO, GaN) nanowires depends on the wavelength of the emitted light and the directional coupling of the photoluminescence (PL) to the emission dipoles of the nanowire. Pumping at high pulse intensity leads to the transition from spontaneous to stimulated emission, and analysis of the polarization, linewidth, and spacing of the spectral features facilitates identification of the transverse and longitudinal cavity modes and their gain properties. The observation of lasing action in arrayed and isolated ZnO/GaN nanowires without requiring fabrication of mirrors suggests the single-crystalline, well-facetted nanowires can indeed function as effective resonance cavities. This concept of using well-cleaved nanowires as natural optical cavities may be extendable to many other different semiconductor systems.

10:30 AM Invited
Nanowire Formation: Interfacial Morphology and Growth Kinetics: T. Savin[2]; A. Golovin[2]; S. H. Davis[2]; F. Ross[3]; J. B. Hannon[3]; J. Tersoff[3]; C. T. Black[3]; M. C. Reuter[3]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Engrg., 2220 Campus Dr., Cook Hall, Evanston, IL 60208 USA; [2]Northwestern University, Engrg. Sci. & Appl. Math, Evanston, IL 60208 USA; [3]IBM Watson Research Center, Yorktown Heights, NY USA

We examine the vapor-liquid-solid (VLS) process that is used to growth nanowires. Motivated by insitu electron microscopy images of the later stages of Si nanowire growth, we have examined the effects of capillarity on the growth rate of a wire, the composition of the liquid droplet, and the evaporation rate of the liquid into the surrounding atmosphere. Using this information we examine the time rate of change of the volume of a liquid droplet during growth and the growth rate of the wire as a function of wire diameter. The planarity of the solid-liquid interface is extremely important to insure that the composition of the resulting solid is uniform. We examine the morphological stability of the growing solid-liquid interface during VLS growth. We find that the diameter of most wires are less than the critical wavelength for the onset of morphological instability, implying that the solid-liquid interface can remain planar without the presence of faceting.

11:00 AM
In Situ Formed Beta-Ti(Ta) Nanorod Arrays Embedded in Continuous Matrix in a Multicomponent Metallic System: *Guo He*[1]; Masuo Hagiwara[1]; [1]National Institute for Materials Science, Light Matl. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

When the size of a solid is reduced into nanoscale in one, two or three dimensions, its properties usually deviate from those of "large" crystals and/or glasses with the same average chemical composition. The novel properties of nanometer scale structure have shown their advantages in many applications, such as nanoelectronics, probe microscopy, photonic crystals, molecular sieves, semiconductors and other functional nanostructured materials. So far, many different physical or chemical methods have been successfully used to synthesize or fabricate one-dimensional nanometer scale structure, e.g., nanowires, nanotubes, and nanorods. For example, using sputter deposition to from tungsten nanorods, using arc-discharge to synthesize GaN nanorods, using chemical vapor deposition to synthesize SiC nanorods, etc. The successful synthesis and/or fabrication of one-dimensional nanometer scale structures mainly include C, Si, Ta, ZnO, GaN, SiC, SiN, etc. In this presentation, we describe an in situ formed beta-Ti(Ta) nanorod arrays embedded in continuous matrix in a multicomponent metallic system.

Arsenic Metallurgy: Fundamentals & Applications: Thermodynamics and Pyrometallurgy

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Tuesday AM Room: 2014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Florian Kongoli, FLOGEN Technologies Inc., Metals Dept., Montreal, QC H3S 2C3 Canada; Trevor M. Bergfeldt, Teck Cominco Metals Ltd., Trail Operations, Trail, BC V1R 4L8 Canada

8:30 AM
Arsenic Removal from a Nickel Concentrate: *Frank Richard Jorgensen*[1]; Terry P. Hall[1]; Steve Sanetsis[1]; [1]CSIRO, Minerals, Bayview Ave., Clayton, Melbourne, Victoria 3168 Australia

The bodies of the future will contain increasing amounts of toxic elements such as arsenic which will pose quality control, environmental and safety issues during downstrem processing. A number of roasting pretreatments were assessed and applied to the treatment of a nickel concentrate containing approximately 0.5% As. The laboratory scale investigation was carried out in a small vibrated bed reactor with additions of sulphur(added either as sulphur vapour or as additional pyrite) or chlorine (added either as elemental chlorine or as chlorine produced by the in situ decomposition of added chlorides such as calcium chloride) to facilitate the decomposition and volatilisation of the arsenic impurity. The pretreatments, which were successful in removing more than 90% of the arsenic from the concentrate, are discussed in terms of the mineralogy.

8:55 AM
Fractional Distribution of Arsenic in the Teniente Continuous Converting Process: *Jonkion Font*[1]; Gerardo Alvear[1]; Alex Moyano[2]; Carlos Caballero[2]; [1]Codelco-Chile, Inst. for Innovation in Mining & Metall., Avenida del Valle 738, Santiago Chile; [2]Codelco-Chile, Div. Codelco Norte, Avenida Tocopilla s/N, Fundición Codelco Norte, Chuquicamata Chile

A thermodynamic model for the distribution of arsenic has been developed for the Teniente Continuos Converting Process, TCCP. The model is based on the quasi equilibrium fractional distribution of As among the copper, white metal, slag and gas phases. The model was validated with the data obtained in pilot and industrial tests performed at the Codelco Norte Smelter of Codelco-Chile. The model demonstrated that can be a very useful tool to predict a-priori the arsenic distribution in different operational configuration of the TCCP.

9:20 AM
Industrial Technologies Related to Arsenic Extraction: *Florian Kongoli*[1]; Robert Budd[2]; Ian McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies Inc., Metals Dept., 5757 Decelles Ave., Ste. 511, Montreal, QC H3S 2C3 Canada; [2]FLOGEN Technologies Inc., 3422 Old Capitol Trail, #791, Wilmington, DE 19808 USA

Arsenic is a particular element whose properties make it equally desirable and undesirable depending on the field of application or the process involved or related to. Arsenic trioxide, one of the common compounds of arsenic is classified as a suspected human carcinogen a cause for certain types of cancers. It has also beneficial effects in other fields such as in wood preservation, agricultural chemicals etc. Similarly arsenic metal is slightly poisonous but it is beneficial since it is primarily used in the manufacture of lead alloys for use in lead-acid batteries. Arsenic is rightly a cause for concern in non-ferrous smelting metallurgy however its extraction as a by products might be beneficial. As a result industrial and feasible technologies for arsenic extraction play an important role. In this paper, in order to have a clear picture of the state-of-the art of the past and present arsenic metallurgy the authors review several industrial technologies used for extraction of arsenic as a by-product or in other ways. Some bench scale technologies or other recently proposed extraction technologies are also reviewed.

9:45 AM
An Approach for Scaling Arsenic Removal from Smelter Gases Using Iron Oxides: *Eduardo V. Balladares*[1]; Roberto F. Parra[1]; Mario M. Sánchez[1]; José G. Palacios[2]; [1]Universidad de Concepción, Metallurgl. Engrg., Edmundo Larenas 285, Concepcion Chile; [2]Universidad de Atacama, Metallurgl. Engrg.

Most of the arsenic contained in copper concentrates is removed in the gas phase during the smelting and converting processes and, according to recent environmental regulations, it is required to fix it in a solid stable form. In this regards, ferric arsenate has been reported as thermal and aqueous stable compound. Therefore, various alternatives for treating gas containing arsenic were considered in this work. From data obtained at laboratory scale on fixation of arsenic using iron oxide, a new process to treat copper concentrates containing arsenic was evaluated. The effect of temperature, oxygen potential and the sample porosity on the fixation of arsenic contained in a gaseous stream was studied, and the iron oxide converted into ferric arsenate was up to 10%. The obtained results suggest a new alternative for gas treatment with an interesting and attractive technical development. The arsenic was retained as ferric arsenate with thermal and aqueous stability, and it allows a safe disposal keeping the environmental restrictions. The kinetics parameters are used to scale up laboratory results to higher processing rates.

Beta Titanium Alloys of the 00's: Phase Equilibria

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Tuesday AM Room: Salon 10/11
February 15, 2005 Location: San Francisco Marriott

Session Chairs: Lothar Wagner, TU Clausthal, Inst. of Matls. Sci. & Engrg., Clausthal-Zellerfeld Germany; Paul Bania, TiPro LLC, Boulder City, NV 89005 USA

8:30 AM
Continuous Cooling Transformation Curve for Beta-Processed Ti-6Al-2Sn-4Zr-6Mo: *John E. Matz*[1]; [1]Pratt & Whitney Aircraft Engines, Matls. & Processes Engrg., 100 Aircraft Rd., M/S 403-35, Middletown, CT 06457 USA

The near-beta alloy Ti-6Al-2Sn-4Zr-6Mo is used for aeroengine applications due to its high-strength and thick-section hardening capability. However, this alloy suffers from poor weld ductility and fatigue crack growth properties when welded using conventional techniques. Orthorhombic martensite forms during rapid cooling behind the weld pool. While this phase is soft and ductile, fine interfacial beta precipitates form during the post-weld stress relief treatment, causing embrittlement. Weld development efforts at P&W are focused on preventing orthorhombic martensite formation through optimization of weld thermal history. To provide a starting point for these efforts, the CCT curve for beta-processed Ti-6246 was determined from dilatometric measurements carried out in a Gleeble test machine. This material has a large prior-beta grain size relative to alpha-beta-processed material and can therefore be expected to more closely simulate the phase transformation kinetics of weld metal.

8:55 AM
Predictions of Titanium Alloy Properties Using Thermodynamic Modeling Tools: *Fan Zhang*[1]; Fanyou Xie[1]; Shuanglin Chen[1]; Y. Austin Chang[2]; Dave Furrer[3]; Vasisht Venkatesh[4]; [1]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; [2]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [3]Ladish Co., Inc., PO Box 8902, Cudahy, WI 53110 USA; [4]TIMET, R&D, PO Box 2128, Henderson, NV 89015 USA

Thermodynamic modeling has become essential tools in understanding the effect of alloy chemistry on the final microstructure of a material. Implementation of such tools to improve titanium processing via parameter optimization has resulted in significant cost savings through the elimination of shop/laboratory trials and tests. In this study, a thermodynamic modeling tool developed at CompuTherm, LLC, is being utilized to predict beta transus, phase proportions, phase chemistries, partitioning coefficients and phase boundaries of multi-component titanium alloys. This tool includes Pandat, software for multi-component phase equilibrium calculations, and PanTitanium, a

thermodynamic database for titanium alloys. Model predictions are compared to experimental results for number of beta and near-beta alpha-beta alloys. The effect of the alloying elements, especially the light elements: O, N, H, and C, on beta transus will be discussed.

9:20 AM
A Combinatorial Approach to the Elemental Optimization of a Beta Titanium Alloy Using Directed Laser Deposition: *Peter C. Collins*[1]; Daniel Huber[1]; Rajarshi Banerjee[1]; Daniel J. Evans[2]; Patrick Martin[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

The chemistry of the alloy Timetal 21S has been selected as a baseline chemistry for the development of a new high temperature beta titanium alloy. A combinatorial approach has been used to rapidly develop the new alloy. This combinatorial approach consists of employing directed laser deposition of blends of elemental powders in order to rapidly process test alloys of varying compositions. Subsequently, mechanical testing and microstructural characterization of these alloys has been carried out to populate databases that will be used for training and testing fuzzy logic based models for predicting the mechanical properties. These models will also be used to optimize the alloy composition for specific property requirements. In addition to varying the amounts of the base elements (Ti, Mo, Nb, Al, and Si), a variety of additional elements are also being tested as potential alloying additions. These include neutral elements (Zr and Sn), b stabilizers (W), and dispersoid formers (C and B). Based on the results of the coupled mechanical tests and computer models, a new group of alloys for application in high temperature thermal protection systems are being developed.

9:45 AM
Characterisation of the α Phase Nucleation in a Two Phase β-Metastable Titanium Alloy: *Astrid Lenain*[1]; Pascal J. Jacques[1]; [1]Université catholique de Louvain, IMAP, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium

α / β titanium alloys are best choice materials for the automotive and aerospace industries due to their high performance to density ratio. Among these alloys, the TIMET Ti LCB is more and more used since it presents excellent mechanical properties and a lower cost compared to other Ti alloys. The present study deals with the nucleation and growth of the α phase during several thermomechanical processes. Indeed, distribution and size of the α phase strongly influence the mechanical properties of the resulting microstructures. Several heat treatments were conducted after either cold rolling or annealing. The resulting microstructures were characterised by SEM, EBSD, XRD and TEM, showing a strong influence of the β grain size or deformation level on the conditions of α nucleation.

10:10 AM Break

10:25 AM
Properties of TIMETAL 555 (Ti-5.5Al-5Mo-5V-3Cr): *John C. Fanning*[1]; [1]TIMET, PO Box 2128, Henderson, NV 89009 USA

TIMETAL 555 is a high strength near-beta titanium alloy designed for improved producibility and excellent mechanical property combinations, including deep hardenability. The nominal chemical composition of TIMETAL 555 is Ti-5Al-5Mo-5V-3Cr (weight percent). This paper provides a summary of available data for this relatively new alloy.

10:55 AM
Influence of Thermo-Mechanical Treatments on Microstructure and Mechanical Properties of Near Beta Titanium Alloy VST55531: *J. Panter*[1]; A. Dalloz[1]; Karl-Heinz Rendigs[2]; *Nathalie Hellard*[3]; Wegmann Gerhard[3]; [1]EADS CCR, Ctr. Commun de Recherches Louis Bleriot, 12, Rue Pasteur, BP 76, 95152 Suresnes, Cedex France; [2]Airbus Deutschland, Abt. ESWG, Hünefeldstraße 1-5, 28199 Bremen Germany; [3]Airbus-France, M0122/4 ESW-T, 316, Rte. de Bayonne, 31060 Toulouse cedex 03 France

Ti-5Al-5Mo-5V-3Cr-1Zr (VST55531) is a beta Titanium alloy recently developed in the frame of a collaboration between AIRBUS and VSMPO. For the introduction of this new alloy on an airplane, a static sized part on the new AIRBUS A380 Engine Pylon has been chosen. In order to increase its use at AIRBUS, and especially to produce in the future damage tolerance sized forged parts with a excellent compromise between strength and damage tolerance resistance, a study has been carried out to increase our knowledge on the relationship between thermo-mechanical treatments, microstructure and mechanical properties of this new alloy. To do so, as received rolled and forged materials, forged in the beta phase field material and forged in the alpha + beta phase field material have been used. On all the different combinations of raw material + heat treatment, microstructure and mechanical properties have been assessed. Microstructures have been characterized using OM and FEG-SEM while mechanical properties have been characterized by tensile tests and fracture toughness tests. The results of this extensive characterization will be discussed in this paper.

11:20 AM
Modeling Property-Microstructure Relationships in Ti-5Al-5Mo-5V-3Cr-1Fe (TIMETAL-5553): *Megan Harper*[1]; Rajarshi Banerjee[1]; Daniel J. Evans[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

One of the more recently developed beta titanium alloys which is promising for a number of different applications is the Ti-5Al-5Mo-5V-3Cr-1Fe alloy, commercially referred to as TIMETAL-555. This alloy exhibits wide variations in microstructure resulting from different heat-treatments. The microstructural evolution and resultant mechanical properties of this alloy have not yet been explored in great detail. As-received, alpha+beta processed Ti-555, was subjected to a number of heat treatments with the objective of studying the microstructure and phase evolution in this alloy and obtain widely varying microstructures. Heat-treatments have been carried out in both the beta as well as the alpha+beta phase fields. The typical heat-treatment consisted of solutionization in the beta phase field or the alpha+beta phase field, cooling at different rates, and a second ageing treatment in the alpha+beta phase field. The microstructure at each stage of the heat-treatment cycle has been characterized in detail using SEM and TEM based studies. These microstructures will be quantified using rigorous stereological procedures and the corresponding room temperature tensile properties will be measured. The resulting database will be used to train and test a fuzzy-logic neural-network model that will allow for the prediction of the mechanical properties from the microstructure. The model will also be used to develop functional dependencies of the mechanical properties on the microstructural features.

Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM Room: 3009
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sungho Jin, University of California, Matls. Sci. & Engrg., La Jolla, CA 92093-0411 USA; Subra Suresh, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA

8:30 AM Invited
Molecular Biomimetics: Materials Science and Engineering Using Genetically Engineered Proteins: *Mehmet Sarikaya*[1]; [1]University of Washington, Matls. Sci. & Engrg., Roberts Hall, Box 352120, Seattle, WA 98195 USA

Physical and chemical functions of organisms are carried out by a very large number (billions) of proteins through predictable and self-sustaining interactions. Using biology as a guide, we design, synthesize, genetically tailor and utilize short polypeptides as molecular linkers and erectors in self-assembly, ordered organization, and fabrication of hybrid systems. Our objectives are accomplished in four focused areas: Selection of inorganic-binding short (7-15 amino acids) polypeptides using combinatorial biology protocols using metals, oxides and semiconductors; Quantitative assessment of the nature of binding and assembly of polypeptides on inorganics using experimental (SPR, QCM, AFM, NMR, TOF-SIMS) and modeling (molecular dynamics) approaches; Nanoassembly using functionalized designer biomolecules (chaperonins, phages, and S-layers, DNA), and electrochemically nanopatterned substrates; Creation of synthetic/biological molecular hybrids and inorganics using polypeptides in functional materials and

systems for potential utility in a wide variety of applications in nano- and nanobio-technology. Research supported by ARO-DURINT.

9:00 AM Keynote
Single-Cell Nanomechanics and Human Disease States: *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg. & Div. of Biologl. Engrg., Rm. 8-309, Cambridge, MA 02139-4307 USA

The mechanical response of living cells and subcellular cytoskeleton can undergo dramatic alterations due to biochemical changes introduced by the progression of human diseases. In this presentation, we provide experimental results on systematic alterations to the elastic properties of human red blood cells parasitized in vitro by Plasmodium falciparum malaria. By recourse to optical tweezers experiments, we extract direct force versus displacement relationships for the cell and examine contributions to cell elasticity from specific proteins transported to the membrane from the parasite. Continuum and molecular-level computational simulations of the deformation of red blood cell are also performed to quantify the nanomechanics of cell response. The mechanical properties of changes to cell deformability from P. falciparum infestation are also compared and contrasted with similar results for the P. vivax parasite. Finally, the similarities and differences in cell elasticity and disease states between malaria and human pancreatic cancer are also examined.

9:45 AM Invited
Protein Self-Assembly Creates a Nanoscale Device for Biomineralization: *Malcolm l. Snead*[1]; Michael L. Paine[1]; Wen Luo[1]; [1]University of Southern Californa, Ctr. for Craniofacial Molecular Bio., 2250 Alcazar St., CSA Rm. 109, Los Angeles, CA 90033 USA

The outer-most covering of teeth, enamel, rarely undergoes catastrophic failure despite a lifetime of repeated loading, in a wet-, acidic-, bacteria-laden-environment. Enamel forms when specialized cells, ameloblasts, synthesize proteins that self-assemble to form an enamel extracellular organic matrix that is competent to control the initiation, rate of growth and habit of the inorganic crystallites. The physical properties of mature enamel stem from the regulation imposed by a self-assembly process at the nanoscale level of organic matrix components. The physical properties of the enamel composite ceramic depend entirely on the number of genes expressed, the timing of their expression, their ability to self-assemble and the stoichiometry among the various types of enamel matrix proteins. Evolutionary change in the organization of the enamel is a reflection of the addition, diminution or change in timing for the expression of a gene and the protein it encodes being recorded during tooth development.

10:15 AM
Using DNA for Formation of Photonic Crystals and Introducing Controlled Defects: *Harris Marcus*[1]; [1]University of Connecticut, Dept. of Matls. Sci. & Engrg., Chmst. Dept, Storrs, CT 06269-3136 USA

Photonic crystals provide an innovative way for controlling light propagation. Light in photonic crystals can be trapped by a point defect in the crystal or can be channeled by a series of voids, a one dimensional waveguide. To create a two-dimensional photonic crystal, polystyrene particles were assembled in close-packed array on a glass substrate by DNA hybridization. The approaches used to create these crystals will be described with the critical impact of the DNA on the formation and subsequent creation of controlled defects. Using a continuous wave single beam laser, polystyrene particles were released to create the defects. For pattern particle release, holographic techniques will be described.

10:35 AM Break

10:50 AM Invited
Titanium Oxide Nanotubes with Controlled Morphology for Enhanced Bone Growth: Brian Oh[1]; *Sungho Jin*[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

Orthopedic and dental implants frequently utilize titanium and its alloys because of their bio-compatibility and desirable mechanical properties. We have studied nanoscale surface structures of titanium oxide with various crystal structures (including anatase) and morphologies. An array of vertically aligned titanium oxide nanotubes with ~80 nm diameter has been synthesized, which is firmly attached onto the solid Ti implant surface as a three-dimensional nanoscale structure. It is found that such a nanotube structure with a very large surface area significantly accelerates the formation of hydroxypaptite phase which is also in the nano regime. Additional nano-inspired nanostructures were also investigated. SEM and TEM microstructural analysis and possible formation mechanisms of titanium nanotubes and calcium phosphate nanostructure will also be discussed.

11:20 AM
Functionalized Magnetic Nanoparticles for Early Breast Cancer Detection: *Jikou Zhou*[1]; Challa S.S. R. Kumar[2]; Carola Leuschner[3]; Josef Hormesa[2]; *Wole O. Soboyejo*[1]; [1]Princeton University, Dept. of MAE & PRISM, E-Quad, Olden St., Princeton, NJ 08540 USA; [2]Louisiana State University, Ctr. for Advd. Microstructures & Devices, Baton Rouge, LA 70806 USA; [3]Pennington Biomedical Research Center, 6400 Perkins Rd., Baton Rouge, LA 70808 USA

Breast cancer is the second leading cause of cancer death in women. At the time of diagnosis using current methods and techniques, 34-40% of breast cancer patients already have occult metastases. These suggest that it is extremely critical and important to develop new techniques for early detection. However, the sensitivity of magnetic resonance imaging (MRI) is not efficient for early cancer detection using current method. We developed a novel approach to send MNPs into cancer tissues, so that MRI contrast is significantly enhanced. Human breast cancer cells express receptors for luteinizing hormone releasing hormone (LHRH) and can be specifically targeted by compounds linked to LHRH. By conjugating these ligands to MNPs, they can be targeted to the cancer cells, and incorporated into the cancer cells through receptor mediated endocytosis. Using this method, early detection of breast cancer would be improved, thus treatment efficiency could be significantly increased.

11:40 AM
Biological Effects of Nanoparticulate Materials: *K. F. Soto*[1]; A. Carrasco[2]; T. G. Powell[2]; L. E. Murr[1]; K. M. Garza[2]; [1]University of Texas, Dept. of Metallurgl. & Matls. Engrg., El Paso, TX 79968 USA; [2]University of Texas, Dept. of Biologl. Scis., El Paso, TX 79968 USA

The use of chrysotile asbestos nanotubes and fibril bundles in a variety of composite blends, including cement and plastics continues to be somewhat pervasive world-wide. For example, at the end of the 20th Century more than 2 million tons of asbestos were processed annually. By contrast, plastic composite development using multi-walled carbon nanotubes, which started around 1995, began to approach 1000 tons of carbon nanotubes processed annually at the same time; and the use of nanoparticulate materials is currently escalating dramatically. Both chrysotile asbestos and multi-wall carbon nanotubes have been treated as toxic materials commercially. Epidemiologic and animal studies have indicated that inhalation of asbestos can result in pulmonary fibrosis, lung cancer, and mesothelioma. In work to be reported herein cytotoxicity assays using a murine lung macrophage cell line have shown that a variety of commercial single wall and multi-walled carbon nanotubes and aggregate morphologies exhibit the same relative toxicity as chrysotile asbestos. In fact, a host of nanoparticulate materials exhibit the same levels of toxicity while characterization of these nanoparticulates by transmission electron microscopy exhibit a variety of nanoaggregate morphologies ranging from fibrils and fibril bundles to complex, branched, fractal-like aggregates of spherules. These include commercial carbon black, TiO2, Si3N4, Al2O3, Fe2O3, ZrO2, and nanoparticulate Ag. Silver is also demonstrated to be more cytotoxic than asbestos, but Ag is also a well-known bactericide; and current ointments utilizing Ag or Ag compound nanoparticulates have remarkable properties in treating wounds and certain skin diseases.The implications for nanotechnology developments and especially nanoparticulate materials processing will be discussed in terms of their parallel with the historical and world-wide asbestos trade developments and health concerns. The atmospheric occurrence of anthropogenic carbon nanotubes will also be briefly discussed in this context. Finally, it will be emphasized that in spite of the fact that nanoparticulate materials may be toxic, this is certainly not a reason to impose a moratorium on their production or use. The important issue is to recognize the necessity for precautions so as not to repeat the materials mistakes of the past. Research supported in part by a University of Texas System Louis Stokes Alliance for Minority Participation (LSAMP) Bridges to Doctorate Fellowship (KFS), RCMI Grant G12RR008124 (AC,TGP,KMG) and Mr. and Mrs. MacIntosh Murchison Endowed Chair (LEM).

12:00 PM
Synthesis and Characterization of Functionalized Magnetic Biomaterials: *Raju V. Ramanujan*[1]; L. Wong[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Block N4.1, Nanyang Ave., Singapore 639798 Singapore

Magnetic materials can be used for a variety of bioengineering applications, including cell separation, immunoassay, magnetic resonance imaging (MRI), drug and gene delivery, minimally invasive surgery, radionuclide therapy, hyperthermia etc. It is often essential to

coat the particles with suitable materials, such coatings can provide increased functionality compared to the uncoated particles. The synthesis and characterization of magnetic powders, followed by coating with dextran and gold will be described. SEM, TEM, XRD, EDX and VSM techniques were used in this investigation. Iron oxide particles were synthesized by the reverse micelle technique and coated with dextran. Gold-coated iron nanoparticles were also synthesized, the gold coating was found to be effective in protecting the particles from oxidation. The optimum coating parameters were determined and the magnetic properties of the coated and uncoated powder were determined. The investigation demonstrated the versatility of the coating techniques in the production of functionalized magnetic biomaterials.

Bulk Metallic Glasses: Fatigue and Fracture

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Tuesday AM Room: 3006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Reinhold H. Dauskardt, Stanford University, Matls. Sci. & Engrg., Stanford, CA 94305 USA; Gary Harlow, Lehigh University, Mechl. Engrg. & Mech., Bethlehem, PA 18015 USA

8:30 AM
High Cycle Fatigue in Zr-Based Bulk Metallic Glass: Damage Initiation and Growth: *Brian Christian Menzel*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matl. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 551A, Stanford, CA 94305 USA

A high-cycle stress-life fatigue study was conducted on a Zr-based bulk metallic glass to elucidate the anomalously low endurance limits that have been reported. Distributed damage was observed to initiate very early in the fatigue process as either shear bands or mixed mode surface cracks. Damage initially grew under mixed mode loading conditions in the maximum shear stress direction. On reaching a characteristic size, they abruptly changed orientation and continued to grow as mode I cracks. A focused ion beam was used to introduce a well-defined distributions of initial defects to systematically elucidate damage initiation and growth processes. High-resolution techniques were used to characterize the effect of defect size, shape and orientation on damage initiation and the early stages of damage growth. The effect of stress state was investigated using cylindrical specimens under uniaxial compression and uniaxial tension-compression loading. Surface modification techniques including coating/anodization and e-beam re-melting were investigated as ways to improve surface quality, remove damage initiation sites from the material and improve fatigue life.

8:50 AM
Free Volume and Mechanical Properties of Zr-Cu-Al: *Yoshihiko Yokoyama*[1]; Peter K. Liaw[2]; R. A. Buchanan[2]; A. Inoue[3]; [1]University of Hyogo, Matl. Sci. & Engrg., Shosha 2167, Himeji 671-2201 Japan; [2]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [3]IMR Tohoku University, Katahira, 2-1-1, Sendai 980-8875 Japan

Relative difference of free volume can be estimated by density measurement using same rod shape bulk glassy alloys (BGAs). In order to decide the standard (as crystalline state), we use ideal solution with close packed structure as mixed faced centered cubic and hexagonal close packed structures. We define the free volume to be a volume expansion ratio from the ideal solution to glassy state. Therefore, the free volume as cast state can be divided into two factors, one is the minimum required free volume for amorphization, and the other is the excess free volume. The origins of the strength and toughness in Zr-Cu-Al ternary BGAs are considered as Zr-Al networks and excess free volume, respectively. A Wöhler curve of Zr50Cu40Al10 BGAs, which composition is close to ternary eutectic point, indicates two drawbacks, i.e., low fatigue limit and significant decrease in fatigue strength in the cycle range of 10^3 to 10^4. However, these drawbacks can be improved by addition of Pd element, which can promote excess free volume and nano-network structure formation. The Zr50Cu37Al10Pd3 BGA indicates a significant increase of fatigue limit over 1 GPa.

9:10 AM
Testing-Volume Effects on the Fatigue Lifetimes of a Zr-Based Bulk Metallic Glass: *William Hutchison Peter*[1]; Peter K. Liaw[1]; C. T. Liu[2]; Raymond A. Buchanan[1]; Mark L. Morrison[1]; Gong Yao Wang[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

After a decade of fabricating "bulk metallic glasses" (BMGs), some fundamental concepts and observations regarding the fracture mechanisms of these glasses have been made. However, a full understanding of the degradation process of BMGs during fatigue has not been realized. Early fatigue studies of Zr-Based BMGs have revealed large variabilities between fatigue-endurance-limits of various testing procedures. While four-point-bend studies of BMGs have resulted in low fatigue-endurance-limits, tensile-tensile fatigue testing of radial notched specimens has resulted in fatigue-endurance-limits comparable or higher than conventional high-strength crystalline materials. Changes in the volume of BMG material that experiences the maximum stress state may cause the variation in lifetime results. The present research will address this issue by studying the lifetimes of tensile fatigue specimens with various testing volumes, and comparing the results. The location and mechanism of crack initiation will also be addressed. This research effort was made possible by the funding of the National Science Foundation Integrative Graduate Education and Research Training (IGERT) Program on "Materials Lifetime Science and Engineering" (DGE 9987548) with Drs. L. Clesceri, W. Jennings, and L. Goldberg as Program Directors; and by the Division of Materials Science and Engineering, Department of Energy under contract DE-AC-00OR22725 with Oak Ridge National Laboratory (ORNL) operated by UT-Battelle, LLC.

9:30 AM
Cyclic Fatigue Damage Zones and Anomalously High Fatigue Crack Growth Rates in Zr-Based Bulk Metallic Glass: *Peter A. Hess*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 551A, Stanford, CA 94305 USA

Zr-based bulk metallic glass (BMG) is susceptible to fatigue processes, however, the fatigue crack growth mechanisms remain largely unknown. The relaxation effect of time-dependent overloading and block loading on subsequent fatigue crack growth was measured. Results indicate a damaged and/or relaxed zone of material ahead of the fatigue crack tip. Anomalously high growth rates and low fatigue thresholds were measured following overloading and at low testing temperatures. This rapid growth was associated with fracture surface features that initiate from a point and widen in a V-shaped manner as the crack propagates. The features were reminiscent of those found in some amorphous polymers, and are associated with simultaneous cracking on multiple planes. It is analogously proposed that the features in BMG are a result of multiple shear band formation ahead of the fatigue crack tip. The role of multiple crack planes on BMG fatigue mechanisms was examined.

9:50 AM
Molecular Dynamics Investigation of Deformation and Fatigue of an Amorphous Metallic Alloy: *Kimberly K. Cameron*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 555F, Stanford, CA 94305 USA

A molecular dynamics study was conducted on a four component, Lennard-Jones amorphous solid in order to understand the atomistic mechanisms of deformation and fatigue. The deformation behavior observed was typical of that seen experimentally involving both elastic and plastic strains, load history dependence and strain rate sensitivity. Changes in free volume and excess strain were monitored during deformation to demonstrate how the stress state affects the distribution of free volume and how regions with excess free volume will preferentially deform. Under monotonic shear loading, the system developed a shear band in the region with the highest free volume. The same system developed a shear band under much lower stress under cyclic fatigue loading. This fatigue behavior can be related to the notion of directional shear transformation zones. Once a particular cluster of atoms undergoes a shear transformation, it does not return to its original state on unloading. Furthermore, in subsequent cycles although this cluster will not transform again, the surrounding region is more likely to have further shear transformations. This localization of the deformation process accelerates the shear banding process and is used to rationalize the poor fatigue properties commonly reported for metallic glasses.

10:10 AM Break

10:30 AM

Influence of Composition and Structure on Fatigue Behavior of Zr-Based Bulk Metallic Glasses: *Gongyao Wang*[1]; P. K. Liaw[1]; Y. Yokoyama[2]; M. Freels[1]; D. L. Weinberg[1]; B. Yang[1]; W. H. Peter[1]; R. A. Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]University of Hyogo, Matls. Sci. & Engrg., Shosha 2167, Himeji City Japan

High-cycle fatigue (HCF) experiments were conducted on zirconium (Zr)-based bulk- metallic glasses (BMGs): $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$, in atomic percent. The X-ray diffraction results show that $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ have an amorphous structure. However, $Zr_{50}Cu_{37}Al_{10}Pd_3$ contains much more free volumes and some nano structures. The HCF experiments were performed using an electrohydraulic machine at a frequency of 10 Hz with an R ratio of 0.1 and under tension-tension loading, where $R = \sigma_{min.} / \sigma_{max.}$, $\sigma_{min.}$ and $\sigma_{max.}$ are the applied minimum and maximum stresses, respectively. The test environments were air and vacuum. A high-speed and high-sensitivity thermographic-infrared (IR) imaging system has been used for the nondestructive investigation of the temperature evolution during fatigue experiments of BMGs. Limited temperature evolution was observed during fatigue. A sparking phenomenon was observed at the final fracture moment of $Zr_{50}Cu_{30}Ni_{10}Al_{10}$. However, no sparking phenomenon was found at the final fracture moment of $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{37}Al_{10}Pd_3$. The vein pattern and droplets with a melted appearance were observed in the apparent melting region. The fatigue lives in vacuum are longer than those in air. The fatigue-endurance limit of $Zr_{50}Cu_{37}Al_{10}Pd_3$ was found to be significantly greater than those of $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, which indicates that the inclusions of Pd that increased the free volume and nano structures improve the fatigue resistances of the Zr-based BMGs. The fracture morphology indicates that fatigue cracks initiate from the outer surface of the sample. A mechanistic understanding of the fatigue behavior of the Zr-based BMGs is suggested.

10:50 AM

An Approach to Modeling the S-N Behavior of Bulk-Metallic Glasses: *D. Gary Harlow*[1]; Peter K. Liaw[2]; William H. Peter[2]; Gongyao Wang[2]; Raymond A. Buchanan[2]; [1]Lehigh University, Mechl. Engrg. & Mech., 19 Memorial Dr. W., Bethlehem, PA 18015 USA; [2]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA

Current approaches to modeling the S-N behavior of materials are primarily empirical based on statistical analyses of experimental data. Consequently, they do not adequately reflect long-term performance. Furthermore, they do not identify the key sources and extent of their contributions to randomness arising from microstructure, environment, or loading. An approach for modeling the S-N response using a standard fatigue crack growth model is proposed. The model captures the variability in fatigue lives by relating it to key material variables, both deterministic and random, that are readily identified in the proposed model. The identification and significance of these variables are paramount for predicting fatigue crack growth and the subsequent damage evolution. The effectiveness of this approach is demonstrated with an amalgamated set of S-N data for bulk-metallic glasses. Variability associated with manufacturing and material variables are considered. Adoption of this approach is recommended for sound scientific and probabilistic life prediction.

11:10 AM

Fatigue Behavior of an Amorphous Steel: *Gongyao Wang*[1]; P. K. Liaw[1]; J. Poon[2]; M. W. Freels[1]; D. L. Weinberg[1]; D. Qiao[1]; R. A. Buchanan[1]; C. R. Brooks[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]University of Virginia, Dept. of Physics, Charlottesville, VA 22904 USA

A non-magnetic amorphous steel, $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ (atomic percent), generally has a three-times greater strength than the conventional steel. Four-point-bend fatigue experiments were conducted on the amorphous steel. Specimens were cycled under a load control using an electrohydraulic machine at a frequency of 10 Hz (sinusoidal waveform) with an R ratio of 0.1, where $R = \sigma_{min.} / \sigma_{max.}$, $\sigma_{min.}$ and $\sigma_{max.}$ are the applied minimum and maximum stresses, respectively. The test environment was in a laboratory air. $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ exhibited a high fatigue-endurance limit (694 MPa). However, the stress versus number of fatigue cycles curve of $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ revealed that the lifetime decreased abruptly with increasing the tensile stress at higher stress levels. The fatigue behavior of Fe-Based and Zr-Based BMGs and compared. The fatigue-endurance limit of $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ was about 5 times greater than that of the Zr-based bulk-metallic glass (BMG). However, at high stress levels, the amorphous steel demonstrated a shorter fatigue life than the Zr-based BMG. The fracture morphology indicated that $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ has a brittle fracture. Fatigue cracks initiated from the outer tensile surface of the specimen, and the fatigue-crack-propagation region was very small. The mechanisms of fatigue-crack initiation and growth are suggested.

11:30 AM

Fatigue Behavior of a Cu-Based Bulk Metallic Glass: *Matthew W. Freels*[1]; Peter K. Liaw[1]; Gongyao Y. Wang[1]; Devin L. Weinberg[1]; Q. S. Zhang[2]; Z. Q. Hu[2]; Raymond A. Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg. Dept., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Chinese Academy of Science, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsch., 72 Wenhua Rd., Shenyang 110016 China

If bulk metallic glasses (BMGs) are to find uses in structural applications, it is important to understand their fatigue behavior. Initial fatigue studies of BMGs showed lower fatigue strengths than were anticipated. However, more recent fatigue studies of Zr-based BMGs have shown much higher fatigue strengths than the initial results. To our knowledge, no fatigue studies have been performed on Cu-based BMGs. In this study, four-point-bend fatigue experiments were conducted on (Cu60Zr30Ti10)99Sn1 (atomic percent) BMGs in air. The fatigue experiments were performed using an electrohydraulic machine under a load control mode at frequencies of 1 and 10 Hz (sinusoidal waveform) with an R ratio of 0.1, where $R = \sigma_{min.}/\sigma_{max.}$, and $\sigma_{min.}$, and $\sigma_{max.}$, are the applied minimum and maximum stresses, respectively. The results are compared to similar conventional crystalline alloys. The present work is supported by the National Science Foundation (NSF), the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program, under DMR-0231320, with Ms. M. Poats, and Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, and C. Huber as contract monitors.

11:50 AM

A Comprehensive Synopsis of Fatigue Behavior in Zr-Based Bulk-Metallic Glasses: *Devin Lance Weinberg*[1]; Matthew Webster Freels[1]; Gongyao Y. Wang[1]; Dongchun Qiao[1]; William H. Peter[1]; Peter K. Liaw[1]; Raymond Allen Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA

A great deal of research has been performed throughout the materials science field in recent years to quantify and understand the high-cycle fatigue (HCF) behavior of zirconium (Zr)-based bulk-metallic glasses (BMGs). However, the published results remain scattered in various academic journals ensuring that comparisons and trends are difficult to analyze. Data from a variety of HCF experiments on Zr-based BMGs as well as common crystalline alloys was combined and organized by testing method and graphed as stress range and stress amplitude/tensile strength versus cycles to failure. Although it is difficult to make true comparisons, given variations in material composition, sample geometry, and testing procedure, several interesting trends were observed, which present a clear opportunity for future fatigue studies. One such trend appears to be decreasing endurance limits as the frequency of the HCF experimental loading is reduced. Furthermore, the comparison implies that four-point bend HCF experiments yield lower endurance limits than axial fatigue tests, which could be contrary to commonly accepted results in the well-studied crystalline alloys. The present work is supported by the National Science Foundation (NSF), the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program, under DMR-0231320, with Ms. M. Poats, and Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, and C. Huber as contract monitors.

Cast Shop Technology: Aluminum Melting: Furnace Design and Refractories

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday AM Room: 2001
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Paul King, Albany Research Center, Albany, OR 97321 USA; Subodh K. Das, Secat Inc., Ctr. for Al Tech., Lexington, KY 40511 USA

8:30 AM
An Analytical Furnace Model for Optimizing Aluminum Melting Furnaces: *Tianxiang Li*[1]; Paul King[2]; Mohamed Hassan[1]; Kazunori Kuwana[1]; Kozo Saito[1]; [1]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0108 USA; [2]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321-2198 USA

An analytical furnace model, originally established by Essenhigh and Tsai, is developed in this paper. The practical application of this model is to predict optimum furnace operation and was verified by experiments conducted in the Experimental Research Furnace (ERF) at Albany Research Center of the U.S. Department of Energy. The development of the Essenhigh/Tsai model is based on a few melting and holding tests with two main assumptions; the thermal conduction loss in aluminum melting is the same for both melting and holding processes, and the heat loss through the flue gases is lineally proportional to the melting rate. The former is reasonable because the thermal conduction loss is small as compared with the firing rate, while the later is quite accurate as shown in the test results. Tests of aluminum melting were conducted on the ERF furnace in the cases of high, normal and low roof heights. From the tests, the relations between firing rate, heat absorption rate, melting rate, and energy efficiency were developed, and the optimum operation conditions under which the maximum energy efficiency can be achieved were predicted. In addition, the effect of roof height on the energy efficiency was determined. This model could be a valuable tool in diagnostic analysis of day-to-day operations in aluminum melting.

8:55 AM
Experimental Investigation on Stress-Corrosion of Refractories Exposed to Liquid Aluminum: Roger Pelletier[1]; *Claude Allaire*[1]; Vincent Ébacher[1]; [1]École Polytechnique de Montréal, Engrg. Physics, CIREP, Campus CRIQ, 8475, Christophe-Colomb, Montréal, Québec H2M 2N9 Canada

The interaction between stress and corrosion is a well known phenomenon observed in many environment/material engineering systems. This paper presents the results of a study whose objective is to verify if stress-corrosion interactions are possible between liquid aluminum and refractories. To observe the possible interaction, specimens are submitted to a creep flexion test while being partially submerged into liquid aluminum. Pure corrosion and pure creep tests are used as references to put forward the interactions. The results obtained so far suggest that tensile stresses enhance corrosion of refractories.

9:20 AM
Scale Modeling of Aluminum Melting Furnace: Sita Rama Raju S. Penmetsa[1]; *Tianxiang Li*[1]; Paul King[2]; Kozo Saito[1]; [1]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0108 USA; [2]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321-2198 USA

Secondary (recycled) aluminum constitutes around 35% of the total aluminum used in the United States. Secondary aluminum melting is accomplished in large reverberatory furnaces, and improving its energy efficiency has been of a major interest to the aluminum industry. To assist the aluminum industry in improving the melting efficiency, an experimental research furnace (ERF as a prototype) of about 907 kg (2000 lbs) has been built at Albany Research Center of the U.S. Department of Energy as part of multi-partner research program. This paper describes the scaling laws of aluminum melting furnace, the design of a small scale model furnace (14 kg capacity), and the scale model experiments. A partial scale modeling technique was applied to derive achievable scaling laws on temperature distributions in and at the furnace walls including the roof. Temperature distributions obtained for the small scale model furnace was favorably compared with prototype. Well agreement of the results from model experiments with these from tests on ERF validates that we can use the scale modeling technique to infer the physics on the melting processes of industrial furnaces.

9:45 AM
Combustion Space Modeling of an Aluminum Furnace: *Brian Mark Golchert*[1]; Chenn Q. Zhou[2]; Antoine Quenette[1]; Quinyou Han[3]; Paul E. King[4]; [1]Argonne National Laboratory, Energy Systems, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Purdue University Calumet, Mechl. Engrg., 2200 169th St., Hammond, IN 46323-2094 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [4]Albany Research Center, 1450 Queen Ave., SW, Albany, OR 97321 USA

Secondary aluminum production (melting from aluminum ingots, scraps, etc.) offers significant energy savings and environmental benefits over primary aluminum production since the former consumes only five percent of the energy used in the latter process. The industry, however, faces technical challenges of further improving furnace melting efficiency and has been lacking tools that can help understand combustion process in detail and that will facilitate furnace design. Computational Fluid Dynamics (CFD) modeling has played increasingly important roles in evaluating industrial processes. As part of a larger program run by SECAT, a CFD model has been developed at Argonne National Laboratory to simulate fuel combustion, heat transfer (including thermal radiation), gaseous product flow (mainly CO2 and H2O), and production/transport of pollutant species/greenhouse gases in an aluminum furnace. Using this code, the surface heat fluxes are calculated and then transferred to a melt code. In order to have a high level of confidence in the computed results, the output from the code will be compared and validated against in-furnace measurements made in the Albany furnace. Once validated, the combustion code may be used to perform inexpensive parametric studies to investigate methods to optimize furnace performance. This paper will present results from the combustion modeling of an aluminum furnace as well as results from several parametric studies.

10:10 AM Break

10:20 AM
Potentials for Increasing Efficiency of Aluminium Melting Furnaces: *Jan Migchielsen*[1]; Jan D. de Groot[1]; [1]Thermcon Ovens B.V., Process Dept., PO Box 97, Geldermalsen 4190 CB The Netherlands

The introduction of heat recovery on aluminium melting furnaces has lead to a considerable reduction of the specific fuel requirement for the melting of aluminium. Since already a considerable time now, melting furnaces equipped with regenerative burners are generally the most economic solution for melting solid metal. With the Kyoto treaty and the increased cost for energy, new incentives are created to further reduce the specific energy cost for melting aluminium. Operating a furnace in a "smart" way by optimizing charging procedures and fine-tuning existing burner systems, does achieve additional energy saving. A smarter operation can also be incorporated into the furnace control system. Dynamically adjusting the air-fuel ratio and optimizing the regenerative burner setting will reduce energy cost and increase productivity. The introduction of a hybrid type furnace with both regenerative burners and high velocity burners to optimize heat transition will lead to a further reduction of the fuel cost for specific applications. This paper addresses the latest developments on the field of aluminium melting furnaces to achieve both by design and smart operation of the furnaces lower specific fuel consumption, minimized maintenance and balanced productivity.

10:45 AM
Design and Operation of an Experimental Reverberatory Aluminum Furnace: *Paul E. King*[1]; Michael C. Hayes[1]; Tianxiang Li[2]; Qingyou Han[3]; Mohamed Hassan[2]; Brian M. Golchert[4]; [1]U. S. Dept. of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506 USA; [3]Oak Ridge National Laboratory, One Bethel Valley, Oak Ridge, TN 37831-6083 USA; [4]Argonne National Laboratory, Process Simulation & Modlg., 9700 S. Cass Ave, ES/362, Argonne, IL 60439 USA

The U. S. Dept. of Energy, Albany Research Center, in cooperation with industrial support through Secat, Inc. has designed, built and is operating a test-bed reverberatory furnace. Studies in the Albany Research Center (ARC) experimental reverberatory furnace (ERF) include melt efficiency as a function of combustion space volume, power input and charge alloy. This paper details the furnace design, experimental equipment, conditions, procedure, and measurements and in-

cludes results and discussions of melt efficiency research. Specific results reported include an analysis of the overall efficiency of the furnace as a function of power input and the effect of changing the combustion space volume has on the melting efficiency. An analytic analysis of the theoretical efficiency of the furnace is carried out to determine overall characteristics of the furnace. Experimental data is utilized to validate numerical (computational fluid dynamics) predictions.

Characterization of Minerals, Metals and Materials: Characterization of Structural Engineering Materials - II

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday AM Room: 2010
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Jian Li, Natural Resources Canada, CANMET-MTL, Ottawa, Ontario K1A 0G1 Canada; Louis Evrard, Universite Catholique de Louvain, Physl. Chmst. & Matls. Engrg., Louvain-la-Neuve Belgium

8:30 AM
A Comparative Evaluation of Precipitation Behavior in Equivalent Strength V-Nb-Ti and V-Containing Microalloyed Steels During Processing of Structural Beams: *Sankaran Shanmugam*[1]; Devesh K. Misra[1]; Dhiren Panda[2]; Fulvio Siciliano[3]; [1]University of Louisiana, Cheml. Engrg. Dept., Matls. Sci. & Engrg. Grp., PO Box 44130, Lafayette, LA 70504-4130 USA; [2]Nucor-Yamato Steel, PO Box 1228, 5929 E. State Hwy. 18, Blytheville, AR 72316 USA; [3]Reference Metals Company, 1000 Old Pond Rd., Bridgeville, PA 15017 USA

The paper describes a comparative evaluation of the precipitation behavior of V-Nb-Ti and only V-containing steels to investigate the effective cumulative role of the addition of Nb-Ti to V-steel. Transmission electron microscopy was employed to characterize the precipitates in equivalent strength V-Nb-Ti and vanadium-containing steels. While the mechanical properties are similar, there are significant differences in the precipitation behavior of the two steels. The microstructure of V-Nb-Ti steels consists of predominantly polygonal ferrite, fine pearlite, and a small % of bainite. Titanium forms coarse cuboidal carbonitrides, and niobium and vanadium form very fine precipitates of carbonitrides. The former tends to preferentially precipitate along the grain boundaries, whereas the latter are dispersed in the matrix. The niobium and vanadium carbonitrides cause ordering of precipitates in the ferrite lattice, which is displayed by the diffraction pattern as chemically sensitive or superlattice reflection. Additionally, niobium carbonitrides exhibit a ring SAD pattern. The different precipitates exhibit an orientation relationship with the matrix and their partitioning is not observed in the pearlitic or bainitic ferrite. The bainite region is characterized by a complete precipitation of cementite. In general, the microstructure is free of dislocations except at the interface of the ferrite/pearlite.

8:55 AM
The Precipitation Behaviors of Inconel 740 and Super 304H for Ultra Supercritical Application: *Quanyan Wu*[1]; Vijay K. Vasudevan[1]; John Shingledecker[2]; Robert Swindeman[2]; [1]University of Cincinnati, Cheml. & Matls. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [2]Oak Ridge National Laboratory, Ceram. & Metals, Bldg. 4500S, MS6155, PO Box 2008, Oak Ridge, TN 37831 USA

New alloys have been developed to meet higher temperature demands for higher thermal efficiency and lower emission in coal power plants. Inconel 740 is a new nickel-base superalloy which has demonstrated good thermal stability and strength up to 750°C. The major precipitates were MC, $M_{23}C_6$, and gamma prime when solutionized at 1050°C. Eta precipitates formed a Widmanstatten pattern when creep tested, in addition to gamma prime rafting. A modified version of Inconel740 showed reduction of eta phase and better creep strength. The advanced austenite stainless steel Super 304H also showed excellent long-term strength, and microstructural study revealed massive precipitation of fine-scale copper-rich precipitates. These precipitation behaviors, after ageing and creep-test under a series of temperatures will be characterized using TEM, EDS, SEM and comparison between these two alloys will be presented.

9:20 AM
Characterization of the Inhibition Layer on Hot Dip Galvanized and Galvannealed Steels: S. Dionne[1]; *J. Li*[1]; V. Guertsman[1]; G. A. Botton[2]; F. E. Goodwin[3]; [1]CANMET-Materials Technology Laboratory, 568 Booth St., Ottawa, Ontario K1A 0G1 Canada; [2]McMaster University, Dept. of Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4M1 Canada; [3]ILZRO, 2525 Meridian Pkwy., PO Box 12036, Rsch. Triangle Park, NC 27709-2036 USA

An iron aluminide inhibition layer is formed on the steel surface during continuous hot dip galvanizing. On a galvanized product, this inhibition layer must be thick enough to prevent the formation of iron-zinc intermetallics and to obtain a ductile coating. In the case of galvannealed products, the galvanizing parameters are adjusted to obtain a thinner inhibition layer which is broken down during the galvannealing treatment to allow interdiffusion of iron and zinc. An understanding of the effects of process parameters and substrate characteristics on the coating microstructure is critical to optimize the coating performance. In the present study, the microstructure of galvanized coatings produced on a variety of steel types under conditions typical of industrial continuous galvanizing was examined using a combination of advanced characterization techniques.

9:45 AM
Characterization of Deterioration in a Shunting Locomotive for Lifetime Assessment: *Jeongguk Kim*[1]; Jong-Duk Chung[1]; Jang-Sik Pyun[1]; Yong-Ki Hong[1]; Dae-Sung Bae[2]; [1]Korea Railroad Research Institute, Railroad Safety Rsch. & Testing Ctr., 360-1 Woulam, Uiwang, Kyunggi 437-757 S. Korea; [2]Hanyang University, Dept. of Mechl. Engrg., Seoul 426-791 S. Korea

The deterioration of a shunting locomotive was characterized for the lifetime assessment. The locomotive was using for shunting works in steel industry, and in this paper, various types of technical evaluation methods for the locomotive parts were introduced. Unlike other rolling stocks in railway applications, the diesel shunting locomotive is composed of major components such as diesel engine, transmission, gear box, brake system, etc., which cover more than 70 percent of the total price of the locomotive. Therefore, in this investigation, each part of major components in the diesel locomotive was analyzed in terms of the degree of deterioration. The performance evaluation tests were performed on the diesel engine and gear box, and precision inspection was conducted on each part of transmission to provide the current wear information. Also, corrosion test and nondestructive evaluation techniques were employed to demonstrate the wear status of coachwork and bogie parts in locomotive. In this investigation, several engineering characterization techniques for the lifetime analysis of a shunting locomotive were applied and presented.

10:10 AM Break

10:20 AM
Development of Heat Resistant Cast Alloy for Engine Exhaust Manifold: *Sung Hwan Park*[1]; *Jong Moon Kim*[1]; *Hak Jin Kim*[1]; *Se Jin Ko*[1]; Hyoun Soo Park[1]; Jong Dae Lim[1]; [1]Hyundai-Motor Company, R&D Ctr./Matl. Rsch. Team, Jangduk 772-1, Whasung, Kyunggi 445-706 S. Korea

The heat resistant cast alloy has been developed for exhaust manifold of new passenger diesel engines. Operating demands on exhaust manifold have increased significantly over the past decade because of higher exhaust gas temperature by tighter emission requirement, improved fuel efficiency and design toward higher specific engine power. This has led to much higher elevated temperature strength and oxidation resistant demands on exhaust manifold alloy. Besides, thermal fatigue occured directly as a result of thermal expansion and mechanical constraint, becomes an important issue. Our reserach work focuses on alloy design to improve durability of engine exhaust manifold. Low cycle fatigue and high temperature oxidation properties are evaluated. Casting design and condition are optimized by numerical simulation for better manufacturing.

10:45 AM
AISI 304 K Steel Subjected to Small Charge Explosions: Microstructural Changes with Limited or No Macro-Deformation: *Donato Firrao*[1]; Paolo Matteis[1]; Giorgio Scavino[1]; Graziano Ubertalli[1]; Maria G. Ienco[2]; Paolo Piccardo[2]; Maria R. Pinasco[2]; Enrica Stagno[2]; Roberto Montanari[3]; Maria Elisa Tata[3]; Giovanni Brandimarte[4]; S. Petralia[4]; [1]Politecnico di Torino, Dip. di Scienza dei Materiali e

Ingegneria Chimica, Corso Duca degli Abruzzi, 24, Torino 10129 Italy; [2]Università di Genova, Dip. di Chimica e Chimica Industriale, Via Dodecanneso, 30, Genova 16146 Italy; [3]Università di Roma "Tor Vergata", Dip. di Ingegneria Meccanica, Via del Politecnico, 1, Roma 00133 Italy; [4]Marina Militare, Istituto di Chimica Esplosivi, Viale S. Bartolomeo n. 400, La Spezia 19100 Italy

Microstructural variations induced on AISI 304 K steel disks by explosions which do not cause gross macro-deformation are illustrated. 50 and 100 g NSP explosive spherical charges and explosive-target distances in the range from 0.05 to 0.8 m were used to achieve pick pressures in the 960 to 3 bars range. Two grain sizes (60 and 30 μm) were tested. Microstructural features were studied by XRD, OM, SEM and STM microscopy. Surface OM and SEM evidenced dark spots, due to oxidation and deposition of explosive components, zones with recrystallization phenomena, grain boundary melting, and twins. Phenomena in the interior of the samples restricted to twins. They can be seen up to some distance from the explosion impinged surface and again for a smaller distance around the reflecting surface. The maximum charge-to-target distance at which the phenomena disappear has been singled out for each charge and grain size and related to the critical resolved shear stress for twinning.

11:10 AM

Textural Inhomogeneities in Drawn and Annealed OFHC Copper Wire: *D. R. Waryoba*[1]; P. N. Kalu[1]; [1]FAMU-FSU College of Engineering, Dept. Mechl. Engrg., 2525 Pottsdamer St., Tallahassee, FL 32310-6046 USA

Textural inhomogeneities have been investigated in oxygen free high conducting (OFHC) copper wire drawn at room temperature to a true strain of 2.31, and isothermally annealed at 250°C and 750°C for annealing times ranging from 10 s to 1 hr. Local orientations were mapped by means of orientation imaging microscopy (OIM). The microtexture of the drawn wires showed a strong <111> + weak <100> duplex fiber texture at the inner core, whereas the mid and surface regions had a comparatively weak texture. Annealing at 250°C resulted into a recrystallization which originated from the mid section, proceed towards the surface regions and ends in the inner core. Recrystallization resulted into a strong<100>+weak<111> duplex fiber texture. A similar textural inhomogeneity was observed during short annealing at 750°C. However, prolonged annealing gave rise to abnormal grain-growth that proceed from the inner core to the outer surfaces with a dominant <111> fiber component at the inner region and mixed components of <111>, <100>, and <112> at the outer surfaces.

11:35 AM

HRTEM Charaterization of Ni-Ni_3Ti Eutectic Alloy: *Y. Sikaddour*[1]; K. Taibi[1]; [1]Université des Sciences et de la Technologie Houari Boumedienne (USHB), Faculté de Génie Mécanique & Génie des Procédés, Dépt. des Scis. des Matériaux, BP 32 EL Alia Bab, Ezzouar 16000 Alger

Ni-Ni_3Ti eutectic alloy as cast exhibit a widmanstätten structure in the nickel matrix, it has been found that depending on cooling rate and chemical composition. The precipitates are thin plates of the hexagonal phase $Ni_3Ti(Do_{24})$. Ni-Ni_3Ti eutectic alloy has been prepared by unidirectional solidification in order to investigate a widmanstätten structure in FCC Nickel phase. Analysis of HRTEM images has demonstrate in Do_{24} structure, the presence of misfit dislocation with cores associated with very small ledges with a height of four close-packed plans spacing and its burgers vectors is $(-1/6)[112]_{Ni}$ parallel to the interface facets.

Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials - I

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee

Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday AM — Room: 2012
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Roderick I.L. Guthrie, McGill University, McGill Metals Procg. Ctr., Montreal, Quebec H3A 2B2 Canada; Michael J. McKelvy, Arizona State University, Ctr. for Solid State Sci., Tempe, AZ 85287-1704 USA

8:30 AM

Characterization of the S-Phase with a Modified Orientation Relationship: *Libor Kovarik*[1]; Stephen A. Court[2]; Michael J. Mills[1]; [1]Ohio State University, 2041 College Rd., Watts Hall #477, Columbus, OH 43210 USA; [2]Alcan Technology & Management, Badische Bahnhofstrasse 16, Neuhausen CH 8212 Switzerland

Two possible orientation relationships (OR) are known for the S-phase in Al-Cu-Mg alloys. It has been previously shown that one of the two OR is metastable and found only for S-phase particles that do not exceed certain dimensions. In our work on alloys with low Cu/Mg ratio and small additions of Si, we employed HRTEM and Z-contrast imaging to study the origin of the metastable OR. As compared to the previously published explanation based on an invariant line concept, we find that the OR may be governed instead by a particular S-phase/matrix interface that has excellent atom-site matching for the metastable OR considered. Microscopy observations are supported by a model interface that shows this atom-site matching. Based on EDX analysis, we also find that the creation of metastable OR may be facilitated by the local enrichment of Si.

8:55 AM

Study of Deformation of Cube-Textured Aluminum Using Laser-Induced Photoelectron Emission: *Mingdong Cai*[1]; Lyle E. Levine[2]; J. T. Dickinson[1]; [1]Washington State University, Physics Dept., Pullman, WA 99164 USA; [2]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., Gaithersburg, MD 20899 USA

Uniaxial deformation of a cube-oriented aluminum sample was monitored by a laser-induced photoelectron emission technique. A retarding-field energy analyzer was used to determine the energy distribution of photoelectrons. Due to anisotropy of the surface work function in aluminum, electrons of 0.5 eV (using 248-nm laser radiation) were found to dominate the photoelectron energy spectra indicating a strong cube texture. After deformation, obvious increases in the intensities of photoelectrons with 0.7 and 1.0 eV kinetic energies were detected, which correspond to increases of {111} and {110} surface area. Quantitative texture analysis by electron backscattered diffraction (EBSD) data showed an increase of {110} surface area after deformation. Laser-induced photoelectron emission is sensitive to slip events and possible grain rotation, while EBSD demonstrates texture evolution as a result of slip and lattice rotation. The higher energies observed in the photoelectron spectra are consistent with the measured evolution in texture during deformation.

9:20 AM

Nucleation and Early Growth of Fatigue Cracks in 2024-T3 Aluminum Alloy: *Jonathan Tsang*[1]; Ali Merati[2]; [1]Carleton University, Dept. of Mechl. & Aeros. Engrg., 1125 Col. By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]National Research Council of Canada, Inst. for Aeros. Rsch., 1200 Montreal Rd., Bldg. M-13, Ottawa, Ontario K1A 0R6 Canada

While the long crack regime has been well established and understood, our understanding of the mechanisms controlling fatigue crack nucleation and short crack growth are still under debate. Crack nucleation and short crack growth consume up to ~95% of the fatigue life. Therefore, early crack growth is of great importance and must be fully understood before an accurate holistic and materials-based life prediction model can be developed. An extensive study has been carried out to evaluate the main factors affecting the fatigue crack nucleation process and to characterize the growth of physically short cracks. In

this work, different crack detection and monitoring techniques such as surface replication, marker bands, and electrical potential drop have been used. It was observed that almost all the fatigue failures originated at constituent particles. Early crack growth patterns were documented and a novel hypothesis for the short crack mechanism was presented.

9:45 AM
Characterization of Al-SiC Particulate Composite by Using Digital Image Analysis for Three-Dimensional Reconstruction of Microstructural Volume: *Harpreet Singh*[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Schl. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30318 USA

Al-SiC metal matrix composites (MMC's) find applications in various fields ranging from automotive, aerospace to electronics (i.e. structural to functional material). The key factors of this MMC that make it useful for the wide variety of applications are light weight aluminum and SiC particles which provide mechanical stability to the composite. In this paper, we present a technique to reconstruct the three dimensional microstructural volume of the Al-SiC MMC from serial sections and characterize three samples with different processing parameters. A total of hundred sections, approximately 1 mm apart, were used to reconstruct the 3D volume. The 3D reconstruction is utilized to characterize local particle morphologies, particle connectivity, and near neighborhood spatial heterogeneities of microstructure in three-dimensional space.

10:10 AM Break

10:20 AM
Interface Reaction of Boron Carbide in Aluminum Matrix Composites and its Control: *X. Grant Chen*[1]; [1]Alcan International Limited, Arvida R&D Ctr., 1955, Mellon Boul., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

Aluminum matrix composites containing reinforcing particles of B4C have been used in certain high-performance applications such as neutron-shielding components and aircraft and aerospace structures. B4C is a desirable reinforcement because of its special capability to capture neutrons, lightweight and superior stiffness and strength. However, further use of the B4C particle-reinforced composite material is limited due to its complicated manufacturing and cost. One of the major challenges is the interface reaction between B4C and Al that is detrimental to the castability and material properties. The present paper describes work conducted to better understand the interface reaction during the liquid mixing process and the resulting cast structure. The instability of B4C in liquid aluminum and its reaction products were investigated using thermodynamic calculation, optical metallography and scanning electron microscopy. A novel method is presented for controlling the interfacial reaction by forming an in-situ barrier layer using a Ti addition in molten aluminum.

10:45 AM
Characterization of Subsurface Damage Accumulation in Aluminum Alloys Subjected to Multiple Asperity Sliding Contacts: *S. S. Akarca*[1]; W. J. Altenhof[1]; A. T. Alpas[1]; [1]University of Windsor, Dept. of Mechl., Auto., 401 Sunset Ave., Windsor, ON N9B 3P4 Canada

In certain tribological applications of aluminum, sliding wear surfaces involves large plastic deformation and subsurface damage, even under light loads. In the subsurface layers adjacent to the contact surfaces, the competition between the plastic strain, which enhances void growth, and the hydrostatic pressure, which suppresses it, is considered to be responsible for the generation of a damage gradient. On the basis of this concept, debris formation can be attributed to the delamination of the subsurface layers at a certain depth where the damage accumulation rate is highest. Numerical analyses are necessary to determine the magnitude and distribution of the hydrostatic pressure and hence determine the subsurface damage gradient under various loading conditions. Accordingly, the main objective of this study is to build a finite element model to analyze subsurface deformation that occurs in aluminum alloys subjected to sliding contact. An Eulerian model capable of accounting for large strain accumulation under multiple asperity contact situation was employed. The effects of friction, normal load and distance between the asperities on multiple asperity contacts were investigated. Numerical results were tested by normal contact experiments.

11:10 AM
Experimental Study and Modelling of Phase Transformation in 3003 Aluminium Alloys During Homogenisation: *Moukrane Dehmas*[1]; Mickael Serriere[1]; Pierre Archambault[1]; Elisabeth M. Aeby-Gautier[1]; Charles-Andre Gandin[1]; [1]LSG2M, ENSMN, Parc de Saurupt, Nancy Cedex 54042 France

This paper aims to study microstructural evolutions in the 3003 aluminium alloys during the heating from the as-cast state. After solidification, both the solid solution and the primary precipitates are far from equilibrium and during heating, a precipitation of dispersoïds and an eutectoïd transformation of the primary particles occur. Quantitative experimental results, obtained by high energy synchrotron radiation and by image analysis, are compared to in-situ electrical resistivity and to a precipitation model. In a first time, the different quantitative methods are analysed (synchrotron results, and MET quantitative image analysis) and lead to evolutions of the volume fraction of dispersoïds and primary particles as a function of temperature. In a second time, the evolution of the electrical resistivity are compared to the manganese content in the solid solution predicted by a precipitation model. This manganese content evolves by the fine precipitation and then, by the enrichment of the primary particles.

11:35 AM
An Electron Microscope Study of Mechanical Twinning and Fracture in Two-Phase Intermetallic Titanium Aluminides: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsch., Geesthacht D-21502 Germany

Deformation twinning is an important mode of plastic deformation in gamma-TiAl based titanium aluminide alloys. The mechanism apparently compensates for the lack of independent slip systems that can operate at comparable stresses and, thus, supports the plasticity of polycrystalline materials. On the other hand, the octahedral planes of gamma -TiAl serve as slip planes, twin habit planes and cleavage planes. Thus, blocked slip may easily lead to fracture. This complex association of twinning and fracture is investigated in the present paper by conventional and high-resolution electron microscope investigations of twinning arrangements. The major areas of the study are: (i) twin nucleation and propagation, (ii) effects of solutes and precipitates on the kinematics and dynamics of twin propagation, (iii) association of mechanical twinning and fracture. The implications of the results will be discussed with respect to alloy design strategies towards improved mechanical properties.

Computational Aspects of Mechanical Properties of Materials: Nano-Scale and Meso-Scale Modeling

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Tuesday AM Room: 3012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: M. I. Baskes, Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; K. S. Ravi Chandran, University of Utah, Metallurgl. Engrg., Salt Lake City, UT 84112 USA

8:30 AM Invited
A Finite-Temperature, Dynamic Coupled Atomistic/Discrete-Dislocation Model: *W. A. Curtin*[1]; V. Shastry[1]; M. Dewald[1]; R. Miller[2]; [1]Brown University, Div. of Engrg., Providence, RI USA; [2]Carleton University, Dept. of Mechl. Engrg., Ottawa Canada

To handle plastic deformation at multiple scales, a computational method has been developed wherein atomistic and continuum regions communicate across a coherent boundary while dislocations seamlessly pass through the boundary, changing from an atomistic to continuum description or vice-versa. Here, the zero-temperature quasistatic method is extended to include finite-temperature molecular dynamics and dislocation dynamics. This is achieved through the use of "stadium boundary conditions" in the atomistic region that simultaneously provide a temperature reservoir for the atoms and prevent reflection at the atom/continuum boundary of energetic pulses emanating from the atomic region. The new method is applied to several basic problems, including brittle and ductile crack growth in Al, Ni, and Fe at finite temperatures, and dislocation pile-up/grain-boundary interactions. These examples demonstrate the power of integrated multiscale modeling for capturing complex mechanical phenomena in solids. Finally, progress on related multiscale models that embed quantum mechanics into the atomistic region and that connect discrete dislocation or

impurity models into continuum crystal plasticity or diffusion, respectively, is reported. [1]L. Shilkrot, R. M. Miller, and W. A. Curtin, J. Mech. Phys. Solids 52, 755 (2004).

9:05 AM
Modeling Elastic and Plastic Deformations of Nanocrystalline Materials: Peter Stefanovic[1]; Mikko Haataja[1]; *Nikolas Provatas*[1]; [1]McMaster University, Dept. of Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, Ontario L8S4L7 Canada

A continuum field theory approach is presented for modeling elastic and plastic deformation, free surfaces and multiple crystal orientations in systems with both hexagonal and cubic symmetry. The model is based on a free energy, and its dynamics incorporates both diffusive and elastic phenomena. By introducing a variable elastic time scale, we are able to maintain mechanical equilibrium while simulating microstructural evolution on time scales well beyond those accessible by conventional atomistic simulation methods (e.g. molecular dynamics). We apply this model to elucidate the role of dislocations and to study texture evolution during deformation of nanocrystalline materials.

9:25 AM Invited
Modeling Dislocation Behavior in Multi-Layer Nanostructures: *Nasr M. Ghoniem*[1]; [1]University of California, Mechl. & Aeros. Engrg., 420 Westwood Blvd., Los Angeles, CA 90095-1597 USA

Understanding dislocation behavior in multi-layer nanostructures is critical to the design of ultra-strong nano-structured materials, which exhibit significant ductility. We present the results of recent research on dislocation properties in anisotropic multilayer thin films utilizing several computational techniques. First, newly developed methods of dislocation dynamics in these systems will be introduced. Such methods are applied to delineate the various modes of material deformation as a function of the nanolayer size and the applied external stress. Second, Molecular Dynamics simulations will be presented, where we focus on the determination of core properties as dislocations approach interfaces, and on the critical Koehler barrier, which determines the resistance of the interface to dislocation transmission. Finally, an extension of the Peierls-Nabbarro dislocation core model is used together with Ab Initio calculations of the gamma surface to describe the structure of dislocation loop cores under a wide variety of conditions.

10:00 AM
Precipitate Shape and Coherency Loss Mechanisms in Ni-Ag Alloys: *Peihua Jing*[1]; Jae-Hyeok Shim[1]; Brian D. Wirth[1]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

In precipitate hardened materials, the strength and creep properties are controlled by dislocation interactions, which depend on the precipitate interfacial structure. We present the results of atomistic simulations specifically designed to investigate the precipitate shape, coherency loss and dislocation bypass mechanisms in a Ni-Ag alloy with a large lattice misfit. Embedded atom method interatomic potentials have been re-derived to accurately describe the elastic properties of Ni and Ag, and the Ni-Ag mixing enthalpy. The results provide insight into the precipitate coherency loss mechanisms and the effect of precipitate coherency on dislocation interaction and bypass.

10:20 AM Break

10:30 AM
Crack-Tip Fields from Discrete Dislocation Simulations: A Comparative Study with Continuum Mechanics: *Silvester J. Noronha*[1]; Nasr M. Ghoniem[1]; [1]University of California, Mechl. & Aeros. Engrg. Dept., 420 Westwood Plaza, #48-121, Los Angeles, CA 90095-1597 USA

Crack-tip plastic behavior is simulated using two dimensional discrete dislocation dynamics simulations. 3D mechanisms are incorporated in 2D dislocation dynamics simulations of crack-tip plasticity to include the effect of dislocation reactions like dislocation annihilation, dislocation junction formation, dynamic source generation, etc. We find that the size and shape of the equilibrated plastic zone compares well with the continuum mechanics predictions. The stress fields ahead of the crack along the crack plane are in excellent agreement with the small scale yielding models of crack tip plasticity. In contrast to the continuum models in which strain hardening exponent are an input parameter, in our simulations the hardening behavior evolves naturally. The variation of the hardening exponent with temperature is also recovered.

10:50 AM
Microstructural Effects in Modeling the Flow Behavior of Single Crystal Superalloys: *Y. S. Choi*[1]; T. A. Parthasarathy[1]; D. M. Dimiduk[2]; M. D. Uchic[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

The [001] orientation deformation of a single-crystal superalloy (CMSX-4) having a high volume fraction of regularly-arrayed cuboidal γ' precipitates was simulated using a non-local gradient-dependent plasticity for the γ matrix and the anisotropic-elasticity for the γ' precipitate. Unit-cell meshes were adopted for representation of the γ-γ' microstructure. Numerical studies focused on clarification of the effects of the γ-γ' geometry, the deformation constraint induced by the γ-γ' geometry, the applied BCs, and the flow properties of the γ and γ' phases on the macroscopic flow behavior. The simulated flow curves showed a softening behavior, which was accompanied by the organized massive plastic flow in the γ matrix and influenced by the thickness of γ-matrix channels, the flow property of the γ matrix, and the geometry of the γ'-precipitate edge. The simulation results also showed a tension-compression (T-C) asymmetry, which was related to the geometric constraint of the γ-γ' unit cell.

11:10 AM
Modeling the Tensile Properties of Alpha/Beta Titanium Alloys: *Sujoy Kar*[1]; Thomas Searles[1]; Eunha Lee[1]; Jaimie Tiley[2]; Gopal Babu Viswanathan[1]; Rajarshi Banerjee[1]; Hamish L Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Materials Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

The development of a set of computational tools that permit microstructurally-based predictions for the tensile properties of commercially important titanium alloys, such as Ti-6Al-4V, is a valuable step towards the accelerated maturation of materials. This paper will discuss the development of Fuzzy Logic and Neural Network Models based on Bayesian statistics to predict the yield strength, ultimate tensile strength and elongation of Ti-6Al-4V at room temperature. The development of such rules-based models requires the building up of extensive databases, which in the present case are microstructurally-based. The steps involved in database development include controlled variations of the microstructure using novel combinatorial approaches to heat-treatments, the use of standardized stereology protocols to rapidly characterize and quantify microstructural features, and mechanical testing of the heat-treated specimens. These databases have been used to train and test Neural Network models to predict the tensile properties. In addition, these models have been successfully used to identify the influence of individual microstructural features on the mechanical properties, consequently guiding the efforts towards development of more robust phenomenological models.

11:30 AM
Prediction of the Microstructure Parameters of Heavily Deformed OFHC Copper Using Artificial Neural Network: *Jamaa Bouhattate*[1]; Daudi R. Waryoba[1]; Peter N. Kalu[1]; [1]FAMU-FSU College of Engineering, Natl. High Magnetic Field Lab., Dept. of Mechl. Engrg., 2525 Pottsdamer St., Tallahassee, FL 32310 USA

An artificial neural network (ANN) model has been developed for predicting key microstructural parameters in heavily deformed OFHC copper. The initial phase of the model centered on the correlation between the processing parameters and the microstructure of the deformed and annealed OFHC copper. The material was heavily drawn to different strains at room temperature, and annealed for one hour at various temperatures. The ANN model was based on multilayer backpropagation neural network, and was trained by using experimental and numerical data. The input parameters were annealing temperatures and the processing strains, while the output parameters were orientation texture, microhardness, grain size and recrystallization temperatures. A good correlation was found between the predicted results and the original training data. Also, the model was used to predict properties of OFHC copper at different temperatures for different strains outside the training data set.

11:50 AM
Modeling the Mechanical Response of Open-Cellular 6101-T6 Foams at Ambient and at Intermediate Temperatures Through Finite Element and Density Compensation Methods: *Ian Ivan Nieves*[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92697-2575 USA

The tensile elastic response exhibited by open cellular 6101-T6 Al foams (Duocell) at 293K, 423K and 523K was simulated through finite element (FE) analysis. These results were compared with previously obtained experimental results and with values obtained from a density compensation (DC) model. FE analysis was performed with MSC Patran preprocessing software using Nastran post-processing. These simulations used a staggered cubic cell geometry that was comprised of hourglass-shaped struts with morphologies similar to those observed through optical and SEM metallography. The simulated cells approximated the relative density and dimensions possessed by the actual foam cells.

Longitudinal and transverse geometries were produced by scaling the appropriate cell dimensions by the experimentally determined aspect ratio. Tetrahedral meshes and numerical values for Al properties were then applied to the cell geometries. Boundary conditions appropriate to plane straining and loads equivalent to those at various stages of tensile testing were then applied to the meshes. Values of the resulting cellular elastic modulus (E*), were then compared with those obtained from the experimental data and with those derived from the DC model. The longitudinal results displayed significantly greater stiffness than the corresponding transverse results as expected. FE, DC and experimental results for 293K were all observed to correspond closely to each other. However, both the FE and experimental values displayed significantly greater stiffness than predicted by the DC values at 423K and 523K. The 523K FE and experimental results also differed greatly from the corresponding values obtained at other temperatures. Comparisons of FE results at all testing temperatures and inspection of the metallographic results indicated this discrepancy is due in part to the greater contribution of localized shear failure at elevated temperatures.

12:10 PM
Dynamic Behavior of IF Steel in the Two-Phase Conditions: *Janusz Majta*[1]; Anna K. Zurek[2]; [1]AGH University of Science and Technology, Metall. & Matl. Sci., Mickiewicza 30, Krakow 30-059 Poland; [2]Los Alamos National Laboratory, MST-8, MS: G755, Los Alamos, NM 87544 USA

There is increasing interest to study dynamic behavior of materials at high temperatures and non stable multi-phase conditions. In order to examine the failure behavior of materials tested in the two-phase conditions, the IF steel heated to austenite-ferrite region was employed. Present study is a continuation of previous analysis of C-Mn steel. Very unexpected results obtained in spall test of A36 steel forced us to continue investigations for the case of IF steel where role of carbon on mechanical response is minimalized. A series of high rate forming experiments are also performed on microalloyed and low carbon steels using SHPB. We evaluated the effects of variations in austenite/ferrite phase ratio during loading in the material flow stress and microstructure, followed by the optical, scanning and TEM microscopy analysis. The impact experiments resulted in obtaining various microstructures and localization of the deformation. However, in each test the absence of failure is observed after shock loading of material in austenite-ferrite two-phase condition. The importance of obtaining such results is very important for computer simulations of dynamic response of non stable two-phase materials. The results and conclusions of the study could be directly employed in estimating the different contribution to the shock-induced plastic deformation without failure in other two- or multi-phase materials.

Computational Thermodynamics and Phase Transformations: Grain Boundaries and Interfaces II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday AM Room: 3005
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Mark Andrew Miodownik, King's College London, Mechl. Engrg., Strand, London WC2R 2LS UK

8:30 AM Invited
Growth of Special Texture Components During Grain Growth: Simulation & Theory: *Anthony D. Rollett*[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The growth of minority texture components during the annealing of deformed metals is a phenomenon that has yet to be fully understood. Many theories have been advanced to explain the origins, for example, the cube component, {001}<100>, in fcc metals. One such theory concerns "microselection" whereby the cube component is favored during the early stages of recrystallization where coarsening takes place in the subgrain structure formed during recovery. To address this possibility, Monte Carlo simulation of grain growth is used to study the behavior of the cube component during grain growth in a polycrystal for which the dominant texture is representative of rolling textures in fcc metals. The cube component was treated as a special texture component, inserted into the microstructure by assigning near-cube orientations to particular grains. A sensitivity analysis varied such parameters as initial cube volume fraction, the grain boundary energy and mobility functions, spatial correlation of the special component, and rotations of the special component away from the exact cube position. The results indicate a strong tendency for the cube component to grow provided that a moderate level of anisotropy exists in the grain boundary energy and mobility. However, long-range correlations in orientation also play an important role in constraining growth in majority texture components, which is an effect known as orientation pinning.

9:00 AM Invited
An Inherently Discrete Approach for Modeling Microstructural Evolution with Elastic Effects: Cubic Anisotropy and Grain Coarsening Influenced by Elastic Stress: *Mark T. Lusk*[1]; T. Schacht[1]; E. Buddy Damm[2]; J. Wei[2]; Elizabeth A. Holm[3]; [1]Colorado School of Mines, Mechl. Engrg. Prog., Golden, CO 80401 USA; [2]The Timken Company, 1835 Dueber Ave. SW, PO Box 6932, Canton, OH 44706-0932 USA; [3]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185-1411 USA

Monte Carlo simulators like the classic Q-state Potts are now used routinely to consider grain coarsening, but coupling these simulations to an evolving elastic stress state has only received passing attention. Such a coupling is established in the present work. Bond stiffnesses in a harmonic lattice model are expressed in terms of the elastic stiffness elements of a tetragonal elastic solid with general tilt orientation. The harmonic model is then used to calculate the mechanical driving force associated with a change in orientation of one lattice site. This additional free energy is included in a Q-state Potts model. The resulting Hybrid Monte Carlo (HMC)model was used to investigate the development of grain texture in response to mechanical loading. The variation in grain orientation reduces with an applied uniaxial tension, and a relationship was established between appliedtraction and the rate at which this variation reduces.

9:30 AM
Grain Size Distribution in 3D Ideal Grain Growth: *Won Tae Kim*[1]; Seong Gyoon Kim[2]; [1]Cheongju University, Applied Sci. Div., 36 Naedok Dong, Cheongju 360-764 Korea; [2]Kunsan National University, Dept. of Matls. Sci. & Engrg., 68, Miryong Dong, Kunsan 573-701 Korea

Details on grain size distribution during the ideal 3D grain growth have been studied by using a phase field modeling. We developed a phase field model of grain growth, allowing unlimited number of grains with greatly improved computational efficiency. For accuracy test of the model showed that von Neumann's law on ideal 2D grain growth was completely satisfied irrespective of number of edges, indicating the model operates in a correct way. Then 3D computations on grain growth with several different initial grain size distributions have been performed on a 420x420x420 grid system. Irrespective of the initial grain size distribution, all computational results converges to a universal size distribution, which is exactly same as the prediction of grain growth model proposed by Hillert about 40 years ago. This study is the first numerical confirmation of Hillert's grain growth model.

9:50 AM
Effect of Magnetic Field on Fe-Si Phase Diagram and Grain Growth Behavior in Fe-1wt%Si: *Michael C. Gao*[1]; Tricia A. Bennett[2]; David E. Laughlin[2]; Anthony D. Rollett[2]; [1]Northeastern University, Dept. of Physics, Boston, MA 02115 USA; [2]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In parallel to the study of grain growth behavior of Fe-1wt%Si alloy under high applied magnetic fields, the effect of the magnetic field on the Fe-Si phase diagram in the Fe-rich corner is investigated. The magnetic Gibbs free energy of fcc austenite, bcc ferrite and ordered bcc phases are calculated using Weiss molecular field theory. The focus of the investigation is on the influence of magnetization and thermodynamics on grain boundary behavior as well as estimation of driving forces between different orientations.

10:10 AM Break

10:45 AM Invited
Energetics of Ge/Si(100) Island Formation: Role of Strain-Dependent Surface Energies: Oleg Shklyaev[2]; Matthew J. Beck[1]; *Mark D. Asta*[1]; Michael J. Miksis[2]; Peter W. Voorhees[1]; [1]Northwestern Uni-

versity, Dept. of Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]Northwestern University, Dept. of Engrg. Sci. & Applied Math., 2145 Sheridan Rd., Evanston, IL 60208 USA

The energetics underlying formation of nanometer-scale, pyramid-shaped Ge islands in vapor-phase, thin-film growth on Si(100) have been calculated using a computational approach combining first-principles results for strain-dependent surface and interface energies with an analytical continuum analysis of the epitaxial elastic strain energy. The calculations point to pronounced effects associated with the strain dependence of surface energies. Island stability is found to be governed in large part by the reduction in the wetting-layer excess energy arising from the compressive strain in the vicinity of the coherent islands. This strain leads to a substantial reduction in Ge(100) surface energy. The origin of the pronounced strain dependence of the surface energy will be discussed in terms of the nature of the rebonded surface reconstructions.

11:15 AM Invited

Atomistic Simulations of Reactive Wetting and Brazing: *J. J. Hoyt*[1]; E. B. Webb[1]; G. S. Grest[1]; D. R. Heine[1]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

Molecular dynamics (MD) simulations utilizing interatomic potentials of the embedded atom type have been used to study reactive wetting in the eutectic Ag-Cu system. For pure liquid Ag on Cu substrates, the radius of the drop, R(t), is found to spread with time according to a power law of the form R(t)~t^1/2. This dissolution controlled regime is compared with the case of Ag-Cu binary droplets on Cu where a lower power law exponent emerges at late times. The slower kinetics suggests a possible crossover to a diffusion controlled reactive wetting mechanism. In addition, brazing has been studied for the case of liquid Cu infiltrating a small channel of solid Ni base metal. The rate of infiltration, the development of the equilibrium contact angle and the dissolution of Ni are monitored as a function of time. For both reactive wetting and brazing the dissolution rate of the solid plays a key role in the kinetics. A model for the dissolution rate is developed based on previous models for the crystallization of pure metals as a function of undercooling.

11:45 AM

Continuum Simulation of Faceted Thin Film Growth: *Danxu Du*[1]; David J. Srolovitz[1]; Michael E. Coltrin[2]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., D214 E-Quad, Olden St., Princeton, NJ 08540 USA; [2]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

Many semiconductor, ionic and oxide thin films grow in a facetted manner. In some cases, the facetted nature of the growth can be used to manipulate defect structures and morphology. We describe a level set formalism for modeling faceted growth based upon knowledge of the growth rate as a function of surface orientation. Simple rules emerge for the growth of both convex and concave shapes. Interestingly, for convex growth, the morphology is dominated by the fastest growing facets, while in concave growth the slower facets are more important. While corners/edges are sharp in convex growth, rounded corners/edges are more common in concave growth. We apply the new level set method for facetted film growth to the case of Epitaxial-Lateral-Overgrowth (ELO) of GaN.

Converter and Fire Refining Practices: Process Improvements and Anode Casting

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee

Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Tuesday AM Room: 2016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Rob L. Stephens, Teck Cominco Metals Ltd, Lead Products, Trail, BC V1R 4L8 Canada; Robert Matusewicz, Ausmelt Limited, Dandenong, Victoria 3175 Australia

8:30 AM

Anode Casting Operation, Process Improvements and Anode Quality: *Victor Raul Alarcon*[1]; [1]Southern Peru Copper Corporation, Ilo Refinery, Caminos del Inca 171, Pampa Caliche Km.9, Surco-Lima, Ilo-Moquegua Peru

The Ilo Refinery was commissioned in 1975, with a cathode production capacity of 150,000 tpy. In 1994, Southern Perú Copper Corporation (SPCC) purchased the Refinery from Minero Perú SA. Subsequently, in November 1999, Grupo Mexico acquired control of Southern Perú. The anode production facility is located at the Ilo Refinery. A number of effective operating and quality control practices have been developed that have improved anode quality, as well as increasing anode production capacity to 350,000 tpy. These, and other optimization actions have allowed for a current density increase from 213 A/m2 to 275 A/m2, while maintaining current efficiency at 98% and sustaining the high quality of the cathode. The paper will outline the scope of operational practices in the Anode Plant and provide some discussion on the Ilo Smelter modernization project, and the resulting impact on the existing anode plant.

9:00 AM

The Application of 'Six Sigma' Methodologies to Improve the Quality of Converter Slag at Atlantic Copper: Jesús Contreras[1]; *Rafael Fernández-Gil*[1]; *Miguel Palacios*[1]; [1]Atlantic Copper, S.A., Smelter Ops., Av Francisco Montenegro, s/n., 21001 Huelva Espana

Within its program for continuous improvement and cultural change, Atlantic Copper has installed the Six Sigma methodology as a basic tool for the improvement of management techniques in all its production centres. Following an implementation period, the methodology is widely used for analysis and improvement, not only in the fields of production and maintenance, but also in other areas of the business such as supplies or energy management. The present paper describes, phase-by-phase, the project that was developed to improve the quality of converter slag through the application of Six Sigma techniques, from the initial set up to the final results obtained.

9:30 AM

New Casting Moulds for Anode Copper: *Torben Edens*[1]; *Dirk Hannemann*[1]; [1]Norddeutsche Affinerie, Copper Production & Recycling, Hoverstrasse 50, Hamburg D-20539 Germany

Norddeutsche Affinerie's (NA's) primary smelter continually increased production in the past decade. The smelter's concentrate capacity was expanded from 500,000 in 1993 to 1,000,000 t/a dry concentrate in 2000 and the goal of the present upgrade is 1,150,000 t/a. These goals were reached without erecting new equipment in the anode area, but instead by refitting the existing plant. The life-span extension of the casting moulds was absolutely necessary to increase the casting performance from 260,000 to 450,000 t anodes per year on a single casting wheel. Different mould manufacturing processes and alloys were tested in order to find the optimal mould. In the past the moulds were cast directly, now the anode mould is milled into NA copper cakes. Accompanying measures taken were the shortening of maintenance and repair times as well as improvements in the refining furnace equipment.

10:00 AM Break

10:15 AM

Hoboken Converter Performance Improvements at the Phelps Dodge Miami Smelter: *Ovidiu Pasca*[1]; Vlad Ushakov[1]; Eugene Welker[1]; [1]Phelps Dodge, Miami Smelter, PO Box 4444, Claypool, AZ 85532 USA

The Phelps Dodge Miami Smelter utilizes four Hoboken converters to process molten matte from the Isasmelt vessel. This paper presents past and present improvements in operation, maintenance and process optimization, based on data collection, which has led to increased converter campaign life and copper throughput.

10:45 AM

Productivity Increase in a Peirce-Smith Converter Using the 'COP KIN' and 'OPC' System: *Thomas Prietl*[1]; Andreas Filzwieser[2]; Stefan Wallner[3]; [1]RHI Non-Ferrous Metals Engineering GmbH and Christian Doppler Laboratory for Sekundary Metallurgy of the Non-Ferrous Metals, Non-Ferrous, Magnesitstrasse 2/RHI, Franz Josef Strasse 15/CD-Lab., Leoben 8700 Austria; [3]RHI Non-Ferrous Metals Engineering GmbH, Non-Ferrous, Wienerbergstrasse 11, Vienna 1100 Austria

The use of gas stirring systems through the bottom of a furnace in the copper industry is common for anode and holding furnaces. The first implementation of a gas stirring COP KIN® system in a Peirce-Smith converter was in Sweden at the New Boliden smelter in Rönnskar. A decrease in process time and a decrease of the oxygen content in the blister copper were observed. To determine the effects of the gas stirring system and the process endpoint, an optical production control 'Semtech OPC system' was used. The light emission of the converter flame as an optical process parameter provides qualitative on-line process information. This information is also used for endpoint determination of the slag making process, on-line control of iron content in white metal, quality control of slag etc. The results, ben-

efits and risks of using the COP KIN® and OPC system for a Peirce-Smith converter are reported.

11:15 AM
Pyrometallurgical Refining of Copper in an Anode Furnace: H. Antrekowitsch[1]; Chr. Wenzl[1]; *I. Filzwieser*[2]; D. Offenthaler[2]; [1]University of Leoben, Christian-Doppler Lab. for Secondary Metall. of Non-Ferrous Metals, Franz-Josef-Strasse 15, A-8700 Leoben Austria; [2]University of Leoben, Dept. of Nonferrous Metall., Franz-Josef-Strasse 15, A-8700 Leoben Austria

The decreasing quality of the input materials in copper recycling leads to a higher content of impurities in the anode copper. An improvement of the pyrometallurgical refining process is therefore necessary to produce high quality anodes for the copper refining electrolysis. The behaviour of the most important accompanying elements (e.g. nickel, tin, lead, zinc etc.) at different reaction conditions has to be investigated in order to improve the metal/slag reactions as well as the vaporisation by selective oxidation in the anode furnace. Therefore the thermodynamic conditions like reaction order and the activity coefficients at the copper refining process have to be known for the optimisation of this technique. Additionally the interaction between different elements has been investigated as a function of the temperature, the content of the elements and the slag composition especially for nickel. These investigations were done at the Christian Doppler Laboratory for Secondary Metallurgy of Nonferrous Metals.

Extractive Metallurgy: Recycling and Waste Minimization

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Tuesday AM Room: 2018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Junji Shibata, Kansai University, Dept. of Cheml. Engrg., Osaka 564-8680 Japan; Boyd R. Davis, Kingston Process Metallurgy Inc., Kingston, Ontario K7P 1S6 Canada

8:30 AM
Mercury Recovery in Autoclave Effluents from Refractory Ore Treatment: *Rainer Bauder*[1]; Steven F. McGrath[1]; Katherine Milidakis[1]; [1]MR3 Systems, Inc., 435 Brannan St., Ste. 200, San Francisco, CA 94107 USA

Refractory Gold deposits are often associated with mercury as contamination. When those ore types undergo oxidation in an autoclave system, a significant mercury level appears in the wash waters and effluents. The paper describes a hydrometallurgical approach to remove mercury directly in the autoclave effluent. Because of the typical very strongly acidic conditions caused by the production of sulfuric acid during the oxidation of the sulfides, a silica based chelating absorbent is utilized to extract the mercury efficiently, providing a way to remove it effectively for the further treatment necessary. This novel approach allows selective isolation of mercury in the high metal environment, and therefore addresses the environmental issues associated with it.

8:55 AM
Metallic Secondary Raw Materials Recycling Strategy in Serbia: *Sasha D. Djokic*[1]; [1]MAG, Zeleni bulevar bb, BOR 19210 Serbia

In developed countries, the difference between demanded and produced quantities of non-ferrous and rare metals are compensated by secondary raw materials recycling and reprocessing of by-products from both ferrous and non-ferrous extractive metallurgy. Utilization of non-ferrous and rare metals from the metallic secondary raw materials is very significant, both for recovery of the metal value and contribution to the environment protection. Similar to all non-developed countries, the extractive metallurgy in Serbia was based on available natural resources and not so strict environment protection regulations, which was either insufficient or completely absent. Today, Serbia is at the beginning of development of efficient and economical technical and technological solutions in order to "ecologize" technogenic systems by waste materials, secondary raw materials and degraded soils regeneration (by recycling and revitalization). Several metallurgical technogenic systems in Serbia, together with secondary raw materials classification according to their potentiality for metal extraction by using the existing technologies, determination of their structure, composition, and expected economical and ecological effects of recycling, are discussed in this paper.

9:20 AM
Valorization of Solid Wastes as Sorbents for Heavy Metals: Luciano R.G. Santos[1]; Rafael Falco Rodrigues[1]; *Versiane Albis Leao*[1]; Eucler B. Paniago[2]; Vagner L. Botaro[2]; [1]Universidade Federal de Ouro Preto, Metallurgl. Matl. Eng., Praça Tiradentes, 20, Centro, Ouro Preto, MG 35400-000 Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Chmst., Inst. de Ciências Exatas e Biológicas, Campus Morro do Cruzeiro, s.n., Ouro Preto, MG 35400-000 Brazil

The majority of the industrial activities produce some kind of solid wastes. In this work two industrial residues (spent zeolite and wood sawdust) were chemically modified to increase their heavy metals sorption capacities. Manilkara longifolia (a Brazilian plant known as parajú) sawdust was first treated with NaOH and modified with different citric acid concentrations (0.6-1.2mol/L) at 120°C, for 90 min. Infrared spectroscopy confirmed the presence of carboxylate groups in the wood structure as result of their mixing. Similarly, spent zeolites were also submitted to a chemical treatment (NaOH) to remove carbon deposited during oil cracking and to recover its sorption capacity. Batch experiments carried out at ambient temperature showed that both materials present sorption capacity for copper and cadmium comparable to weak acid ion exchange resins. A maximum of 78mg-Cd/g and 59.7mg-Cu/g as well as 29.9mg-Cd/g and 14.6mg-Cu/g were observed for the sawdust and the zeolite, respectively. The results were modeled according to the Freundlich and Langmuir equilibrium isotherms and compared with results of other studies.

9:45 AM
Aqueous Oxidative Precipitation of Manganese by SO2/O2: *Vincent Menard*[1]; George P. Demopoulos[1]; [1]McGill University, Mining, Metals & Matls. Dept., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

In many hydrometallurgical processes, manganese is an impurity that needs to be removed from solution prior to metal recovery. The simplest method of removal is precipitation as hydroxide, i.e. Mn(OH)2, by pH adjustment. However, this method is not selective as most of the other elements present in the solution precipitate as well. An alternative method of manganese removal is by oxidative precipitation. This method consists in oxidizing Mn2+ to Mn3+/Mn4+ which precipitate readily as hydrous manganese oxide compounds. The purpose of the present work is to remove selectively manganese from a zinc-rich solution using a gas mixture of SO2/O2 as oxidizing agent. Several semi-batch experiments were performed where the effects of redox potential, SO2/O2 ratio, mixing intensity, etc. were investigated. These tests were followed by setting-up and running a multi-reactor continuous circuit employing pH/ORP control and product recycling in an effort to improve purity and solid-liquid separation of the precipitates.

10:10 AM Break

10:25 AM
Aqueous Processing of Lithium Cobalt Ion Battery Scrap for Cobalt Recovery: *Raj P. Singh*[1]; Thomas A. Wolfe[1]; [1]Chemicals & Powders R&D, Precision Matl. & Components Div., Osram Sylvania, Towanda, PA 18810 USA

Cobalt is a strategic metal, which is used in many diverse industrial and military applications such as super alloys, magnets, hard metals (carbide), catalysts, colors, sulfate, batteries, tires and paint driers. Cobalt is mainly produced from byproducts of other more abundant metals. For example, more than one-half of the world's supply of cobalt is produced from byproduct of copper mining and refining in African countries Zaire and Zambia. Cobalt production in most other countries is carried out from byproducts of nickel mining and / or refining. The United States is the world's largest consumer of cobalt, and is 85% dependent on imports for its supply of primary cobalt. About 15% of U.S. cobalt consumption comes from recycled scrap. Since their commercialization in 1991, lithium-cobalt batteries have been widely used in electronic applications. These batteries have certain advantages over nickel-cadmium and nickel-metal hydride batteries and due to this reason, in near future; their market share is expected to grow large. Current research on these batteries is concerned on the development of large sized batteries for their use in electric vehicles. This is a fast developing area and will result into appreciable growth of such batteries. The production and use of large sized lithium-cobalt batteries for electric vehicles applications would generate large amount

of primary and secondary scrap, which is expected to be a potential feed source for cobalt. In this paper we will present characterization of lithium cobalt oxide battery scrape from industrial sources and aqueous processing of these scraps for cobalt via a cobalt hexammine chloride process. Chemistry, efficiency and accountability of various steps of the method for cobalt processing will be discussed.

10:50 AM
The Behavior of Heavy Metal, Alkali and Chloride Derived from Waste Sludge on the Cement Industrial Process: *Kwang-Suk You*[1]; Ji-Whan Ahn[1]; [1]Korea Institute of Geoscience & Mineral Resources, Taejeon 305-350 Korea

The purpose of this study is to use the waste sludges generated at Pohang Iron & Steel Co. Ltd.(POSCO) by the raw materials of ordinary portland cement(OPC). To evaluate the possibility of its application, in this study, it was investigated that the behavior of heavy metal, alkali and chloride generated from waste sludge of steel making industry when it is used as raw mixture for cement. The waste sludge was mainly composed of CaO, SiO2, Al2O3, and Fe2O3, similar as the main component of OPC. It was reported that alkali and chloride occur various troubles in the Kiln, and heavy metals affected the properties of cement clinkers. This study discussed the hazardous effects by them and the characteristic of cement manufactured from waste by POSCO sludge. From the result of study, it was concluded that POSCO sludge can be used as the source materials of iron and calcium oxide.

11:15 AM
Tin and Zinc Recovery and Pellet Preparation from Iron Ores Containing Tin and Zinc: *Yuanbo Zhang*[1]; Yufeng Guo[1]; Guanghui Li[1]; Yongbin Yang[1]; Zhucheng Huang[1]; Xiaohui Fan[1]; Tao Jiang[1]; [1]Central South University, Sch. of Resources Procg. & Bioengrg., Changsha, Hunan 410083 China

The complex iron ores containing tin, zinc are typical refractory ores. Great reserves of the ores are found in China and have not been utilized efficiently. A new process of tin and zinc recovery and pellet preparation from iron ores containing tin and zinc by controlled roasting is developed in this investigation. The results of compression strength of product pellets of 2573 N/P, the volatilization of tin and zinc of 91.49% and 82.22% respectively, are achieved for a concentrate containing 0.39% tin and 0.28% zinc. The compression strength and chemical compositions of the product pellets meet the requirement of blast furnaces. Volatilization mechanism of tin and zinc are investigated. Thermodynamic study shows that the reduction balance curves of SnO2 and SnO are proximate under the condition of the existence of carbon, and the standard free enthalpy of FeO and SnO2 is also approachable. Therefore, it is difficult to realize the effective volatilization of SnO by selective reduction. But SnO2 and SnO will form SnCl4 or SnCl2 and can be easily volatilized from the ores if they are transferred into their chlorides.

Friction Stir Welding and Processing III: Friction Stir Processing

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday AM Room: Nob Hill C/D
February 15, 2005 Location: San Francisco Marriott

Session Chair: Murray W. Mahoney, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA

8:30 AM Keynote
Friction Stir Processing for Microstructure Modification of NiAl Bronze: *Terry R. McNelley*[1]; Keiichiro Oh-ishi[1]; Alex P. Zhilyaev[1]; Robert A. Williams[1]; [1]Naval Postgraduate School, Mechl. & Astronautical Engrg., 700 Dyer Rd., Monterey, CA 93943-5146 USA

Friction stir processing (FSP) has been applied to as-cast NiAl bronze materials, which are widely used for marine components, in order to selectively convert the microstructures in the near-surface layers from a cast to a wrought condition. The physical metallurgy of NiAl bronze is complex and interpretation of the effects of FSP on the homogenization and refinement of microstructure has required detailed analysis by optical and electron microscopy methods. Peak temperatures in the stir zone due to thermomechanical cycle of FSP have been estimated from microstructures and compared to model predictions. Isothermal hot rolling and hot compression testing has been employed to confirm microstructure-based estimates of stir-zone peak temperatures. The variation of mechanical properties was assessed by use of miniature tensile samples and correlated with microstructure for samples from stir zones as well as hot rolled material. Extension of these results to multi-pass processing will be presented.

9:00 AM
Friction Stir Processing of Ferrous Alloys for Increased Wear Resistance: *Uma Ramasubramanian*[1]; Glenn J. Grant[2]; Glen A. Stone[1]; William J. Arbegast[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD USA; [2]Pacific Northwest National Laboratories, Matls. Procg. & Performance, 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

The Advanced Materials Processing Center (AMP) at the South Dakota School of Mines and Technology in conjunction with the Pacific Northwest National Laboratories is developing methods to stir micron size TiB2 particles into the surface of gray cast iron. Surface enrichment with ceramic particles may results in improved brake materials for heavy-duty brake systems. The objective is less high temperature fading and increase in wear resistance. The present work investigates the distribution of particles as a function of the pin tool design, processing parameters, powder size, slit geometry and position.

9:20 AM
Incorporating Titanium Powder to Create In-Situ Composites on the Surface of Cast Iron via Friction Stir Reaction Processing (FSRP): *Raja L. Veluchamy*[1]; Glenn J. Grant[2]; Glen A. Stone[1]; William J. Arbegast[1]; [1]South Dakota School of Mines and Technology, AMP, 501 E. St. Joesph St., Rapid City, SD 57701-3995 USA; [2]Pacific Northwest National Laboratories, Matls. Procg. & Performance, 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

The Advanced Materials Processing Center (AMP) at the South Dakota School of Mines and Technology in conjunction with the Pacific Northwest National Laboratories is exploring in-situ chemical reactions during FSP. The titanium/graphite system is thermodynamically favorable so as to produce TiC during FSP. It is expected that excess carbon (graphite) contained within gray cast iron and Ti powders stirred into the material by FSP, will form hard, wear resistant, TiC compounds dispersed within matrix. This research is designed to test the feasibility of initiating and controlling this chemical reaction during FSP. The end point goal of the program is to increase the friction on the surface of cast iron brake disks and to improve the wear characteristic of brake rotors, drums and pads. Brake cast iron typically has type A graphite, with a pearlitic matrix and low ferrite and carbide content.

9:40 AM
Friction Stir Technology for Superplastic Forming: Sky5083 Alloy: *Alok Vats*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; Darrell R. Herling[3]; Glenn J. Grant[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [3]Pacific Northwest National Laboratory, Matls. Sci. Div., K2-03, 902 Battelle Blvd., Richland, WA 99352 USA

The scientific understanding of superplastic forming (SPF) has evolved immensely over the last decade driven primarily by the need for economic competitiveness compared to metal stamping methods. Much research has focused on aluminum's thermomechanical processing schedules to produce microstructures that exhibit superplastic behavior. It is agreed that for aluminum-magnesium alloys an equiax fine-grain microstructure is the most desirable. Typical methods of processing aluminum sheet for SPF is by a series of hot and extensive cold rolling schedules. Although a robust method sheet materials, it requires either starting with large billet cross sections or ending up with a limited gage thickness to achieve the required strain energy. Friction Stir Processing (FSP) is an emerging processing method that has been proved can produce a fine-grain structure in most aluminum alloys. The objective was to develop FSP parameters for both linear and spot welding to produce optimum microstructures and behavior.

10:00 AM
High Strain Rate Superplastic Properties of 7075 Aluminum via Multiple Pass Friction Stir Processing: *L. B. Johannes*[1]; I. Charit[1]; R. S. Mishra[1]; [1]University of Missouri, Dept. of Matls. Sci. & Engrg., Ctr. for Friction Stir Procg., Rolla, MO 65409 USA

Friction stir processing (FSP) leads to fine grained microstructure in commercial 7075 aluminum plates. In this study, the effects of performing multiple, overlapping passes of FSP on 7075 Al were examined. Samples ranging from one to four passes and a nine pass sample were used with a 6.4 mm separation between passes (~50% overlap). In the temperature range of 400°C to 490°C, it was shown that the area of overlapping passes exhibits elongations >200% at the commercially important strain rate of $1x10^{-2}$ s^{-1}. In the nine pass sample, elongations averaging greater than 550% were found at 490°C at this strain rate. The unprocessed 7075 exhibited elongations <200% over the same range of temperatures and hence, no superplastic properties. Strain rate sensitivities of regions in the various passes were also found to be around 0.5, indicating grain boundary sliding dominant mechanism. These results show the potential for using FSP to obtain superplastic properties in commercial aluminum alloys.

10:20 AM Break

10:40 AM Invited
Thick Plate Bending of Friction Stir Processed Aluminum Alloys: *Murray W. Mahoney*[1]; Christian Fuller[1]; Mike Miles[2]; William Bingel[1]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]Brigham Young University; Coll. of Engrg. & Tech. 265 CTB, Provo, UT 84602 USA

Friction stir processing (FSP) was used to modify the surface microstructure in thick plate 7050-T7451 aluminum to enhance room temperature bending. Plate, 254 x 216 x 50mm thick, was friction stir processed on the pre-tensile surface to shallow and different depths (3.1 and 6.3 mm) using different approaches, i.e., the FSP raster included both linear and spiral patterns. Following FSP, plates were bent at room temperature to different degrees including a compound curvature. Results illustrate the ability to significantly enhance room temperature formability via FSP. To illustrate the extreme capability of this technique, similar processing was applied to15cm thick 6061 aluminum. In this case, FSP was applied to a depth of 6mm and this very thick plate bent to 30º at room temperature without failure. Thick plate bending can be used to substitute for welding of corner structures or to shape plate for machining of a monolithic structure where a plate thickness is not attainable.

11:00 AM
Friction Surface Reaction Processing on Aluminum Substrates: Stanley M. Howard[1]; *Bharat K. Jasthi*[1]; William J. Arbegast[2]; Glenn J. Grant[3]; Darrell R. Herling[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA; [3]Pacific Northwest National Laboratory, Matls. Sci. Div., K2-03, 902 Battelle Blvd., Richland, WA 99352 USA

Friction Stir Processing (FSP) is a variation of Friction Stir Welding that uses a spinning tool to modify the material through severe plastic deformation. Recent work has shown that surface modified regions in some aluminum materials can contain increased quantities of fine-grained silicide phases stabilized during nugget formation. Plastic work energy combined with the "new" surface created during shearing may promote some solid-state reactions. The work outlines results of several metallurgical systems to determine if energetic solid-state reactions during FSP can be sustained to produce novel surface materials and microstructures. The potential exists to form regions enriched in nitrides, borides or carbides in-situ by stirring elemental or oxide powders into the surface of monolithic materials. A goal of this work was to determine: 1) the efficacy of FSP mechanical energy in initiating and maintaining these energetic reactions, 2) product distribution and morphology, and 3) reaction control.

11:20 AM
The Effect of Friction Stir Processing and Subsequent Rolling on the Superplastic Behaviour of Aluminium Alloys: *Stavros Katsas*[1]; Graham Todd[1]; Martin Jackson[1]; Roger Grimes[1]; Richard J. Dashwood[1]; [1]Imperial College London, Dept. of Matls., S. Kensington Campus, London SW7 2AZ UK

Friction stir processing (FSP) can dramatically reduce grain size conferring excellent superplastic behaviour in certain aluminium alloys. FSP of thick plate followed by rolling to sheet could potentially be used as a method to improve performance of established superplastic alloys or to induce superplastic behaviour in alloys not normally associated with this phenomenon. An extruded Al-Mg-Zr alloy was FSP?d prior to rolling to sheet. The development of microstructure and superplastic behaviour was characterised using a combination of light, scanning, orientation imaging, and transmission microscopy, coupled with hot uniaxial tensile testing. FSP transformed the coarse, highly textured extruded, structure into a very fine (approximately 600nm) randomly orientated, equiaxed material. The structure of the material after rolling and thermal treatment was complex and in certain cases significant grain coarsening resulted. A detailed investigation of factors responsible for this will be described.

11:40 AM
Influence of Prior Microstructure on Friction Stir Processing of Cast A356 Plates: *S. R. Sharma*[1]; R. S. Mishra[1]; [1]University of Missouri, Dept. of Matls. Sci. & Engrg., Ctr. for Friction Stir Procg., Rolla, MO 65409 USA

Friction stir processing (FSP) leads to microstructural refinement and, in case of a cast microstructure, leads to significant improvement in static and dynamic mechanical properties. The present work investigates the influence of prior heat treatment on the microstructure and mechanical properties of cast A356 after FSP. Cast plates were friction stir processed in as cast, solution treated and T6 conditions. Influence of prior heat treatment on forces during FSP and mechanical properties after FSP was evaluated.

12:00 PM
Superplastic Forming of Friction Sir Welded Ti-6-4 Sheet: *Glenn J. Grant*[1]; D. Sanders[2]; A. P. Reynolds[3]; Wei Tang[3]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA; [2]Boeing, Seattle, WA 98124 USA; [3]University of South Carolina, Columbia, SC 29208 USA

Titanium 6-4 alloy sheets, 2 mm thick, were friction stir welded in a square butt configuration. The friction stir welded sheets were subsequently characterized via optical and scanning electron microscopy. Elevated temperature, transverse (loading perpendicular to the welding direction), tensile tests were performed to assess the superplastic performance of the sheet and the weld regions. After establishing parameters for superplastic forming of the welds, pans were formed from the welded sheets. Superplastic behavior of the sheet was retained or enhanced in the welds. In this paper, weld and base metal microstructure and superplastic performance of the welded structure will be presented.

Frontiers in Solidification Science: Nucleation

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday AM Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: John Perepezko, University of Wisconsin, WI USA; Mark D. Asta, Northwestern University, Evanston, IL 60208 USA

8:30 AM Invited
Coupled Processes in Nucleation: *Kenneth F. Kelton*[1]; [1]Washington University, Dept. of Physics, Campus Box 1105, One Brookings Dr., St. Louis, MO 63130 USA

It is becoming increasingly clear that coupling plays an important role in many nucleation processes. Generally, this cannot be explained quantitatively within the commonly used classical theory of nucleation. For example, often the initial and final phases have different chemical compositions, requiring a new model that couples the fluxes of long-range diffusion and interfacial attachment. Recently, we have identified a case of order parameter coupling for nucleation from an undercooled metallic liquid. From high-energy x-ray and undercooling studies of an electrostatically-levitated Ti-Zr-Ni liquid, we demonstrated that fluctuations in the short-range order of the liquid determined the primary crystallizing phase, linking the local structural order parameter of the initial phase with the nucleation barrier. These and other examples of coupled processes in nucleation are discussed. The implications of coupling on nucleation theory are discussed. Supported by NASA under contract NAG 8-1682, and by the NSF under grant DMR 03-07410.

9:05 AM Invited
Short-Range Order in Undercooled Metallic Melts and its Influence on Solidification: *Dirk Holland-Moritz*[1]; [1]DLR, Inst. fuer Raumsimulation, Linder Hoehe, Koeln D-51147 Germany

This work presents investigations on the short-range order of deeply undercooled liquids of the pure elements (Ni, Co, Zr, Ti and Fe) and of

alloys forming quasicrystalline or polytetrahedral phases ($Al_{60}Cu_{34}Fe_6$, $Al_{65}Cu_{25}Co_{10}$ and $Al_{13}(Fe,Co)_4$). The liquids were containerlessly processed and undercooled by application of the electromagnetic levitation technique, which was combined with elastic neutron scattering at the Institute-Laue-Langevin (ILL) in Grenoble and with energy dispersive diffraction of synchrotron radiation at the European Synchrotron Radiation Facility (ESRF) in Grenoble in order to determine the structure factors of the liquids as a function of the temperature. For all of the investigated metallic liquids the experimental data are well described if an icosahedral short-range order is assumed to prevail in the melt. This icosahedral short-range order is observed already at temperatures above the melting temperature and becomes more pronounced if the melt is undercooled. The impact of the icosahedral short-range order in the undercooled melt on the energy of the interface between the melt and nuclei of solid phases of different structures is discussed. This structure-dependence of the solid-liquid interfacial energy decisively influences the solidification behavior of the liquids.

9:40 AM
Do Icosahedral Phases Melt Into Icosahedral Liquids? A Combined Experimental and Theoretical Study of Liquid CdYb: *M. J. Kramer*[1]; D. J. Sordelet[1]; Y. Ye[1]; T. A. Lograsso[1]; Ulf Dahlborg[2]; J. R. Morris[3]; [1]Iowa State University, Ames Lab., 37 Wilhelm, Ames, IA 50011 USA; [2]Ecole des Mines, LSG2M, UMR 7584, Parc de Saurupt, Nancy 54042 France; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

There has been a significant amount of recent research arguing that some simple metals, i.e., Ni, show icosahedral structure in the undercooled liquid. Other indirect evidence for icosahedral order in liquids is based on fast nucleation rates of certain icosahedral structures and their approximants. Local icosahedral order is also indicated by the formation of metastable quasicrystalline phases in some devitrified metallic glasses. We present results from diffraction experiments and ab initio calculations, that demonstrate that the converse does not always hold true: Cd5.7Yb forms a binary, congruently melting quasicrystal which melts into a non-icosahedral liquid. This provides an explanation for the observed slow nucleation rate of this stable quasicrystal. These results are still in agreement with Frank's hypothesis; the difference in structure from the liquid and solid phases indicates a large crystal-melt interfacial free energy, resulting in a large nucleation barrier. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract W-7405-ENG-82 with Iowa State University of Science and Technology and contract DE-AC05-00OR-22725 with UT-Battelle, LLC.

10:00 AM Invited
Heterogeneous Grain Initiation in Solidification: *A. L. Greer*[1]; T. E. Quested[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Grain refinement in casting of aluminum alloys has recently been analyzed by assuming that the barrier for grain initiation on an inoculant particle is that for free growth from the particle rather than for nucleation of the solid. Adsorption effects can stabilize a thin layer of solid on the particle even above the liquidus temperature. In modeling so far undertaken, the approximation has been made that grain initiation depends only on undercooling, with no stochastic behavior. In this paper the nature of the barrier for grain initiation is examined quantitatively. The relative importance of deterministic and stochastic elements of grain initiation is explored as a function of inoculant particle size and melt undercooling. The effect of dormant solid on the particles is taken into account. The nature of the adsorption and templating on the inoculant surface are also considered.

10:35 AM Break

10:45 AM Invited
Atomistic Simulations of the Nucleation of Nickel: *Michael I. Baskes*[1]; Frank J. Cherne[1]; Ricardo B. Schwarz[1]; S. G. Srinivasan[1]; William Klein[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Boston University, Dept. of Physics, Boston, MA 02215 USA

The dynamics of homogeneous nucleation and growth of crystalline nickel from its super-cooled melt is examined during rapid quenching using molecular dynamics and a modified embedded atom potential. The character of the critical nuclei of the crystallization transition is examined using common neighbor analysis and visualization techniques. We find that the critical nucleus consists of a small number of atoms arranged in a stacking of planar structures. There is two-dimensional triangular order within each plane but the planes are randomly stacked. Because the stacking is not fcc (nor hcp), our results do not support classical nucleation models. They agree, however, with recent non-classical nucleation models (Shen and Oxtoby (1996); Klein and Leyvraz(1986)). Three-dimensional order develops after the nucleus grows and becomes supercritical. W. Klein and F. Leyvraz, Phys. Rev. Lett. 57, 2845 (1986). Y. C. Shen and D. W. Oxtoby, Phys. Rev. Lett. 77, 3585 (1996). This work was supported at Los Alamos National Laboratory by the U. S. DOE under contract W-7405-ENG-36.

11:20 AM Invited
Phase Field Theory of Nucleation and Polycrystalline Solidification: *László Gránásy*[1]; Tamás Pusztai[1]; James A. Warren[2]; Jack F. Douglas[3]; [1]Research Institute for Solid State Physics and Optics, POB 49, Budapest H-1525 Hungary; [2]National Institute of Standards and Technology, Metall. Div., Gaithersburg, MD 20899 USA; [3]National Institute of Standards and Technology, Polymer Div., Gaithersburg, MD 20899 USA

A phase field theory of crystal nucleation and polycrystalline growth is presented. The model is tested for crystal nucleation in the hard sphere system. Evaluating the model parameters from molecular dynamics simulations, the phase field theory predicts the nucleation barrier accurately. The formation of spherulites is discussed in an extension of the model that incorporates branching with definite orientational mismatch induced by a metastable minimum in the orientational free energy. The mechanism of polycrystalline growth is the quenching of orientational defects (grain boundaries) into the solid due to a reduced ratio of rotational and translational diffusional coefficient expected at high undercoolings. It will be demonstrated that a broad variety of spherulitic patterns can be recovered by changing only a few model parameters (anisotropy, free energy of metastable minimum, branching angle, orientational mobility).

11:55 AM Invited
Growth Front Nucleation: A Mechanism for Polycrystalline Growth: *James A. Warren*[1]; Laszlo Granasy[2]; Tamas Pusztai[2]; Jack F. Douglas[1]; [1]National Institute of Standards and Technology, CTCMS/Metall., 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20852 USA; [2]RISSPO, Budapest Hungary

The formation of a polycrystal is usually considered to occur either by the impingement of nucleating grains in a liquid (equiaxed grains), or via the nucleation of columnar grains on a surface. In this presentation we consider the formation of a polycrystal via a third mechanism: growth front nucleation. This mode of growth manifests when new orientations nucleate on the front of a growing crystal, yielding a densly branched, moprhology, where the classical effects of surface energy anisotropy are disrupted by the nucleation process. We develop a phase field model to describe the phenomenon, and demonstrate that this type of growth can be initiated by either static or dynamic heterogeneities in the solidifying system. We examine several types a growth forms, reexamine some older experiments, and examine prospects for models of this phenomenon in three dimensions.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Thin Films, Coatings and Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Tuesday AM Room: 3020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Stephen J. Pennycook, Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN 37831-6030 USA; Bhushan Sopori, National Renewable Energy Laboratory, Golden, CO 80401 USA

8:30 AM
Control of Bonding and Epitaxy at Metal/Sapphire Interface: Sang Ho Oh[1]; Christina Scheu[1]; Thomas Wagner[1]; *Manfred Rühle*[1]; [1]MPI für Metallforschung, Heisenbergstr. 3, Stuttgart 70569 Germany

The atomic structure and composition of the terminating plane of the sapphire (a-Al2O3) surface is one most crucial parameter control-

ling the bonding mechanism at metal/sapphire interfaces. We report here the experimental methods to control the nature of bonding at copper/sapphire interfaces by utilizing various surface cleaning processes in ultrahigh vacuum (UHV), which can tailor the termination of the sapphire (0001) surface. A clean hydroxylated surface is obtained by room temperature oxygen plasma cleaning, on which Cu atoms adhere principally by polarization and with some characteristics of ionic bonding. The surface can be sufficiently dehydroxylated by using Ar ion sputtering and annealing in UHV, resulting in an Al-rich surface, where Cu grows as a polycrystalline film and CuAl2-type intermetallic Cu-Al bonding forms at the interface. High temperature oxygen plasma treatment can sufficiently oxidize the surface Al ions in a structure similar to •-Al2O3 on which Cu2O-type ionic-covalent Cu-O bonding can be activated to form at an elevated temperature after growth.

9:00 AM Invited
Effects of Grain Size from Millimeters to Nanometers on the Flow Stress of Metals and Compounds: *Hans Conrad*[1]; Kang Jung[1]; [1]NC State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA

Data on the effect of grain size d from millimeters to nanometers on the flow stress of metals and compounds are evaluated. Three grain size regimes are identified: Regime I (d > ~1um), Regime II (d = 10nm - 1um) and Regime III (d < ~10nm). Lattice dislocations are active in Regimes I and II, but absent in Regime III. The mechanisms responsible for plastic deformation in each of the three regimes are discussed. More than one mechanism, or even a different mechanism, may become operative in the transitions between the grain size regimes.

9:30 AM Invited
Influence of Dopants on Embrittlement of Metal Grain Boundaries: *Gerd Duscher*[1]; Matthew F. Chisholm[2]; Wolfgang Windl[3]; [1]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA; [2]Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN USA; [3]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH USA

The combination of atomic-column resolved Z-contrast imaging, spatially resolved electron energy loss spectroscopy and ab initio density functional theory is used to study the origin of embrittlement of metal grain boundaries. The mechanical properties of these grain boundaries are dramatically influenced by dopant segregation. We will discuss three different materials (Cu, Ni3Al, and Al), in which pristine and doped grain boundaries behave differently. Our findings suggest a new concept for the determination of brittle grain boundaries.

10:00 AM Invited
Oxidation Kinetics of Boron Coating on Ferrous Alloys at High Temperature: *Roumiana S. Petrova*[1]; Naruemon Suwattananont[1]; [1]New Jersey Institute of Technology, Dept. of Physics, 161 Warren St., Univ. Hgts., Newark, NJ 07102 USA

The oxidation behavior of boron coating on steel substrate was investigated at elevated temperature. For oxidation process high temperature range 600-900°C was used. The isothermal oxidation behavior was investigated by means of TGA, Optical Microscopy, SEM, and XRD techniques. The oxidation kinetics of boron coating follows about parabolic rate law. At the oxidation temperature 600°C two fluctuations appear at approximately 12h and 18 h. At temperature 700°C only one distinct step occurs. Forming of oxides on the surface of the coated samples tends to prevent the surface from oxidation.

10:30 AM Break

10:45 AM
Photoluminescence from an Er-Doped Ge-Rich SiO2 Sputtered Films: The Influence of Sputter Gas and Annealing: *Chenglin Heng*[1]; Terje G. Finstad[1]; Anette E. Gunnæs[1]; Preben Storås[2]; *Yanjun Li*[3]; [1]University of Oslo, Dept. Physics, PO Box 1048, Blindern, Oslo N-0316 Norway; [2]SINTEF, ICT, PO Box 124, Blindern, Oslo N-0314 Norway; [3]University of Oslo, Ctr. for Matls. Sci. & Nanotech., PO Box 1126, Blindern, Oslo N-0318 Norway

We report on PL from Er-doped Ge-rich SiO2 films deposited on a Si substrate by magnetron sputtering. The sputtering ambience and annealing temperature has been varied. The structural evolution and the distribution of the elements have been studied with high resolution transmission electron microscopy in combination with analysis of emitted X-rays and energy loss spectroscopy. There is very weak or no luminescence for the as-prepared films. Large PL intensity from the film is reached after annealing at 700°C the films sputtered in Ar. These contain Ge-rich amorphous clusters. After annealing to higher temperatures the PL intensity decreases and there are Ge nanocrystals in the film. For films sputtered in an Ar+O2 gas the PL intensity increases with annealing temperature up to 1100°C while no nanocrystals form. These films are all amorphous while some segregation of Ge-rich and Si-rich oxide takes place.

11:15 AM
Effect of Processing on Microstructure and Electrical Properties of Ta Thin Films: *Vikram M. Bhosle*[1]; Ashutosh Tiwari[1]; P. Kumar[2]; R. Wu[2]; [1]North Carolina State University, MSE, 2153, Burlington, Campus Box-7916, Raleigh, NC 27695 USA; [2]H. C. Starck, Inc., 45 Industrial Place, Newton, MA 02161-1951 USA

We have fabricated thin films of tantalum with grain size ranging from nanosize to single crystal and amorphous tantalum (a-Ta) by nonequilibrium pulsed laser deposition techniques, and compared their electrical properties and diffusion characteristics with properties of Beta tantalum (â-Ta) films produced by magnetron sputtering. Single-crystal Ta films are formed by domain matching epitaxy where integral multiples of lattice planes match across the film-substrate interface. Microstructure and atomic structure of these films were studied by X-ray diffraction and high-resolution electron microscopy, while elemental analysis was performed using electron energy loss spectroscopy and X-ray dispersive analysis. The four-point probe resistivity measurements in the temperature range (10-300K) showed room-temperature values to be 15-30µ Ù- cm for á-Ta, 180-200µ Ù-cm for â-Ta and 250-275µ Ù-cm for a-Ta. The temperature coefficient of resistivity (TCR) for á-Ta and â-Ta were found to be positive with characteristic metallic behavior, while TCR for a-Ta was negative, characteristic of high-resistivity disordered metals. We discuss the mechanism of formation of a-Ta and show that it is stable in the temperature range 650-7000C with a superior Cu diffusion barrier characteristics.

11:30 AM
Microstructural Evolution of Nickel Nanoparticle Catalysts Supported on Gadolinium-Doped Ceria During Autothermal Reforming of Iso-Octane: V. Palaniyandi[1]; *Earl T. Ada*[1]; M. Shamsuzzoha[1]; Giovanni Zangari[2]; R. G. Reddy[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904 USA

The microstructure and composition of a nanoparticle Ni catalyst supported on gadolinium-doped ceria (Ce1-xGdxO(4-x)/2) were studied using Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD), and X-ray Photoelectron Spectroscopy (XPS). The support of the fresh catalyst exhibits a homogenous aggregation of crystalline grains, with sizes ranging between 20 and 50 nm. The crystalline structure of the fresh catalyst support is of the CeO2 phase, in which gadolinium atoms exist in a solid solution of CeO2. Nickel in the fresh catalyst is highly dispersed and forms granular crystals 5 - 30 nm in size on the surface of the ceria support. The support of the used catalyst exhibits a bimodal distribution of grains in which smaller grains have similar structure and morphology as those in the fresh catalyst, while the larger sized grains appear dull and exhibit non-faceted crystal morphology resulting either from the sintering of a number of CeO2 grains, or by the occupation of highly defective crystals of Ce2O3 and CeO phases. A thin amorphous layer of carbon also covers most of the larger grains in the used catalyst. Ni particles could not be imaged by TEM in the used catalyst, but Energy Dispersive X-ray Spectroscopy (EDX) detected their presence. XPS analysis of the catalyst samples suggests the participation of lattice O atoms from the ceria support in the catalytic reaction. XPS data also show the presence of carbonate species and a higher hydrocarbon concentration in the used catalyst.

11:45 AM Invited
Nitride-Based Thin Films Processed by Pulsed Laser Deposition: *Haiyan Wang*[1]; Xinghang Zhang[2]; Ashutosh Tiwari[3]; A. Gupta[3]; Jagdish Narayan[3]; [1]Los Alamos National Laboratory, MST-STC, MS K763, Los Alamos, NM 87545 USA; [2]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [3]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA

Nitride-based materials have attracted broad interest due to their high hardness, superior wear resistance, high temperature thermal stability, and exceptional optical and electrical properties. These types of materials have wide applications as super-hard coatings, diffusion barriers in semiconductor industry, and light-emitting-diodes. Various PVD and CVD techniques have been explored to synthesize nitride-based materials. Among them, pulsed laser deposition (PLD) shows exceptional advantage in producing high quality complex composites. Some successful examples on TiN, TaN, and TaN-TiN binary components will be shown. Microstructures of these thin films were studied extensively by X-ray diffraction, transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM). The concept of domain matching epitaxy will be introduced for understanding the epitaxial growth of high lattice mismatch systems. Elec-

trical, mechanical properties and diffusion barrier characteristics of these nitride-based materials were also studied.

12:15 PM
Growth of High Quality Epitaxial ZnO/Pt Bilayer and ZnO-Pt Nanocomposite Structure on Sapphire (0001) for Transparent Conducting Applications: *Amit Chugh*[1]; [1]North Carolina State University, Matls. Sci. & Engrg., 2141 Burlington Labs, CB 7916, Raleigh, NC 27695-7916 USA

We have grown high quality ZnO/Pt bilayer and ZnO-Pt nanocomposite structure on Sapphire substrate (0001). These films were grown heteroepitaxially on sapphire (0001) substrates by Pulsed Laser Deposition (PLD). Epitaxial relationship between sapphire and platinum, platinum and zinc oxide was studied using X-Ray diffraction, high resolution transmission microscopy (HRTEM) and SAED experiments. Experimental results and epitaxial analysis was compared with theoretical simulations. Electrical property measurements were performed using four-point probe method over the temperature range of 15 K - 300 K. Optical transmission and absorption studies were performed using Hitachi spectrophotometer, quite high transmission of visible light was observed. Room temperature photo luminescence studies also indicate high optical quality of these films. These heterostructures demonstrate the feasibility of integrating them with optoelectronic devices.

TUESDAY AM

General Abstract Session: Nanostructured and Lightweight Materials

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday AM Room: 2007
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Farghalli A. Mohamed, University of California, Dept. of Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA

8:30 AM
Grain Growth Behavior in Near Nanostructured Al 5083 Cryomilled Alloy on Isothermal Annealing: *Indranil Roy*[1]; Manish Chauhan[1]; David Edward McDougall[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

Grain growth behavior in the near nanostructured Al 5083 alloy of an average grain size of 300 nm, processed by consolidating nanocrystalline cryomilled powder particulates, was investigated in the temperature range of 0.55 to 0.85 Tm, where Tm is the melting temperature, for different annealing times varies from 1 to 50 hours. Appreciable grain growth was observed at 0.85 Tm, whereas there was nominal grain growth at 0.67 Tm. The value of the time exponent 'n' was deduced from the grain growth equation of the general form D1/n - Do1/n = kt was 0.1 at 0.85Tm and 0.08 at 0.73Tm which decreased with decreasing temperature and approached 0.03 as the temperature was reduced down to 0.55Tm.

8:55 AM
Carbon Nanotube/Light-Metal Composites: *Efraín Carreño-Morelli*[1]; Robert Schaller[2]; [1]University of Applied Sciences of Western Switzerland, Design & Matls. Unit, Rte. du Rawyl 47, Sion CH-1950 Switzerland; [2]Swiss Federal Institute of Technology Lausanne, Inst. of Physics of Complex Matter, Lausanne CH-1015 Switzerland

Novel light metal matrix composites have been processed by powder metallurgy. The feasibility of manufacturing magnesium- and aluminium-based composites reinforced with carbon nanotubes has been assessed. Blends of metal powders and CVD processed multi-wall carbon nanotubes were compacted by uniaxial hot pressing followed by hot isostatic pressing. A uniform dispersion of nanotubes in the metal matrix was obtained. Sintering conditions were found, which allowed to obtain bulk specimens with sound mechanical properties.

9:20 AM
Structure-Property Relationships in Cryomilled Al-Mg Alloys: *David B. Witkin*[1]; Bing Q. Han[2]; Enrique J. Lavernia[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA; [2]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Cryomilling, or ball milling within a cryogenic medium, is a mechanical attrition technique that both refines the microstructure and introduces nanometer-scale dispersoids that stabilize the grain size and strengthen the material. Consolidation and extrusion of cryomilled Al 5083 gives a material whose strength is more than twice that of conventional wrought, strain-hardened Al 5083, with comparable ductility. During the course of processing, the average grain size increases from 25 nm in the as-milled powder, to approximately 100 nm in the primary consolidated form (HIPped) and then to between 200 and 250 nm in the final extrusions. At room temperature, the yield stress, tensile strength, and flow stress of the extrusions are roughly 20 percent higher than that of the HIP material despite the larger grain size. This behavior can be understood in light of a bimodal microstructure and the processing history. The microstructural changes that occur during thermomechanical processing were further investigated by uniaxial compression of the HIP material at elevated temperatures bracketing the extrusion temperatures.

9:45 AM
Numerical Simulations of the ECAE Process with Copper, Second Pass: *Andrey Smolyakov*[1]; Petr Nizovtzev[1]; Vyacheslav Solovjev[1]; Alexander Korshunov[1]; [1]RFNC VNIIEF, Theoretical, Mira, 37, Sarov Russia

ECAP second pass simulation has been performed with isotropic model of material behavior. Satisfactory agreement between numerical and experimental data has been obtained on billet final shape and pressing force during ECAP. This approach enables simulations of subsequent ECAP passes for different routes.

10:10 AM Break

10:30 AM
Study on Method of Increasing Viscosity in Fabricating Aluminium Foam: *H. J. Luo*[1]; G. C. Yao[1]; X. M. Zhang[1]; Y. H. Liu[1]; [1]Northeastern University, Sch. of Matls. & Metall. 110004 China

The method of directly foaming in molten Al to prepare closed-cell aluminum foam is descried in this article. Different kinds of ingredients are put into the molten Al alloy to make its viscosity increased. The TiH2 as foaming agent is also added into these molten Al to fabricate aluminum foam. The effectiveness of the method to increase the viscosity of molten Al on foam structure is researched by macroscopic observation and microstructure analysis. The results are showed as follow. After these ingredients, such as calcium, magnesium, Al2O3 and coal ash, etc, had been added into molten Al, the phase component of Al alloy changed. However the phase component and the mechanism of its increasing viscosity is different slightly. Meanwhile, the obtained Al foam also has great difference in its structure. The foams obtained by calcium and coal ash hold thick cell wall and high intensity, in the other hand the foams obtained by magnesium and Al2O3 hold thin cell wall and low intensity.

10:55 AM
Effect of Cu on the Formation Behavior of Intermetallic Compounds in Al-Mn Wrought Alloy: *Young-Ok Yoon*[1]; Dae-Guen Kim[1]; Young-Jig Kim[1]; Hanshin Choi[2]; Hoon Cho[2]; Hyung-Ho Jo[2]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Chunchun-Dong, Jangan-Gu, Suwon, Gyounggi-Do 440-746 S. Korea; [2]Korea Institute of Industrial Technology, Advd. Matls. Ctr., 994-32, Dongchun-Dong, Yeonsu-Gu, Incheon 406-130 S. Korea

During the extrusion of Al-Mn wrought alloy to manufacture tube and fin for heat exchangers, sticking between alloy billet and die is regarded as the main drawback for workability. In fact, the sticking makes various deteriorations in die cleanness and surface properties of as-extruded tube or fin. It is well known that the sticking mainly depends on the intermetallic phase formation. The main emphasis of this study is to investigate the effect of Cu content on the formation behavior of intermetallic compounds. The quantification and the phase evolution of intermetallic compounds were intensively conducted. With increasing Cu content, the fraction and size of intermetallic compounds were notably increased and the distribution of them was improved. The tensile strength and hardness of them were also increased.

11:20 AM
A Study on the Casting and Properties of Al Foam: *Bo-Young Hur*[1]; Sang-Youl Kim[1]; Kwang-Ho Song[2]; Yong-Su Um[1]; [1]Gyeongsang National University, Jinju 660-701 Korea; [2]Daelim College, Kyungkido Korea

Al alloy foam used in this study was prepared that Al alloy was molten using a high-frequency induction furnace. The molten Al alloy

was put in a pot furnace for a specific period to make it reach the temperature appropriate for foaming. Thickening and foaming agent were added to the molten Al alloy at a specific temperature, which was stirred. An impeller with 3-stage screw welded was used to stir the molten Al alloy. The impeller was rotated clockwise to generate vortex flow toward the center. For uniform distribution of thickening and foaming agent, the impeller was rotated with high-speed about 400-1000rpm. When left in a furnace for a specific period after uniform dispersion of the foaming agent, gas was generated from the foaming agent and the cell size and distribution inside Al alloy foam could be controlled by the viscosity and surface tension of the molten Al alloy. Al alloy foam has good sound and energy absorption properties.

11:45 AM
Plasma Coating and Magnetic Alignment of Carbon Nano Fibers in Polymer Composites: *Donglu Shi*[1]; Peng He[2]; Jie Lian[3]; Xavier Chaud[4]; Eric Beaugnon[4]; Luming Wang[3]; Rodney Ewing[3]; Robert Tournier[4]; [1]University of Cincinnati, Cheml. & Matl. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [2]University of Cincinnati, Mechl. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [3]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; [4]Consortium de Recherches pour l'Emergence de Technologies Avancées et Laboratoire de Cristallographie, Grenoble Cedex 38042 France

An extremely thin layer of polymer film has been coated onto both outer and inner surfaces of the nanotubes. Due to surface modification, the dispersion of nanotubes in the polymer matrix is significantly enhanced. HRTEM images, SIMS results of coated surface films on nanotubes, and mechanical properties of the composites will be presented. For fundamental study and novel engineering applications, carbon nanotubes also need to be aligned along certain specified directions. In this study, we present a novel method by which these nanotubes can be well aligned in a polymer matrix at moderate magnetic field. Both TEM and SEM results show clear evidence of well aligned nanotubes in the polymer composite. The magnetic alignment mechanism is discussed.

General Abstract Session: Advances in Steels

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday AM Room: 2011
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Philip Nash, IIT, Mechl., Matls. & Aeros. Engrg. Dept., Chicago, IL 60616 USA

8:30 AM
The Role of Impurities and Processing in Damascus Steels: *Khalid Mawani*[1]; [1]Ghulam Ishaq Khan Institute, Matls. & Metallurgl. Engrg., Rm. No. 94, Hostel 5, Topi Swabi, NWFP 23460 Pakistan

Sir Walter Scott, in his book "THE TILSIM" writes about the encounter of Sultan Salahuddin Ayubi and Richard in the thirteenth century. Describing the blades of the swords used by the army of Sultan was not so heavy like the swords used by his European opponent. It was dull blue and marked with millions of meaningless lines. Sultan used to astound people by flying the handkerchief and slicing it into two. Most important thing about these swords were that they never became blunt even after the heavy clashes and were sharp enough to cleave a man in half with only one hand. The secret of these swords lies in the Damascus steel. This steel was not produced in Damascus but since the European army first came across this blade in Damascus that is why it is known as Damascus steel. It is believed that the raw materials for these blades were made in India which is known as the "Wootz Steel", where the process of making Wootz continued till the 19th century. Unfortunately, the technique of producing Wootz Damascus steel blades is a lost art. There are two schools of thought on the processing of these blades. One is that the beautiful patterns present in the swords were because the pattern-welded steels were produced by forge welding alternating sheets of high- and low-carbon steels. This composite was then folded and forge-welded together, and the fold/forge cycle was repeated until a large number of layers were obtained while, other is that the pattern on the sword are because of the hypereutectoid carbon level of these steels. In this review paper we will discus the processing method on the basis of facts of history and the knowledge of metallurgy in the ancient times.

9:00 AM
Influence of Co and Ni on Mechanical Properties in Ultrahigh Strength Secondary Hardening Steels: *Ho Seop Sim*[1]; Kon Bae Lee[1]; Heang Ryeal Yang[2]; Hoon Kwon[1]; [1]Kookmin University, Sch. of Advd. Matls. Engrg., 861-1, Jongnung-dong, Songbuk-ku, Seoul 136-702 Korea; [2]Incheon City College, Dept. of Mechl. Engrg., Incheon 402-750 Korea

Alloys were designed by the variation of Co(5-13 wt%) and Ni(8-14 wt%) based on AerMet 100(0.23C-1.2Mo-3.1Cr-13.4Co-11.1Ni). These alloys were severely rolled by 50%/2p at 700 or 850 C in the low austenite region and then direct quenched after reheating in the range 900-1200 C. In the as-quenched condition, tensile strength to hardness ratio was higher as compared to the aged condition. Influences of reheating and rolling temperatures on hardness/tensile strength(UTS) and impact toughness did not show a clear difference in the aged condition since the aging appeared to screen the effects of those prior treatments. In the aged condition, the hardness(Rc)/UTS(MPa) was arranged as follows; 11Ni-13Co(57/2130) > 14Ni-13Co(57/2115) > 8Ni-13Co(56.5/2065) > 11Ni-9Co(56/2025) > 8Ni-9Co(55/1945) > 11Ni-5Co(54/1905). Co did not promote aging despite the increased hardness/UTS whereas Ni promoted aging accompanied by an increase in hardness.

9:30 AM
Microstructure and Elevated Temperature Stability of 9-12% Cr Steels: *Omer N. Dogan*[1]; Jeffrey A. Hawk[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

Medium Cr steels have been used in fossil fired power plants for many years because of their excellent high temperature stability and mechanical properties. As the desire to increase the efficiency of power plants continues, the operating temperature (>650C) continues to go up. Currently available low and medium Cr containing steels will not withstand the new operating temperature and must be reassessed in terms of their solid-solution and precipitation strengthening schemes. Three medium Cr steels were developed to investigate high temperature alloy strengthening strategies: 0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti. The microstructure of the alloy will be described in the as-cast and thermo-mechanically worked states. In addition, the effect on microstructure from long-term high temperature exposure will also be discussed. Finally, the overall stability of these steels will be compared against currently available power plant steels.

10:00 AM
Oxidation Resistance of 9-12% Cr Steels: Effects of Rare Earth Surface Treatments: *Omer N. Dogan*[1]; *Jeffrey A. Hawk*[1]; David E. Alman[1]; Paul D. Jablonski[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

Medium Cr steels have been used in fossil fired power plants for many years because of their excellent high temperature stability and mechanical properties. The environment in a fossil fired power plant is extremely aggressive in terms of corrosion, especially oxidation. This is only accelerated as the operating temperature increases to 650C and beyond. For any new steel to be qualified for power plant use, in addition to adequate strength at the operating temperature, material wastage from all corrosion processes must be kept to a minimum acceptable level. The use of medium Cr steels provides a means to improve overall corrosion resistance. Three medium Cr are under development for use as high temperature power plant steels: 0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti. Oxidation tests were performed on the steels for times greater than 1000 hours in order to determine the oxidation kinetics and extent of material wastage. Also, rare earth oxides were incorporated into the outer surface layers of the steels to see if the oxidation resistance could be improved. These results will be compared to current power plant steels.

10:30 AM Break

10:50 AM
Room and Elevated Temperature Mechanical Behavior of 9-12% Cr Steels: Omer N. Dogan[1]; *Jeffrey A. Hawk*[1]; Karol K. Schrems[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

The mechanical properties of medium Cr steels used in fossil fired power plants are very good because of their excellent high temperature microstructural stability. However, as the desire to increase the operating temperature (>650C) of the plant goes up, the need for steels that maintain their strength at these temperatures also increases.

The mechanical properties of three medium Cr steels (0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti) were investigated through hardness, hot hardness and tensile measurements. The strength of the 9-12%Cr steels at room temperature after long-term isothermal aging (750C; 1000 hours) compares favorably with that of other power plant steels (e.g., P91). In addition, the elevated temperature strength and hot hardness also behave similarly. The mechanical behavior will be discussed in terms of the strength, elongation and tensile fracture characteristics.

11:20 AM
Using Hardness to Model Yield and Tensile Strength: *Jeffrey A. Hawk*[1]; Omer N. Dogan[1]; Karol K. Schrems[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

The current direction in hardness research is towards smaller and smaller loads as nano-scale materials are developed. There remains, however, a need to investigate the mechanical behavior of complex alloys for severe environment service. In many instances this entails casting large ingots and making numerous tensile samples as the bounds of the operating environment are explored. It is possible to gain an understanding of the tensile strength of these alloys using room and elevated temperature hardness in conjunction with selected tensile tests. The approach outlined here has its roots in the work done by Tabor for metals and low alloy and carbon steels. This research seeks to extend the work to elevated temperatures for multi-phase, complex alloys. A review of the approach will be given after which the experimental data will be examined. In particular, the yield stress and tensile strength will be compared to their corresponding hardness based values.

11:50 AM
Bainite Transformation in Low Carbon Steel Simulated by Gleeble: *Smati Chupatanakul*[1]; Philip Nash[1]; Robert Binoniemi[2]; [1]IIT, Thermal Process Tech. Ctr., IL 60616 USA; [2]DANA Corporation

Low carbon 43 series alloy steel is a popular carburizing grade steel for automotive gears. Generation of a high carbon bainitic case microstructure and low carbon martensitic core microstructure may provide significant benefit for gear applications where toughness dictates gear performance. In this paper, Gleeble 3500 is used to simulate the real process. In order to get a homogenous starting microstructure in the samples, each specimen is austenitized at 915°C for 5 minutes. After that the samples are cooled down to an austempering temperature above the Ms temperature and held for a period of time to get a bainitic structure. SEM and dilatometry were used to determine the phase fraction in the steel. These data are put into Avrami's equation to get the time exponent n, about 1.5 for transformation from austenite to bainite.

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Tuesday AM Room: 3008
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chmst. & Matls. Sci. Direct., Livermore, CA 94551 USA; Igor A. Abrikosov, Linköping University, Dept. of Physics & Measurement Tech., Linköping 58183 Sweden

8:30 AM Invited
Pseudo-Gap at the Fermi Level in Al Based Intermetallics: *Esther Belin-Ferre*[1]; [1]CNRS, LCPMR UMR 7614, Université P. & M. Curie, 11 rue Pierre et Marie Curie, Paris 75231 France

Series of alloys are stabilized by a Hume-Rothery mechanism, namely interaction between Bragg planes and electronic waves. This induces a depletion in the electronic densities of states at the Fermi level, the so-called pseudo-gap. Many Al-Cu-based quasicrystals are formed of elements with small differences in atomic radii and electronegativity and their average electron per atom ratio equal same values as in Hume-Rothery phases. Therefore, it was proposed that a Hume-Rothery mechanism should be responsible for their stability. Actually, a pseudo-gap at the Fermi level was predicted from band structure calculations of model phases with local order similar to that of the quasicrystals. It was also observed with different experimental spectroscopic techniques on various quasicrystalline systems. The paper will discuss from the standpoint of experimental spectroscopy data, to which extent the electronic density of states in Al-based quasicrystalline compounds is sensitive to Hume-Rothery stabilization by comparing to Al-Cu and Al-Cu-Fe Hume-Rothery alloys.

9:00 AM Invited
Electronic, Magnetic and Transport Properties of the Pseudogap Fe2VAl System: *Yoichi Nishino*[1]; [1]Nagoya Institute of Technology, Dept. of Matls. Sci. & Engrg., Showa-ku, Nagoya 466-8555 Japan

While the Heusler-type Fe2VAl compound exhibits a semiconductor-like behavior of electrical resistivity, band calculations predict that it is a nonmagnetic semimetal with a sharp pseudogap right at the Fermi level. A substantial mass enhancement deduced from electronic specific-heat measurements suggests that the unusual electron transport is mainly interpreted in terms of the effect of strong spin fluctuations in addition to the possession of a low carrier density. Doping of quaternary elements causes a sharp reduction in the resistivity and a large enhancement in the Seebeck coefficient. Remarkably the Seebeck coefficient plotted against the average electron concentration, instead of the composition of doping elements, is found to fall on a universal curve irrespective of doping elements. Substantial enhancements for the Seebeck coefficient can be explained by using the electronic structure where the Fermi level shifts slightly from the center of the pseudogap.

9:30 AM Invited
Semiconducting Al-Transition-Metal Alloys: *Marian Krajci*[1]; Juergen Hafner[2]; [1]Slovak Academy of Sciences, Inst. of Physics, Dubravska cesta 9, Bratislava SK-84511 Slovak Republic; [2]University of Vienna, Inst. for Matls. Physics, Sensengasse 8/12, Vienna A-1090 Austria

In intermetallic compounds formed by transition-metals (TM) and Al, in some cases a special Al-TM ordering can lead to the formation of a semiconducting band-gap in the electronic spectrum. On the basis of ab-initio electronic structure calculations we studied semiconductivity in some crystalline and quasicrystalline phases. We found that the semiconducting behavior is accompanied with a formation of chemical bonds between aluminum and TM atoms with a high degree of covalency. The bonds form a network with a special Al-TM ordering. Any deviation from this ordering, for instance a existence of substitutional defects, leads to formation of localized states in the gap. We investigated the mechanism of the band-gap formation. The hybridization plays here an important role but for a true band-gap formation the hybridization alone is not sufficient. A breaking of symmetry of the crystal structure via Peierls-like mechanism is also very essential.

10:00 AM Break

10:20 AM Invited
Non-Metallic Properties in Metallic Alloys: An Electron Valence Effect: *Joseph Poon*[1]; [1]University of Virginia, Physics, McCormick Rd., POB 400714, Charlottesville, VA 22904 USA

The electronic properties of quasicrystals and intermetallic compounds will be discussed as examples of Fermi-surface-Jones-Zone interaction effects. When the valence electronic counts are right, it can give rise to a pseudogap or semiconducting gap. Novel properties including electron localization and robust bandgap behaviors unexpected of metallic systems are observed when strong hybridization is coupled with complex crystal structures. Some examples obtained from insulating quasicrystals and semiconducting intermetallic compounds that exhibit promising thermoelectric properties will be described.

10:50 AM Invited
Prediction of Site Preference and Phase Stability of Transition Metal Based Frank-Kasper Phases: *Marcel H.F. Sluiter*[1]; Alain Pasturel[2]; Yoshiyuki Kawazoe[1]; [1]Tohoku University, Inst. of Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan; [2]CNRS, Lab. de Physique et Modélisation des Milieux Condensés, Grenoble France

Site preference of transition metals for crystal sites in Frank-Kasper type tetrahedrally close packed phases has been computed using density functional based electronic structure methods and cluster expansions. Comparisons with results from X-ray and neutron scattering experiments, where available, indicate that theoretical results are accurate. The cluster expansion method is used to analyze whether site preference is driven by onsite effects (such as atomic size) or chemical ordering effects (such as maximization of unlike bonds). Moreover the

role of vibrational degrees of freedom on the sire preference is discussed.

11:20 AM Invited
Phase Stability in Refractory Metal Silicide Phases: Ridwan Sakidja[1]; *John H. Perepezko*[1]; [1]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

Refractory metal (RM) silicide phases are attractive as high temperature structural materials and at low temperature for microelectronic applications. While silicides can develop oxidation resistant SiO2 coatings, multiphase alloy designs are necessary to achieve satisfactory structural performance. The observed alloying trends in ternary and higher order systems highlight the fundamental factors such as geometric rules that govern phase stability. Often, the drive to achieve a high packing density of metal and metalloid constituents yields a strict range of atomic size ratios of metal to metalloid that favors structural stability. By following the guidance offered by the geometric rule it has been possible to design new multiphase microstructures by systematically modifying the phase stability. Usually, silicides are stoichiometric, but there are cases with a homogeneity range where defect structures are important. The basis for the structural stability analysis is derived from the behavior in RM-Si-B and RM-Si-C systems.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Lead-Free Solder Alloy Development

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Tuesday AM Room: 3014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: James Lucas, Michigan State University, Chem. Engrg. & Matl. Sci., E. Lansing, MI 48824-1226 USA; Iver E. Anderson, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

8:30 AM Invited
Development of Low Melting Temperature Lead-Free Solder Pastes for High Temperature Applications: *Randall M. German*[1]; Louis G. Campbell[1]; [1]Pennsylvania State University, Ctr. for Innovative Sintered Products, 147 Research W., Univ. Park, PA 16802 USA

Gas atomization was used to formulate rapidly solidified tin-based alloy powders for use in a high temperature application. The powders were mixed with reactive alloying additions and formed into pastes useful in automated assembly. For target applications such as outdoor lighting, the creep properties of lead-free tin-based solders is too low. Yet rapid flow during assembly requires a low melting temperature solder. The normally low melting temperature solders required formation of an reactive alloy containing titanium to induce transient liquid phases and precipitation reactions for strengthening. Elevated temperature testing showed that only reactive powders could pass the standard tests. Phase relation rules were formulated with respect to the reaction events needed to form the desired transient phases; the rules considered both solder flow and subsequent reaction with the substrate to form an elevated temperature alloy solder.

9:00 AM
Microstructure and Long Term Reliability of Lead-Free Solder Joints Using Low Temperature Solder Paste: *Wang Ju Lee*[1]; Un-Byoung Kang[1]; Hee Kook Choi[1]; Young-Ho Kim[2]; Se Yong Oh[1]; [1]Samsung Electronics, Memory Div., San #74 Buksu-Ri, Baebang-Myeon, Asan, Chungcheongnam-Do 336-711 Korea; [2]Hanyang University, Div. of Matls. Sci. & Engrg., 17 Haengdang-dong, Seongdong-Ku, Seoul 133-791 Korea

A low temperature packaging is required for some electronic devices containing heat-sensitive components or for low cost Pb-free solution in the near future. In this paper, the ball grid array (BGA) technique using high temperature Pb-free solder ball and eutectic Sn-Bi solder paste is introduced. Sn-3.0Ag-0.5Cu solder balls with a diameter of 450 §- were attached on a Fine-pitch Ball Grid Array(FBGA) package, and eutectic Bi-Sn solder paste was printed on OSP-Cu and Au/Ni pads of PCB substrate. The peak temperature during reflow was fixed at 175¡É which is much lower than that of eutectic Sn-Pb solder, and the reflow time over 139¡É, the Bi-Sn eutectic temperature, was 110 sec. During soldering at 175¡É, the lead-free ball of the package does not melt but the eutectic Sn-58Bi paste does and wets to the lead-free ball. The long term reliability of Pb-free solder joint using Sn-58Bi paste was evaluated, and its microstructure change and intermetallic compound formation was investigated before and after reliability test. After soldering, the Bi content in the bottom of solder joint was confirmed to be decreased down to 12 wt%-20 wt% by energy dispersive spectroscopy (EDS). The melting temperature of solder joint after soldering increased up to 190¡É-200¡É, which was attributed to the interdiffusion of Bi and Sn during soldering process. The intermetallic compound thickness formed between Sn-Bi solder paste and PCB pad was much smaller than that of Sn-3.0Ag-0.5Cu paste in both OSP-Cu and Au/Ni PCB pads. The reliability of Pb-free solder joint using Sn-58Bi paste was as high as that of using Sn-3.0Ag-0.5Cu paste at soldering temperature over 250¡É in thermal cycling test. After thermal 2000 cycles, the intermetallic compound thickness formed between Sn-Bi solder paste and PCB pad was same as that of Sn-3.0Ag-0.5Cu paste in both OSP-Cu and Au/Ni PCB pads. The Cu6Sn5/Cu3Sn double intermetallic layers were formed at the interface between Sn-Bi solder paste and OSP-Cu pad after thermal cycles while the Ni3Sn4 layer was only found at the interface of Au/Ni pad. It is expected that the bonding technique using Sn-Ag-Cu solder ball and eutectic Bi-Sn solder paste is a promising method for the board level packaging. Especially low temperature soldering using Sn-Bi paste is expected to be used widely in application area which has low temperature user environment such as consumer, office, etc. for low cost lead-free solution in the near future.

9:20 AM
Role of Shape-Memory Alloy Reinforcements on the Evolution of Strain Localization in Lead-Free Solder Joints: Deng Pan[1]; Chanman Park[1]; Shuwei Ma[1]; *I. Dutta*[1]; B. S. Majumdar[2]; [1]Naval Postgraduate School, Dept. Mech. Astro. Engr., 700 Dyer Rd., Monterey, CA 93943 USA; [2]New Mexico Tech, Dept. of Matls. Sci. & Engrg., Socorro, NM 87801 USA

Microelectronic solder joints are exposed to aggressive thermo-mechanical cycling (TMC) conditions during service. During TMC, severe inelastic strain localization can occur within joints, eventually causing low-cycle fatigue failure. In order to mitigate the effects of strain localization, a composite solder reinforced with NiTi based shape memory alloy (SMA) particles are being developed. In this scheme, the initially martensitic SMA reinforcement is heavily deformed along with the solder in the early stages of TMC. Upon transforming into austenite at the As temperature, the reinforcement undergoes shape recovery, thereby placing the adjoining solder matrix in reverse shear, and thus reducing strain localization within the solder joint. In this paper, we present the results of experimental work on evaluating the impact of the martensite-to-austenite (M?¨A) transformation on matrix strain redistribution in the immediate vicinity of a reinforcement, using in situ thermo-mechanical loading experiments on a cross-sectioned sample inside an SEM. A comparison of in situ strain localization within monolithic Sn-4.7%Ag-1%Cu joints, joints reinforced with 'passive' Cu reinforcements, and joints reinforced with 'active' NiTi reinforcements will be presented. The results of concurrent finite element modeling (FEM) to elucidate the role of phase transformations within NiTi on the evolution of strain distribution within the joint will also be presented.

9:40 AM
Effect of Zn Content on the Vibration Fracture Behavior of Sn-Zn and Sn-Zn-Bi Solders: *Jenn-Ming Song*[1]; Yea-Luen Chang[2]; Truan-Sheng Lui[2]; Li-Hui Chen[2]; [1]National Dong Hwa University, Dept. of Matls. Sci. & Engrg., Hualien 974 Taiwan; [2]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., Tainan 701 Taiwan

Given that plastic deformation, even failure, may occur due to vibration, the vibration fracture resistance of the solder should be taken into consideration during alloy design. To realize the effect of Zn content on the vibration properties of the Sn-Zn-Bi system, this study aimed to explore the vibration fracture behaviors of binary Sn-

xZn (x=7, 9, 11, 13wt%) solder alloys, as well as ternary Sn-xZn-3Bi(x=5, 8, 11wt%). Sn-9Zn has the poorest damping capacity and the lowest critical cycles to failure among the Sn-xZn alloys. On the other hand, the Sn-13Zn samples with massive primary Zn needles possess superior damping capacity and vibration life. As to Sn-xZn-3Bi alloys, both the damping capacity and vibration fracture resistance decrease in turn from Sn-11Zn-3Bi, Sn-8Zn-3Bi to Sn-5Zn-3Bi. The main crack of the Sn-5Zn-3Bi tends to propagate along the phase boundaries between proeutectic Sn and Sn-Zn eutectics.

10:00 AM
A Study on Oxidation of Sn and its Alloys by Electrochemical Reduction Analysis: *Sungil Cho*[1]; Jin Yu[1]; Sung K. Kang[2]; Da-Yuan Shih[2]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. & Engrg., 373-1 Gusung-dong, Yusung-gu, Daejon 305-701 S. Korea; [2]IBM, T. J. Watson Rsch. Ctr., 1101 Kitchawan Rd., Rte. 134, Yorktown Heights, NY 10598 USA

In a microelectronic system, solder joints are constantly exposed to temperature, humidity, stress and other reactive environments. Oxidation (and/or corrosion) of solder joints could be one of the major yield and reliability risk factors. Hence, an in-depth understanding on oxidation behaviors of solder interconnections becomes a critical issue in making solder interconnection technologies successful. However, the oxidation behaviors of Sn and Sn alloys have not been investigated in detail. Tin is a base material of Sn-Pb and Pb-free solders and little is known about its oxidation behavior at various conditions of processing or applications. In this study the solid-state oxidation of pure Sn, Sn-Pb alloys and Sn-base Pb-free solders was investigated by electrochemical reduction analysis method, which can provide the information on the chemistry and the amount of oxides. Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) were also employed to confirm the results obtained from the electrochemical analysis.

10:20 AM Break

10:30 AM
Unusual Spalling of Cu_6Sn_5 Induced by Au/Ni Surface Finish in Sn3.0Ag0.5Cu Solder Joints: *Cheng-En Ho*[1]; Wei-Chen Luo[1]; Eu-Wei Lin[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Chungli City 320 Taiwan

Gold/nickel bi-layer is one of the most common and important surface finishes for treating Cu pads in advanced packages. In this talk, a very interesting phenomenon induced by the Au layer of the Au/Ni surface finish in contact with the Sn3.0Ag0.5Cu solder will be presented. Experimentally, Sn3.0Ag0.5Cu spheres were reflowed on Au/Ni/Cu pads, and chemical interactions between the solder and the pads were investigated. Two kinds of intermetallics, $(Cu,Au,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, coexisted at the solder/Ni interface after reflow. It was found that with increasing reflow time $(Cu,Au,Ni)_6Sn_5$ exhibited a very different morphology, from chunk-like to a continuous layer. Thick $(Cu,Au,Ni)_6Sn_5$ layer eventually spalled from the $(Ni,Cu)_3Sn_4$ into the molten solder as the reflow time reached 60 sec. In contrast, when Sn3.0Ag0.5Cu was reflowed on Ni/Cu, only two dense layers, $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, formed at the interface. The result indicated that the Au layer played an important role in this spalling phenomenon.

10:50 AM
Synthesis of Ni_3Sn_4 and Cu_6Sn_5 Nanoparticles in Deriving Lead-Free Composite Solders: *Li-Yin Hsiao*[1]; Szu-Tsung Kao[1]; Hsiang-Yi Lee[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Intermetallic compounds (IMC) of Ni_3Sn_4 and Cu_6Sn_5 play important roles in the interfacial reaction between the lead-free solder and the Ni/Cu under-bump metallization during reflowing in electronics packaging. In this study, SnAgCu and SnAgNi composite solders were produced by mechanical alloying (MA) process with doping Cu_6Sn_5 and Ni_3Sn_4 nanoparticles, respectively. The nanoparticles of the Cu_6Sn_5 and Ni_3Sn_4 intermetallic compounds were synthesized by chemical precipitation with $NaBH_4$ in aqueous solutions. The nanoparticles were prepared by the precursor reacting with $NaBH_4$. A solution of appropriate metal-precursors was rapidly added to a $NaBH_4$/NaOH solution under strong stirring. After mixing these two solutions, black precipitates were immediately observed, which were washed with distilled water and then dried at room temperature. The structures of particles were characterized by X-ray diffraction (XRD), and field-emission scanning electron microscopy (FE-SEM) analysis was employed to analyze the morphology of the particles. The SnAgCu composite solder joint doped with the nanoparticles of Cu_6Sn_5 intermetallic compound formed thinner $(Cu, Ni)_6Sn_5$ layers at the solder/electroless Ni-P interface than that formed in the commercial solder joint and MA solder joint. The nanoparticles of Ni_3Sn_4 intermetallic compound doped into SnAgNi composite solder joint exhibited the similar effect.

11:10 AM
Effects of Volumetric Contraction of Electronic Solders: *Girish S. Wable*[1]; Srinivas Chada[2]; Bryan Neal[2]; Raymond A. Fournelle[3]; [1]SUNY, Dept. of Sys. Sci. & Industl. Engrg., PO Box 6000, Binghamton, NY 13902 USA; [2]Jabil Circuit, Inc., AMT/FAR Lab, 10800 Roosevelt Blvd., St. Petersburg, FL 33716 USA; [2]Jabil Circuit, Inc., AMT/FAR Lab, 10800 Roosevelt Blvd., St. Petersburg, FL 33716 USA; [3]Marquette University, Dept. of Mechl. & Industl. Engrg., 15151 W. Wisconsin Ave., Milwaukee, WI 53233 USA

Volumetric contraction during solidification is a characteristic that is exhibited by all metals, with the exception of gallium. Alloys that undergo solidification over a broad range of temperature generally exhibit a difference in the contraction behavior of their ensuing phases. Furthermore, dissolution of substrate metals during process reflow leads to meta-stable phases and volumetric contraction artifacts. The extent and frequency of surface roughness, shrinkage voids, fillet lifting and hot tearing seen in "lead-free" solders are significantly different than for "eutectic tin lead" solder. Shrinkage effects have been reported in Sn/Pb, Sn/Pb/Ag, Sn/Ag/Cu, Sn/Cu/Ni solders for various components, but few presented their impact on solder joint reliability. Nevertheless, they warrant a close observation and proper identification due to shift towards lead-free solders. This paper is a review of various defects resulting from shrinkage as well as the factors that contribute to their formation and a methodology to identify these defects effectively.

11:30 AM
Electrical and Mechanical Studies of the (Sn-Ag)eut+Cu+Sb+Bi Soldering Materials: *R. Kisiel*[1]; Gasior Wladyslaw[2]; Moser Zbigniew[2]; J. Pstrus[2]; K. Bukat[3]; J. Sitek[3]; [1]Warsaw University of Technology, Inst. of Microelect. & Optoelect., 00-662 Warszawa, Koszykowa Str. 75 Poland; [2]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., 30-059 Kraków, Reymonta Str. 25 Poland; [3]Tele and Radio Research Institute, 03-450 Warszawa, Ratuszowa Str. 11 Poland

Electrical (solder resistivity and solder joint resistance) and mechanical (tensile strength and shear strength of solder joints) parameters of quinary $(Sn\text{-}Ag)_{eut}$+Cu+Bi+Sb were investigated starting from binary eutectic Sn-Ag and close to ternary eutectic Sn-Ag-Cu alloys. The four-probe technique was used for electrical parameters measurements. Special equipment was constructed for the tensile strength measurement and also for determination of the shear strength calculation of solder joints between a typical circuit component and a Cu contact on a printed circuit board (PCB). It was found that electrical properties of the investigated alloys are comparable to the data from literature for eutectic Sn-Ag and the traditional tin-lead solders. The strong influence of Bi and Sb at the mechanical properties is observed.

Magnesium Technology 2005: Thermodynamics [Magnesium Alloys]

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday AM Room: 2004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Bob R. Powell, General Motors Corp, R&D Ctr., Warren, MI 48090-9055 USA; Rainer Schmid-Fetzer, TU Clausthal, Inst. fuer Metall., Clausthal-Zellerfeld D-38678 Germany

8:30 AM
Experimental Investigation of the Equilibria in Mg-Al-(Ca, Sr) Systems: M. A. Parvez[2]; X. Wang[2]; *Elhachmi Essadiqi*[1]; Mamoun Medraj[2]; [1]CANMET- MTL, CANMET - Matls. Tech. Lab., 568 Booth St., Ottawa, ON K1A 0G1 Canada; [2]Concordia University, Montreal, Quebec Canada

The phase diagrams of Mg-Al-Sr and Mg-Al-Ca systems were investigated experimentally by differential scanning calorimeter (DSC) and X-ray Diffraction (XRD) techniques. The experimental work focused on the critical regions after reviewing the phase diagrams developed by thermodynamic modeling. Differential scanning calorimetry has permitted real time measurement of the phase change involved in these systems. The temperature ranges for the phase change peaks

have been determined. Enthalpy of melting and enthalpy of formation of the compounds are also reported. Comparison between these results and thermodynamic findings will be discussed. These results along with the XRD analysis are used to establish the equilibria in Mg-Al-Sr and Mg-Al-Ca systems. XRD was used to identify the phases in the studied samples. Al4Sr and Al2Ca were found to be the dominating phases in Mg-Al-Sr and Mg-Al-Ca systems, respectively.

8:50 AM
Laves Phase Stability in the Mg-Al-Ca System: *Zi-Kui Liu*[1]; Yu Zhong[1]; Alan Luo[2]; J. F. Nie[3]; Jorge O. Sofo[4]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16803 USA; [2]General Motors Research and Development Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA; [3]Monash University, Sch. of Physics & Matls. Engrg., Victoria 3800 Australia; [4]Pennsylvania State University, Dept. of Physics, Univ. Park, PA 16802 USA

Mg-Al-Ca alloys exhibit appealing creep strength at temperatures over 100C. It is believed that it is due to the substitution of the -Al12Mg17 phase by laves phases of higher thermal stability. In the literature, the C15-Al2Ca phase in the Al-Ca binary system and the C14-Mg2Ca phase in the Ca-Mg binary system have been reported. Their relative stability in the ternary system may play an important role in the development of the Mg-Al-Ca alloys. Furthermore, recent experimental studies and first-principles calculations in the literature have revealed the existence of a C36 laves phase in the Mg2Ca-Al2Ca pseudo-binary. In the present work, special quasirandom structures (SQS's) for the three laves phases, Al2Ca, Mg2Ca and (Mg,Al)2Ca, were developed to mimic their most relevant pair and multisite correlation functions in random solutions. First-principles calculations on the present SQS's were performed for the three laves phases to predict their stability over the whole composition range. Experimental investigations using individual alloys and diffusion couples were also carried out to examine the relationships and stability of the three laves phases obtained theoretically.

9:10 AM
Stress-Strain Response in Skin and Core Regions of Die Cast Magnesium Alloy AM60B Determined from Spherical Microindentation: *J. P. Weiler*[1]; J. T. Wood[1]; R. J. Klassen[1]; R. Berkmortel[2]; G. Wang[2]; [1]University of Western Ontario, Dept. of Mechl. & Matls. Engrg., London, Ontario N6A 5B9 Canada; [2]Meridian Technologies Inc., 25 MacNab Ave., Strathroy, Ontario N7G 4H6 Canada

Spherical indentation testing is used to analyze the dependence of the mechanical properties upon local microstructure of the high-pressure die cast magnesium alloy AM60B. Indentation testing is performed on two samples from the skin and the core regions of the die-casting. The difference in the indentation stress-strain flow curve between the skin and the core, and comparisons with results from uniaxial tensile testing are analyzed. It was found that results from the skin region compare well with the tensile stress-strain flow curve, while results from the core region produce a different strain hardening coefficient and a lower stress at each loading level. This study indicates that the skin region controls the flow properties of tensile samples cut from AM60B die-castings.

9:30 AM
Characterization of Local Deformation of Magnesium Alloys Using Micro-Indentation Techniques: *Lihong Han*[1]; Henry Hu[1]; Derek Northwood[1]; [1]University of Windsor, Dept. of Mechl., Auto. & Matl. Engrg., Windsor, Ontario N9B 3P4 Canada

The microstructure of die cast magnesium alloys varies significantly with the depth from casting surface. To characterize the behavior of local deformation within the vicinity adjacent to the casting surface, a micro-indentation technique has been employed in this study. The paper discusses the time-dependent mechanical response of Mg alloys subjected to a shallow indentation made by a 20μm radius spherical indenter and a three-sided pyramidal (Berkovich) diamond indenter at room temperature. Hardness, composite modulus and local stress-strain curve were obtained by measuring the penetration depth changing with time during testing. The creep characteristics under different constant load were also analyzed using this technique, and the possible mechanisms contributing to indentation creep have been identified.

9:50 AM
Thermodynamics of Mg-Zn-Zr: Implication on the Effect of Zr on Grain Refining of Mg-Zn Alloys: R. Arroyave[1]; *Z. K. Liu*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16803 USA

Due to their high specific strength, magnesium alloys have become increasingly attractive for automotive structural applications. In recent years it has been shown that Zr is a very effective grain refiner of Mg-Zn alloys. The grain refining properties of Zr are due to the presence of both undissolved Zr particles as well as Zr-containing intermetallics. Therefore, an accurate knowledge of the phase state of the system at any composition and temperature is required. In this work, we present a thermodynamic model of the Mg-Zn-Zr system by combining ab initio calculations and the CALPHAD method. Using this model, calculations on both stable and metastable isothermal sections as well as liquidus surfaces and equilibrium solidification are performed and related to experimental observations. The possible formation of metastable intermetallic phases during solidification is also discussed. Solubility of Zr in Mg-Zn-Zr alloys as well as phase fractions of intermetallic compounds are systematically studied as these parameters greatly affect grain refining.

10:10 AM Break

10:25 AM
Influence of Strong Static Magnetic Field on the Solution and Aging Behavior in AZ91 Magnesium Alloys: *Zhifeng Li*[1]; J. D.[1]; X. Q. Zeng[1]; Y. X. Wang[1]; C. Lu[1]; [1]Shanghai Jiao Tong University, Natl. Engrg. Rsch. Ctr. of Light Metal Forming, Shanghai 200030 China

A strong static magnetic field (SSMF) of about 10 T was applied when AZ91 magnesium alloy was undertaken solution and aging heat treatment for different time. Microstructures of conventional solutioned specimens and solutioned specimens under SSMF were investigated by Optical and SEM micrographs which indicated that the solution process of $Mg_{17}Al_{12}$ in the alloy was retarded under the SSMF condition. The microstructures of both conventionally and SSMF aged specimens at 453K were observed by OM and TEM. The results showed that $Mg_{17}Al_{12}$ discontinuous precipitates at grain boundary were promoted in the SSMF condition which resulted in a quick age hardening response in the first stage of aging. The acceleration of discontinuous precipitation may be attributed to the relatively lower body diffusion rate in the matrix under the SSMF condition which resulted in a higher chemical free energy driving force for the nucleation and growth of the discontinuous precipitate.

10:45 AM
Thermodynamics and Constitution of Ca-Zr and Mg-Al-Ca-Zr Alloys: Andreas Janz[1]; Djordje Mirkovic[1]; Joachim Gröbner[1]; *Rainer Schmid-Fetzer*[1]; [1]University of Clausthal, Inst. of Metall., Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

This is a first report in our series of ongoing studies of quaternary Mg-Al-Ca-X systems. The addition of X = Zr is of interest in order to understand the limitations imposed by the alloy phase equilibria on the use of Zr for example as grain refiner. The practical problems in using Zr together with Al are ascribed to the formation of stable Al-Zr compounds. Our thermodynamic calculations reveal that at, say, 800°C in Mg-Al-Zr alloys up to 0.1 wt.% Al and 0.1 wt.% Zr the compounds AlZr, Al_3Zr_4, and Al_2Zr_3 may be formed. We studied the impact of Ca on these equilibria and also the impact of Zr on a wider range of Mg-Al-Ca alloys. One obstacle is that the Ca-Zr binary system is not known. Experimental work and thermodynamic modeling of the phase equilibria in this binary and the higher order alloys are performed. Selected phase diagram sections of the quaternary Mg-Al-Ca-Zr system are shown.

11:05 AM
Precipitation Hardening in Mg-Zn-Sn Alloys: *S. Cohen*[1]; G. R. Goren-Muginstein[1]; S. Avraham[1]; M. Bamberger[1]; R. Rashkova[2]; G. Dehm[2]; [1]Technion-Israel Institute of Technology, Technion City, 32000 Haifa Israel ; [2]Max-Planck-Institut für Metallforschung, Heisenbergstr. 3, 70569 Stuttgart Germany

New Al-free Mg-Zn alloys with alternative alloying elements are currently developed in order to overcome the poor creep behaviour of commercially available Mg-Alloys and to improve their corrosion resistance and castability at elevated temperatures. Based on thermodynamic calculations Zn and Sn additives are expected to form stable intermetallic phases with Mg and cause precipitation hardening. In the present work we studied a Mg alloy containing 4.46 wt.% Zn and 3.75 wt.% Sn. Precipitation hardening mechanisms were investigated in the temperature range of 175-200°C by Vickers hardness measurements and analysis of the microstructured evolution using XRD, SEM, and TEM. During aging at 175°C for 1 to 96 hours, two hardness maxima occurred: the first after 2 hours, where the hardness increased from 55 to 88 HV, followed by a rapid decrease to 73 HV and then the second peak at 87 HV after 16 hours. The hardness decreased after 48 hours to 70 HV and remained constant up to 96 hours. Corresponding XRD spectra and TEM analyse of the sample indicate that the occurrence of two hardness peaks can be related to a precipitation sequences. Firstly, MgZn2 precipitations form, and secondly, Mg2Sn particles precipi-

tate. The precipitates are uniformly distributed in the Mg-matrix with two morphologies: needle- and plate–like shapes. The study was partially supported by the German Israeli foundation for scientific research and development (GIF) under contract number I-704-43.10/2001. The foundation is acknowledged for its support.

Magnesium Technology 2005: Wrought Magnesium Alloys III

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday AM Room: 2006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Robert E. Brown, Magnesium Monthly Review, Prattville, AL 36067-3806 USA; Karl Ulrich Kainer, GKSS Research Center, Ctr. for Mg Tech., Geesthacht D-21502 Germany

8:30 AM
Strain Path and Temperature Effects on Texture and Microstructure Evolution of AZ31: *Chris Huw John Davies*[1]; Fei Xiong[1]; [1]CRC for Cast Metals Manufacturing, Sch. of Physics & Matls. Engrg., Bldg. 26, Monash Univ., VIC 3800 Australia

The effect of strain path and temperature on texture and microstructure evolution in AZ31 was investigated by plane strain (channel die) compression, uniaxial tension, and uniaxial compression. Both as-cast and extruded structures were used as starting textures, and different strain paths were imposed upon the samples, including complete reversal, and 90° rotation in plane strain compression. After each step in the strain path, texture and microstructure were examined. The effect of thermal path has been examined in a similar manner. In this way, a picture of the evolution of texture and microstructure was compiled. Results will be presented showing the effect of strain path on flow stress, and of thermal path on microstructure, and these are interpreted in terms of texture evolution and the propensity for twinning. Simple models are used to show the types of twinning active during deformation.

8:50 AM
Prediction and Measurement of Residual Strains in a DC Cast AZ31 Magnesium Billet: Hai Hao[1]; *Daan M. Maijer*[1]; Mary A. Wells[1]; Steve L. Cockcroft[1]; Ron Rogge[2]; Steve G. Hibbins[3]; [1]University of British Columbia, Matls. Engrg., 309 - 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]National Research Council of Canada, Steacie Inst. for Molecular Scis., Chalk River, ON K0J 1J0 Canada; [3]Timminco Metals Ltd., Haley, ON K0J 1Y0 Canada

As part of a research programme to improve casting efficiency, a thermo-mechanical finite element (FE) model of DC cast AZ31 magnesium billets has been developed to predict defect formation such as hot tearing and cold cracking. The model simulates the evolution of temperatures, stresses and strains inside the billet. Thermal boundary conditions have been selected based on knowledge of the physical process and through comparison with measured temperature data. The temperature predictions show good agreement with the measurements for both transient and steady state conditions. The constitutive behaviour of AZ31, a critical input to the thermal-stress model, was measured using a Gleeble 3500 thermo-mechanical simulator over a wide range of temperatures. In order to validate the predicted strain, residual strain measurements were carried out using neutron diffraction through the National Research Council of Canada's Neutron Program for Materials Research. Strain measurements in the radial, axial and hoop orientations were made at a variety of axial and radial locations in a billet. The measured strains were compared with the predicted results and used to validate the thermo-mechanical model.

9:10 AM
Evaluation of the Surface Heat Flux in the Secondary Cooling Zone During the Direct-Chill Casting: *Etienne J.F. R. Caron*[1]; Mary A. Wells[1]; Dimitry Sediako[2]; Steve G. Hibbins[2]; [1]University of British Columbia, Dept. of Matls. Engrg., 309 - 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Timminco Metals, Tech. Dvlp. & Metall., Haley, ON K0J 1Y0 Canada

Accurate knowledge of the boundary conditions is essential when modelling the Direct-Chill (DC) casting process. Determining the surface heat flux in the secondary cooling zone, where the greater part of the heat removal takes place, is therefore of critical importance, particularly for the process start-up phase. Industrially DC cast AZ31 magnesium alloy samples were instrumented with thermocouples, preheated in an electrical furnace and sprayed with water jets to simulate the secondary cooling zone of the DC casting process. The surface heat flux in both the water jet impingement zone and the water film free-falling zone was then evaluated using the measured thermal history data in conjunction with a two-dimensional inverse heat conduction (IHC) model developed at the University of British Columbia. The effects of various parameters (water flow rate, impingement angle, surface morphology) on the rate of heat removal by the water jets was investigated.

9:30 AM
A Mathematical Model of Heat Transfer and Fluid Flow in the Direct Chill Casting of AZ31 Magnesium Billets: *E. H. Lu*[1]; D. M. Maijer[1]; D. Sediako[2]; [1]University of British Columbia, Dept. of Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Timminco Metals, R&D, Haley, Ontario K0J 1Y0 Canada

A 2-D axisymmetric mathematical model of heat transfer and fluid flow during direct chill (DC) casting of AZ31 magnesium billets was developed using the finite volume modeling package FLUENT. Model boundary conditions were based on those used in a 2-D axisymmetric finite element model previously developed in ABAQUS, which approximates the effects of fluid flow by manipulating material properties. Materials properties were based on literature and knowledge of the physical phenomena occurring during the DC casting process. Temperature predictions from both models are compared with plant trial temperature data acquired from billets produced with different casting conditions at Timminco Metals in Haley, Ontario. Following this initial comparison, the high temperature material properties and boundary conditions of the FLUENT model were altered to fit the measured temperatures. The significance of fluid flow on the apparent material properties of AZ31 and the thermal boundary conditions is presented.

9:50 AM
Process and Alloy Development for Hydrostatic Extrusion of Magnesium: The European Community Research Project MAGNEXTRUSCO: *Jan Bohlen*[1]; Wim H. Sillekens[2]; Piet-Jan Vet[3]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre, Ctr. for Mg Tech., Max-Planck Str. 1, Geesthacht 21502 Germany; [2]TNO Industrial Technology, De Rondom 1, Postbus 6235, Eindhoven 5600 HE The Netherlands; [3]Outokumpu Copper HME b.v., Veerweg 14, Waalwijk 5145 NS The Netherlands

While magnesium cast products have been established in industrial lightweight applications, the development is now focussing on semi-finished wrought products. This leads to a broader variety of shapes as well as to improved material properties of magnesium alloys. In particular, extrusion as a shaping technology offers the possibility to produce such products. While the technology for processing magnesium alloys is available today, the industrial use is not well established because of technical and economical limitations of the direct and indirect extrusion process. The European Commission research project MAGNEXTRUSCO has been conducted as a GROWTH project within the 5th Framework programme in order to overcome these limitations in the production of magnesium wrought components. A consortium of 9 European partners from industry and research institutions focused on the use of the hydrostatic extrusion process for the production of magnesium structural components. The advantage of using this process is to increase the extrusion speed and to lower the extrusion temperature. Alloy development, process research, post-processing techniques and the development of magnesium demonstrators by end users are the main topics of the work programme along the complete process chain. This paper gives an overview on the project's outline regarding such issues as motivation, objectives and partnership. Results with respect to micro-structural evolution and mechanical properties due to the settings of the extrusion parameters will be presented and discussed.

10:10 AM Break

10:25 AM
An Efficient Route to Magnesium Alloy Sheet: Twin Roll Casting and Hot Rolling: *Lothar Loechte*[1]; Hakon Westengen[2]; John Rodseth[3]; [1]Hydro Aluminium, R&D Ctr., Georg-von-Boeselagerstr. 21, Bonn 53117 Germany; [2]Hydro Magnesium Competence Center, R&D Ctr., Porsgrunn 3908 Norway; [3]Hydro Aluminium, R&D Karmoy, Havik 4265 Norway

In the last decade a significant market pull, especially from the automotive industry, has been observed for magnesium alloy sheet. Two main production routes for this material are currently under world-

wide discussion; specifically, the classical route via DC casting plus hot rolling is competing with continuous casting techniques. A particularly flexible route for producing magnesium alloy sheet is provided by twin roll casting, followed by a hot rolling plus annealing sequence to the final gauge and temper. Early results and evaluations from trials with semi-scale (up to 700mm width) twin roll cast and hot rolled AZ31B are presented. Microstructures of as cast strip, as well as intermediate gauges and final thickness sheet, are discussed and related to production parameters such as casting speed and hot rolling process schemes. The promising mechanical properties which have been achieved are compared to those generated by a more conventional route and are discussed in terms of microstructural features, e.g., grain size and texture.

10:45 AM
Control of Wall Thickness Distribution in Magnesium Tube Gas Forming With Large Diameter Expansion: *Ramnath Krishnamurthy*[1]; Wuhua Yang[2]; Xin Wu[1]; Micheal L. Wenner[2]; [1]Wayne State University, Mechl. Engrg., 5050 Anthony Wayne Dr., Detroit, MI 48202 USA; [2]General Motor Corporation, Mfg. Sys. Rsch. Labs., Warren, MI 48090 USA

For developing lightweight structures for automotive applications, the feasibility of forming magnesium AZ31B tubes using Hot Metal Gas Forming process was conducted previously, and the results indicate that tube expansion with very large strain (above 100%) can be achieved. To take full advantages of the material formability enhancement and the new forming technique, the thinning of the tube wall thickness must be compensated, which is very challenging with very large deformation and with sensitive temperature dependence of the material. In this study, the understanding of metal flow and the strategy for thickness compensation are focused. The thickness control is achieved through combined control of temperature distribution, gas pressure and tube end-feeding velocity. Both experimental and analytical results are reported, and a comparison between them is provided. A concept of regional heating/forming and sequential multi-stage operation is proposed that allows local deformation control.

11:05 AM
An EBSD Study on Microstructural Evolution During Superplastic Deformation of a Fine-Grained AZ31 Magnesium Alloy: Yi Liu[1]; *Xin Wu*[1]; [1]Wayne State University, Mechl. Engrg., Detroit, MI 48202 USA

Superplastic deformation has been conducted on AZ31 magnesium alloy with initial fine grains (<10 micron) at 623-773K with the strain rate of 10^-3 to 1s^-1. A maximum elongation of over 500% was obtained at 773K with 10^-3 s^-1. Dynamic grain growth was found, that was temperature dependent. EBSD analysis indicated that, during deformation, the pre-existing strong (0001) fiber texture was not destroyed during deformation, although grain boundary sliding was observed. Together with optical microscope and SEM observations, the deformation mechanisms are discussed.

11:25 AM
Effects on Microstructures and Fracture Morphology of SiCp/AZ91D Composites by Equal-Channel Angular Pressing: *Yan Yin Biao*[1]; [1]Nanjing University of Science & Technology, Matls. Sci. & Engrg., 200 Xiaolingwei St., Nanjing, Jiangsu 210094 China

The equal channel angular pressing (ECAP) were performed for SiCp reinforced AZ91D based composites prepared by stirring-casting process. The structures of AZ91D based composites and fracture morphology were observed and analyzed by optical microscope and scanning electron microscope after ECAP. The results show that the casting defects such as pores were eliminated, the size of grains was be reduced and the uniformity of particle was improved after ECAP. The connection of matrix and particles, the harm accommodation to SiCp also were improved; SiCp masses were the main cause of fracture of the composites with tenacious fracture of the matrix alloy companied with some disconnection between particles and matrix and the cross-grain fracture exists. After ECAP, principal part was tenacious fracture of the matrix with little quantitative cross-grain fracture in the structures and disconnection between particles and matrix was the main form in the area of dense distribution of SiCp.

11:45 AM
High Strength Mg-Zn-Y Alloys with Long Period Stacking Structure: *Yoshihito Kawamura*[1]; Shintaro Yoshimoto[2]; [1]Kumamoto University, Matls. Sci. & Tech., Kurokami 2-39-1, Kumamoto 860-8555 Japan; [2]Kumamoto University, Grad. Sch. of Sci. & Tech., Kurokami 2-39-1, Kumamoto 860-8555 Japan

Mg-Zn-Y alloys have a long period stacking ordered (LPSO) structure of 18R. We have previously reported the Mg97Zn1Y2 rapidly solidified powder metallurgy (RS P/M) alloy exhibited high yield strength (0.2% proof stress) of 610 MPa, elongation of 5%, a high-strain-rate superplasticity and high corrosion resistance. In this study, the effect of RS P/M processing on mechanical properties of LPSO-type Mg-Zn-Y alloys was investigated by comparing with ingot metallurgy (I/M) alloys that were produced by extruding cast ingot. The tensile yield strength of the I/M Mg97Zn1Y2 alloy was 375 MPa and 278 MPa at ambient temperature and 473 K, respectively. It was found that the RS P/M processing improved the tensile yield strength by 63% at ambient temperature and by 40% at 473 K in comparison with the I/M processing.

Materials Processing Fundamentals: Smelting & Refining I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Tuesday AM Room: 3001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

8:30 AM
Phase Field Model of Electrochemistry for the Ti-Mg-Cl Ternary System: *Wanida Pongsaksawad*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-043, Cambridge, MA 02139 USA

The mechanism of titanium product formation is investigated in the small-scale Electronically Mediated Reaction process (EMR). A Cahn-Hilliard model of electrochemistry for the Ti-Mg-Cl ternary system with composition-dependent mobility is applied to study morphology evolution of titanium sponge. The formulation used in this model assumes rapid charge redistribution in the electrolyte and transport-limited electrochemical reactions. Two-dimensional simulation results of a solid-liquid system with fluid flow are presented as a function of applied voltage, surface energy and viscosity, and as expected, the cathode interface is less stable under high electric field, low surface energy, and high viscosity. In addition, three dimensional results are presented illustrate the sponge formation relative to the two-dimensional case.

8:55 AM Cancelled
Mathematical Modeling of the Fray Farthing Chen (Ffc) Process for Titanium Extraction

9:20 AM
Dissolution of Copper Oxides in Molten Na2O- and CaO-Based Slags: *Peng Fan*[1]; Weol D. Cho[1]; [1]University of Utah, Dept. of Metallurgl. Engrg., Salt Lake City, UT 84112 USA

The dissolution rate and the solubility of copper oxides (CuO and Cu2O) in various Na2O-B2O3-SiO2 and CaO-B2O3-SiO2 slags have been studied at high temperatures. The effects of temperature and slag composition on the dissolution rate and the solubility have been determined for the two copper oxides. Based on the physical and chemical properties of the molten slags and the interaction between solid copper oxides and the molten fluxes, the mechanism of the dissolution is discussed.

9:45 AM
Main Parameters on the Performance of a Desulfurization of Ferro-Nickel by Slag Reaction: *Markus Hochenhofer*[1]; Helmut Antrekowitsch[1]; Matjaz Juhart[3]; [1]Christian-Doppler-Laboratory for Secondary Metallurgy of the Non-ferrous Metals, Franz-Josef-Strasse 18, Leoben 8700 Austria; [3]Treibacher Industrie AG, Althofen 9330 Austria

To produce ferroalloys like ferro-nickel for alloying purposes in a quality that meets the requirements of today's steel industry, refining processes like desulfurization and dephosphorization are indispensable. The performance of a desulfurization-treatment by slag reaction depends on various parameters, such as: sulfide capacity and viscosity of the slag, sulfur distribution between metal and slag, activity of sulfur

in the melt and movement of the melt. Within the framework of this work, the combined influences of these parameters on the desulfurization of a ferro-nickel alloy were examined. As a basis for this examination, a mathematical model, based on data from plant trials, was created. This model demonstrates the dependence of the desulfurization rate on the initial values of the above mentioned parameters. With this model the relevant parameters were evaluated and assessed concerning their influence on desulfurization.

10:05 AM Break

10:20 AM
Electro-Deoxidation of Solid Chromium Oxide in Molten Chloride Salts: *George Zheng Chen*[1]; Derek J. Fray[2]; Elena Gordo[3]; [1]University of Nottingham, Sch. of Chem. Environ. & Mining Engrg., Univ. Park, Nottingham NG7 2RD UK; [2]University of Cambridge, Dept. of Matl. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [3]University of Carlos III de Madrid, Dept. of Matl. Sci. & Metall. Engrg., Avda. de la Universidad 30, 28911 Madrid Spain

Chromium is industrially produced by aluminothermic reduction of Cr2O3 or electro-deposition from the aqueous NH4Cr(SO4)2 solution. The former makes more expensive products than what would be from an electrolytic method because aluminium is produced by electrolysis. In the aqueous electrolysis, the redox recycling of the multivalent chromium species between electrodes leads to low current efficiency (~45%) and high energy consumption (~18.5 kWh/kgCr). Recent reports have demonstrated that solid metal oxides can be directly electro-deoxidised to the respective metals/alloys in molten salts. For electrolysing solid Cr2O3, less than 0.2wt% oxygen could be achieved in the powdery product with the current efficiency and energy consumption being 75% and 5 kWh/kgCr, respectively. In this paper, previous work is reviewed in detail, followed by in-depth discussion on the mechanisms of (1) the electro-deoxidation process and (2) the formation of the cubic and nodular morphologies of the chromium powders obtained under different electrolysis conditions.

10:40 AM
Simulation of Reactive Pellets in a Pyrometallurgical Bath Using SPH: *Paul W. Cleary*[1]; Nick Stokes[1]; Joseph Ha[1]; Mahesh Prakash[1]; Geoff A. Brooks[2]; [1]CSIRO, Math. & Info. Scis., PB 10, Clayton S., Vic 3169 Australia; [2]CSIRO, Minls., Normanby Rd., Clayton S., Vic 3169 Australia

In several pyrometallurgical processes, reactive pellets are added to a melt. Heat is transferred to the pellets and reaction products such as gases, metal and slag are formed. For many pyrometallurgical processes traditional grid based CFD methods, such as using the Finite Element Method (FEM) and Finite Volume (or Control Volume methods), produce suitable predictions. However, the inclusion of solids in the bath represents significant challenges for these methods. Smoothed Particle Hydrodynamics (SPH), a Lagrangian simulation method, is able to simulate both the fluid component of the bath and any immersed solid materials, tracking the motion of these solids, and their interaction with the fluid, including gas generation. In this paper, we will summarize the key aspects of the SPH method and will show a series of structured examples illustrating the behaviour of the different physics sub-systems that are used in the model of the overall system.

11:00 AM
An Experimental Study on Floating Solids in a Liquid Bath: Mohamed Nabil Noui-Mehidi[1]; Hugh Blackburn[1]; Richard Manasseh[1]; *Geoff A. Brooks*[2]; Murray Rudman[1]; [1]CSIRO, Mfg. & Infrastruct. Tech., PO Box 56, Graham Rd., Highett, Melbourne, Victoria 3190 Australia; [2]CSIRO, Minls., PO Box 312, Clayton S., Melbourne, Victoria 3169 Australia

In some pyrometallurgical applications pelletised solids react in a bath of liquid metal at high temperatures. A key factor controlling the rate at which the reaction takes place is the extent and nature of the submergence process of the solids in the liquid bath. This study is concerned with an experimental investigation of the minimum speed and the associated power consumption to achieve the submergence of floating solids in a stirred vessel. The physical modeling was achieved by using water as liquid medium and pine wood pellets as buoyant particles. Particular attention was given to the effects of pellet size and pellets load on submergence. Power consumption was found to increased when larger pellets were used. The tests performed on the study of the load effect, have shown an increase in the power consumption when the total load increased; still higher power consumption was required to submerge larger pellet sizes.

11:20 AM
Reduction of Chromium Oxide from Liquid Slags: *Antonio Romero-Serrano*[1]; Juliana Gutierrez[1]; Victor Arredondo[1]; José Manuel Hallen[1]; [1]National Polytechnic Institute, Metall. & Matls. Dept., ESIQIE-IPN, Apdo. Postal 118-431, Mexico, D.F. 07051 Mexico

Experimental and theoretical analysis were carried out in this work to estimate the effect of slag basicity and amount of reducing agents on the reduction of chromium oxide from the slag which interacted with molten steel at 1600°C. The slag system contained CaO, MgO, SiO2, CaF2 and Cr2O3 together with Fe-alloys (Fe-Si or Fe-Si-Mg). Some experiments were also conducted to study the effect of initial Cr content in steel on the chromium oxide reduction. Three initial Cr contents were tested (0.15, 2 and 4 mass%). Argon was injected at the bottom of the furnace in these last experiments to increase the stirring of the system. Some estimations were made to determine the theoretical effect of temperature, slag basicity, (CaO+MgO)/SiO2, and amount of reducing agents in the slag on the chromium recovery. The FACT (Facility for the Analysis of Chemical Thermodynamics) computational package was used to determine the equilibrium between the slag and the molten steel.

11:40 AM
Studying Solvent Extraction Settler Process by Using CFD: *Timo Tapani Kankaanpää*[1]; [1]Helsinki University of Technology, Lab. of Matls. Procg. & Powder Metall., PO Box 6200, Espoo FIN-02015 HUT Finland

The liquid-liquid dispersion phases are separated by gravity in a solvent extraction settler. This separation step is made more effective by picket fences. The picket fence geometry and its physical placement have a significant effect on the separation process, because the purpose of a fence is to smoothen and control organic-aqueous dispersion flow in the first part of the settler and achieve a deep and dense dispersion layer in the front end of the settler resulting in a clean phase separation. In this study, physical phenomena in the solvent extraction process and phase separation in a solvent extraction settler with and without picket fences have been examined using a commercial CFX software package. It can be concluded that the used CFD-approach proved to be a complementary tool for optimizing and designing the solvent extraction settler.

12:00 PM
Mathematical Modeling of Molten Steel Flow in the Vacuum Circulation Refining Process: *Ji He Wei*[1]; Han Tao Hu[1]; Hui Fa Huang[1]; [1]Shanghai University, Dept. of Metallic Matls., 149 Yan Chang Rd., Shanghai 200072 China

A three-dimensional model for the flow of the molten steel in the whole unit during the RH refining process has been proposed and developed with considering the physical characteristics of the process, particularly the behaviors of gas-liquid two phase flow in the up-snorkel. The flow field of liquid, the gas holdup of the liquid phase in the up-snorkel and the circulation flow rate in a water model unit with an 1/5 linear scale of a 90 t RH degasser have been computed using this model. The results showed that the flow pattern in the whole RH unit could be well modeled by the model. The liquid can be fully mixed during the refining process except the area close to the free surface of liquid in the ladle and the zone between the two snorkels, but there is a boundary layer between the descending liquid stream from the down-snorkel and its surrounding liquid, which is a typical liquid-liquid two phase flow, and the molten steel in the ladle is not in a perfact mixing state. The lifting gas blown is rising mos tly near the up-snorkel wall, the flow pattern of the bubbles and liquid in the up-snorkel is closer to an annular flow. The calculated circulation flow rates are in good agreement with the measured values.

Mechanical Behavior of Thin Films and Small Structures: Stability, Strain and Stress

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Tuesday AM Room: 2024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Judy Schneider, Mississippi State University, Dept. of Mechl. Engrg., Mississippi State, MS 39762 USA; Carl V. Thompson, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA

8:30 AM Invited
Stress Evolution During Deposition of Polycrystalline and Epitaxial Films of Cu and Ag: *Carl V. Thompson*[1]; Cody A. Friesen[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Currently at Arizona State University, Cambridge, MA 02139 USA

Polycrystalline films can develop very large tensile or compressive stresses during deposition. We have characterized stress evolution during evaporative deposition and during interruptions of evaporative deposition of both polycrystalline and epitaxial thin films of Cu and Ag. In all cases, a reversible stress change is observed when growth is interrupted and resumed. We have correlated this stress change with changes in the surface defect structure, through both modeling and experimental characterization with reflected high energy electron diffraction. These results demonstrate that kinetic roughening of film surfaces during deposition leads to a compressive surface stress, and may also be related to mechanisms that result in compressive residual stresses. The compressive stress associated with kinetic roughening is superimposed on mechanisms that lead to residual tensile stresses, such as the development of coalescence strains in polycrystalline films and misfit strains in epitaxial films.

8:55 AM
The Effect of Linewidth Scaling on the Stress State of Passivated Interconnects: Application to Stress Voiding: *Raju V. Ramanujan*[1]; D. Ang[1]; C. Wong[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Blk. N4.1, Nanyang Ave., Singapore 639798 Singapore

Critical dimensions of interconnects are in the nanometer range, such small dimensions can lead to stress induced diffusive voiding (SIDV) failure. Experimentally, a complex dependence of mean time to failure on linewidth dimensions is observed. To rationalize these experimental results and to correctly extrapolate reliability results to finer interconnects, the effect of interconnect dimensions on SIDV was studied. This scaling effect was examined by numerical analysis and the Eshelby model; the effect of linewidth scaling on the hydrostatic stress in passivated metal lines was determined. An increasing trend of hydrostatic stress with linewidth for narrow lines, and decreasing trend of hydrostatic stress with linewidth for wide lines, with a maximum hydrostatic stress at a critical linewidth was observed. The hydrostatic stress in copper lines for all linewidths is larger than that in aluminium lines. The effect of grain boundaries on SIDV was also examined using this model.

9:10 AM
Atomistic Simulation of Stress Evolution in the Early Stages of Volmer-Weber Growth: *Chun-Wei Pao*[1]; Mikhail I. Mendelev[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Olden St., Princeton, NJ 08544 USA

Wafer curvature experiments show that during Volmer-Weber growth, the stresses in the islands are compressive; the magnitude of which increases as the islands grow. The origin of these stresses is speculative. We performed a series of hybrid static-relaxation/molecular dynamics simulations of the growth of islands on an amorphous substrate that confirm that compressive stresses form and increase as the islands grow. To identify the origin of this effect, we perform a series of simulations as a function of island/substrate bond strength. Stronger interfacial bonding leads to smaller wetting angle and larger compressive stresses. Weaker bonding leads to slipping at the island/substrate interface and islands that are nearly stress-free. These observations are consistent with experimental results in which stronger interfacial bonding leads to larger compressive stresses in the islands and smaller film thicknesses at which the ubiquitous development of tensile stresses during film growth occurs.

9:25 AM Invited
Adatom Diffusion on Strained FCC (001) Surfaces: Wei Xiao[1]; P. Alex Greaney[1]; *D. C. Chrzan*[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Berkeley, CA 94720-1760 USA

The growth of thin films often takes place in the presence of significant strains. These strains influence both the thermodynamics and kinetics of nucleation. Specifically, these strains may alter substantially the energy barriers associated with adatom diffusion. Further, under certain circumstances, surface strain can give rise to new types of adatom diffusion mechanisms. This talk considers the specific example of Cu adatom diffusion on strained Cu (001). Embedded atom method calculations employing the nudged-elastic-band method, are used to study adatom diffusion as a function of surface strain. These studies reveal interesting trends in adatom diffusion energy barriers, as well as the appearance of a surface crowdion mediated adatom transport mechanism. The structure and a rudimentary picture of the dynamics of these surface crowdions are presented. This work is supported by the National Science Foundation.

9:50 AM
Phase Transformation and Reorientation in Gold Nanowires: *Ken Gall*[1]; Jiankuai Diao[1]; Martin L. Dunn[1]; [1]University of Colorado, Dept. of Mechl. Engrg., Boulder, CO 80309 USA

Atomistic simulations with modified embedded atom method (MEAM), embedded atom method (EAM) and surface embedded atom method (SEAM) potentials reveal that, at certain sizes, a face centered cubic (fcc) gold <100> nanowire reorients into an fcc <110> nanowire. In MEAM simulations, the reorientation consists of two successive processes. First, surface stress and thermal vibrations cause the fcc <100> nanowire to transform into a body centered tetragonal (bct) nanowire. Second, the bct nanowire becomes unstable with respect to shear and transforms into an fcc <110> nanowire. In EAM and SEAM simulations a different reorientation mechanism exists. The surface stress in the fcc <100> nanowire induces slip on a {111}<112> system. Progressive slip on adjacent {111} planes changes the stacking sequence of these {111} planes from ABCABC to ACBACB, and the nanowire reorients into an fcc <110> nanowire. The difference in reorientation mechanism is rooted in the differences in the unstable stacking fault energy and orientation dependence of electron density in the potentials. In spite of these differences, the final structures of the reoriented nanowires are the same, which helps to explain why uniform fcc <110> nanowires are observed much more often in experiments than nanowires of other orientations. Results of preliminary first principal calculations are presented in an effort to better understand the semi-empirical results.

10:05 AM Break

10:20 AM Invited
Strain-Induced Coarsening in Nanograined Films: *John William Morris*[1]; Miao Jin[1]; Andrew M. Minor[2]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, 1 Cyclotron Rd., Berkeley, CA 94720 USA

In-situ nanoindentation within a transmission electron microscope has been used to study the deformation mechanisms in ultrafine-grained Al films. An initially surprising result of these studies was the frequent observation of spontaneous grain growth during indentation, which appears to be triggered by the deformation. This deformation-induced coarsening is clearly observed in micrograined Al films. In situ studies of nanograined films suggest that the same mechanisms are operative, though the difficulty of imaging nanosized grains makes the evidence less clear. The coarsening is promoted by two driving forces: decreasing the surface energy and increasing plastic work. The apparent mechanism is associated with the role of deformation in enhancing grain boundary mobility. The results suggest that grain growth and coalescence are important modes of response in the deformation of ultrafine- and nanograined materials.

10:45 AM
Thermomechanics of Thin Film Au for Micro/Nano Scale Engineering: *Ken Gall*[1]; David Miller[1]; Nancy Yang[1]; Cari Herrmann[1]; Hans Maier[1]; Steve George[1]; Conrad Stoldt[1]; [1]University of Colorado, Dept. of Mechl. Engrg., Boulder, CO 80309 USA

Gold is a promising material in emerging micro and nano systems owing to the ease of fabricating one- and two- dimensional Au nanostructures, its biocompatibility and capacity for biofunctionalization, and its favorable electrical properties. However, the thermomechancial properties of Au in various small-scale forms are not well characterized or understood, presenting a potential roadblock to the use of Au in devices. We have examined the thermomechanical behavior and microstructural evolution of gold thin films adhered to silicon microcantilevers with a chrome barrier layer. Such microcantilevers have application in optics, electronics (DC and RF), chemical and biological sensors, actuators, and fabrication process monitoring. Arrays of Au/Cr/Si microcantilever specimens subject to isothermal hold conditions exhibited dimensional instability that is dynamic in nature. We have characterized the thermomechanical response of our gold thin film based structures using curvature-temperature-time experiments in the temperature range from 50 to 225ºC. Drastic changes in curvature were observed for specimens annealed in air at 225ºC for less than 24 hours. The changes observed were significant and are certain to influence design performance as well as device reliability. Cross-sectional TEM evidenced an initial microstructure containing twins and dislocations that evolved during annealing. The free surface of the gold layer was observed to increase in roughness. FE-SEM imaging revealed extreme grain boundary grooving and grain growth. The observed changes in microstructure and morphology may be influenced by the diffusion and oxidation of the chrome layer. EDX analysis of the TEM foil specimens qualitatively suggests diffusion of the Cr barrier layer to the Au surface. Furthermore, it was found that the use of nanometer thick Atomic Layer Deposition (ALD) grown alumina coatings can greatly influence the evolution of the gold mcirostructure. Differences in mechanical and structural behavior were observed when surface coatings were applied. In particular, alumina coatings were observed to limit surface evolution, mitigate the migration of chrome, and suppress changes in curvature.

11:00 AM
Mechanical Property Enhancement of Silicon Component with Laser Shock Peening: *Gary J. Cheng*[1]; D. Pirzada[1]; Dave Bahr[1]; [1]Washington State University, Sch. of Mechl. & Matls. Engrg., Pullman, WA 99163-2920 USA

This paper will investigate a fundamental and general problem about processing of brittle material: How can we generate plastic deformation in silicon crystal to favorable stress/strain distribution for mechanical property enhancement? Silicon-based micro-components are dominant structural materials for micro-machines. However, silicon has the disadvantage of brittle material behavior. Fracture toughness of silicon is several orders lower than that of metals. Silicon thin film often fails under cyclic loading conditions even in ambient air and at room temperature. These result in serious problems when silicon-based materials are subjected to aggressive mechanical, thermal and chemical environments. However, it has been shown that silicon displays “metal like” stress-strain relationship, and high dislocation mobility at elevated temperature. This work will explore the silicon’s plastic behavior by laser shock peening and its benefit to mechanical property enhancement. Laser shock peening (LSP) experiments and thermomechanical simulation will be conducted. Mechanical properties (fatigue life and fracture toughness) will be predicted and compared to experiments. Nanoindetation testing will be used to investigate the changes in mechanical property. TEM will be employed to investigate the dislocation structure after LSP. X-ray diffraction will be used to measure the residual stress.

11:15 AM Invited
Thermo-Mechanical Stability of Metallic Nanolaminates: *Amit Misra*[1]; Richard G. Hoagland[1]; [1]Los Alamos National Laboratory, MST Div., MS G755, Los Alamos, NM 87545 USA

Sputter deposited metallic nanolaminates exhibit unusually high hardness when the bilayer periods approach nanometer dimensions. In this presentation, we report on the thermal and mechanical stability of sputter deposited Cu-Nb nanolaminate foils. The nanolaminates exhibit extraordinary plastic stability upon room temperature rolling undergoing uniform reduction in layer thickness to high levels of plastic strain. Large reduction in thickness is accomplished without the formation of the classical dislocation cell structures. Furthermore, no out-of-plane lattice rotations are observed. These results are interpreted in terms of symmetric slip occurring by the glide of single dislocations on multiple slip systems in both layers. The interface stability under mechanical straining is compared to the morphological stability of these nanolaminates following elevated temperature annealing. The effect of nanolayering length scale on the thermal and mechanical stability is also discussed. This research is funded by DOE, Office of Science, Office of Basic Energy Sciences.

11:40 AM
High Temperature Mechanical Properties of Cu/Nb Nanoscale Multilayers at Diminishing Length Scales: *Nathan A. Mara*[1]; Alla V. Sergueeva[1]; Tammy Tamayo[1]; Xinghang Zhang[2]; Amit Misra[3]; Amiya Mukherjee[1]; [1]University of California, Div. of Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

The microstructure and high temperature mechanical properties of textured, polycrystalline Cu-Nb nanolayered composites prepared by magnetron sputtering were evaluated. The layer thicknesses less than or equal to 75 nm were tested in an effort to investigate high temperature mechanical properties of freestanding thin film multilayers. Effects of decreasing layer thickness on high temperature properties are presented, and show a dependence of strength and ductility on layer thickness and test temperature. The deformed specimens were characterized using transmission electron microscopy. The role of elevated-temperature deformation mechanisms such as interlayer and grain boundary are discussed. This investigation is supported by the National Science Foundation, grant # NSF-DMR-0240144 and LANL CARE grant #69757. Work at LANL is supported by DOE, Office of Basic Energy Sciences.

11:55 AM
Stability of Nanoscale Twins in Sputtered 330 Stainless Steel Thin Films: *Xinghang Zhang*[1]; Amit Misra[2]; Haiyan Wang[2]; Richard G. Hoagland[2]; [1]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

We have recently discovered that sputter-deposited austenitic 330 stainless steel (330 SS) thin films have a nanoscale twinned structure. These twins are of {111} type with an average twin spacing of a few nanometers and the twin planes have a preferred orientation normal to the growth direction. The as-sputtered films have hardness values approaching 7 GPa, about an order of magnitude higher than that of bulk 330 SS. The unusually high strength originates from the high resistance of twin interfaces to slip transmission, as revealed by molecular dynamics simulations. In this paper we report on the thermal stability of these nanoscale twins. The evolution of mechanical behavior and electrical transport properties during annealing are correlated to the variation of microstructure such as the average twin spacing and columnar grain sizes. In addition, we explore the effect of residual stress on the formation of nanoscale twins in 330 SS.

12:10 PM
Crystal Growth and Superhardness Effect of Nano-Scale Multilayers: Nan Shao[1]; *Qianxi Lai*[1]; Yunshan Dong[1]; Fanghua Mei[1]; Geyang Li[1]; [1]Shanghai Jiao Tong University, Sch. of Matls. Sci. & Tech., Shanghai 200030 China

A comprehensive introduction is given to the coherent growth of stable phases, the stabilization of metastable phases and the crystallization of amorphous phases in magnetron sputtered nanomultilayers due to coherent interfaces by summarizing the recent work of authors. The superhardness effect resulting from this coherent growth is also discussed.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Diffusion and Atomistic Modeling

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Tuesday AM Room: 3000
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: B. B. Rath, Naval Research Laboratory, Matls. Sci. & Component Tech. Direct., Washington, DC 20375-5341 USA; S. Ankem, University of Maryland, Matls. Sci. & Engrg., College Park, MD 20742 USA

8:30 AM Invited
Atomistic Models of a Grain Boundary: *F. R.N. Nabarro*[1]; [1]University of the Witwatersrand, Sch. of Physics, PB 3, WITS 2050, Johannesburg S. Africa

The classical theory of grain boundaries is based on Bollmann's coincidence site and O lattice analysis. Special significance is given to those atomic sites in the lattice of grain 2 which would also be lattice sites or be close to lattice sites in grain 1 if that grain was extended into the domain of grain 2. No attention is given to the orientation of the grain boundary with respect to the lattices of the two grains. J.F. Nie and others have shown that in some systems the interface between two phases is governed by the requirements that lattice planes in the two phases should meet along a line lying in the interface. The physical basis is not clear, because there will generally be no atoms lying on this line. Moreover, the model does not predict discrete sets of relative orientations of the two grains and of the orientation of the boundary between them. Using the simple model of a square two-dimensional lattice, we attempt to relate these two models. For a pair of grains in contact, a low value of Bollmann's S indicates that special boundaries probably exist between the two grains. For such a case, we introduce a parameter S which depends on the orientation of the grain boundary. Boundaries with a low value of S/S are usually special. None of these models allow for physical considerations such as the directionality of bonding.

8:55 AM Invited
Coupling Grain Boundary Motion to Shear and Grain Rotation: John W. Cahn[2]; *Yuri Mishin*[1]; Akira Suzuki[1]; Jean E. Taylor[3]; [1]George Mason University, MSN 5C3, Sch. of Computatl. Scis., 4400 Univ. Dr., Fairfax, VA 22030 USA; [2]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., Gaithersburg, MD 20899-8555 USA; [3]New York University, Courant Inst. of Math. Scis., 251 Mercer St., New York, NY 10012 USA

We present a theory in which motion of grain boundaries (GBs) results in a coupled tangential shear of the region swept by the motion. This coupling of normal motion to tangential motion has been studied by molecular dynamics simulations on high- and low-angle tilt GBs. The coupling shear is found to be a discontinuous function of misorientation with a change in sign at a critical tilt angle. An understanding of the atomic mechanism has led to a prediction and verification of the misorientation dependence of the shear. Many disparate phenomena, and interrelationships between applied shear stress, normal GB motion, GB sliding, tangential motion of grains, and grain rotation, can be explained or reinterpreted with this theory. This work also sheds some light on some of J.C.M. Li's early results on subgrain rotation and the relationship between the curvature of a tilt GB and its velocity.

9:20 AM Invited
Grain Growth and Deformation in Nanocrystalline Materials: *Chandra Shekhar Pande*[1]; Robert A. Masumura[1]; [1]Naval Research Laboratory, Physl. Metall. Branch, Code 6325, Washington, DC 20375 USA

In case of nanocrystalline materials several additional features need to be taken into account in considering grain growth and deformation whose existence was first anticipated by Prof Li years ago. Grains can increase their size by grain rotation as well as by curvature driven motion. Grain rotation has been considered in detail by Li (J. C. M Li, J. Appl. Phys., 1962, 33, 2958.) theoretically and recently other researchers by simulation. We show that in nanocrystalline materials this mode is quite possible especially if the grain growth is retarded by finite triple junction mobility. New deformation modes occur in these materials leading to the so called Inverse Hall-Petch Effect which will be discussed in detail.

9:45 AM Invited
Spontaneous Sn Whisker Growth and Impression Creep: *K. N. Tu*[1]; [1]University of California, Matls. Sci. & Engrg., Los Angeles, CA 90095-1595 USA

Spontaneous Sn whisker growth is a creep phenomenon. Because it is spontaneous, stress relaxation is accompanied by stress generation. The latter is due to the room temperature reaction between Cu and Sn to form Cu6Sn5 in the grain boundaries of Sn. The in-diffusion of one Cu atom into Sn to grow Cu6Sn5 requires the out-diffusion of one Sn atom, roughly speaking, otherwise, compressive stress will build up in the Sn. The growth rate of a whisker from the diffusion of Sn atoms to the base of the whisker driven by a stress gradient is similar to the impression creep rate due to the out-diffusion of atom under the plunger at a very slow impression rate. On the other hand, the free surfaces of Sn and Sn whiskers have a protective oxide which tends to eliminate the source of vacancies needed for diffusion, hence the oxide affects greatly the spontaneous Sn whisker growth. In this talk, a mechanism of spontaneous Sn whisker growth will be presented.

10:10 AM Break

10:15 AM Invited
Atomistic Modeling for Heat Capacities of Carbon Nanotubes: Chunyu Li[1]; *Tsu-Wei Chou*[1]; [1]University of Delaware, Dept. of Mechl. Engrg., 126 Spencer Lab., Newark, DE 19716 USA

Carbon nanotubes have many potential applications, such as nanotube-reinforced composites, nanodevices, and nanowires. These applications take advantage of their remarkable mechanical and physical properties. Recently, increasing attention has been paid to the thermal behaviors of carbon nanotubes. Some experimental and theoretical studies have been devoted to the determination of their heat capacity. However, some important issues, such as the effects of tube diameter and tube chirality, and the differences in the heat capacity among isolated SWNTs, SWNT bundles and MWNTs, are still not clear. In this paper, we study the heat capacity of carbon nanotubes by using an atomistic modeling technique, namely, the molecular structural mechanics method. The vibrational modes of the nanotube are quantized according to the theory of quantum mechanics. The dependence of the heat capacities of carbon nanotubes on temperature, tube diameter and tube chirality is investigated and our modeling predictions are compared with existing results.

10:40 AM Invited
Molecular Potential Finite-Element Method (MPFEM) for Cell Mechanics: Ju Li[2]; Ming Dao[1]; *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg. & Div. of Biologl. Engrg., Rm. 8-303, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]Ohio State University, Dept. of Matls. Sci. & Engrg., 494 Watts Hall, 2041 Coll. Rd., Columbus, OH 43210 USA

Akin to the interatomic potential finite-element method (J. Mech. Phys. Solids 52, 691), we have developed an on-the-fly homogenization scheme for studying the mechanics of living cells that comprise two-dimensional/three-dimensional molecular networks as structural bases. For the particular case of the spectrin network undercoat of the human red blood cell (RBC) wall that provides most of its shear elasticity, we use the worm-like chain (WLC) potential for single spectrin molecular response. However more accurate molecular potentials, once measured from single-molecule stretching experiments, can be employed as well. Multiple local energy minima in RBC shape are found, that include the biconcave, the cup, and even randomly crumpled shapes. Optical tweezers stretching up to 100% elongation (mimicking RBC deformation in capillaries) are simulated and compared with experiments. MPFEM provides a bridge between whole-cell mechanics and single-molecule stretching response and the underlying structure of the spectrin network. Triangular, cubic and hexagonal spectrin networks with various defects and disorder are studied. We are also starting MPFEM calculations using real AFM scanned spectrin network micrographs as the homogenization area element for cell wall.

11:05 AM Invited
The Effect of Geometry on Chemical Stresses in Boundary Layer Diffusion: *Sanboh Lee*[1]; Sun-Chien Ko[1]; Y. T. Chou[2]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Kuang Fu Rd., 2nd Sec., Hsinchu, Taiwan 300 China; [2]University of California, Dept. of Chemi. Engrg. & Matls. Sci., Irvine, CA 92606 USA

Consider a layer A of thickness 2a sandwiched between two semi-infinite layers B of different chemical compositions. Three geometries of cross section are considered: slab, solid circle and square. Two types of diffusant sources are studied. One is the constant surface concentration source and the other is instantaneous surface concentration source. Assume that the diffusivity of diffusant in layer A is much greater than that in layer B. This problem has similar mathematical equations to the grain boundary diffusion. The concentration distribution in layer A of slab cross section was obtained using Fourier Laplace transform technique. This solution was applied to GaAs/AlAs/GaAs layer system. The stress distribution in layer A was solved using Hsueh method. Hsueh proposed three equations of total force, bending force and moment to solve bending axis, uniform strain and curvature. The stress distribution in layer A of circular cross section was solved the force equilibrium equations. The tangential and radial stresses were obtained in a close form. The force equilibrium equations of layer A of square cross section were digitized. The numerical solutions of stresses developed in layer of square cross section were obtained. They have common characterization that the maximum stresses occur near the free surface at a short time. A comparison among the chemical stresses in slab, solid circle and square cross sections are made.

11:30 AM
Interaction Between Diffusion and Chemical Stresses: *Fuqian Yang*[1]; [1]University of Kentucky, Chem. & Matls. Engrg., Lexington, KY 40506 USA

The interaction between chemical stresses and diffusion is studied. Based on the results of Li[1] and Larch and Cahn,[2,3] a new relation between hydrostatic stress and the concentration of solute is established. For a solid free of the action of body force, the Laplacian of the hydrostatic stress is proportional to the Laplacian of the concentration of solute - that is, deviation of the hydrostatic stress from its local average is proportional to deviation of the local concentration of solute. A general relationship among the surface concentration, the normal stress and the surface deformation of a solid is obtained, in which the normal stress is a function of the mean curvature of the undeformed surface and tangential components of the surface displacement. Using the new relationships, the evolution of chemical stresses in a thin plate is discussed. A closed-form solution of the steady state concentration of solute is derived. It turns out that linear distribution of solute is non-existe nt due to the interaction between chemical stresses and diffusion. [1]J.C.M. Li, Metall. Trans. 9A(1978) 1353-1380; [2]F.C. Larche and J.W. Cahn, Acta Metall. 30(1982) 1835; [3]F.C. Larche and J.W. Cahn, J. Res. Natl. Bur. Stand. 89(1984) 467.

11:50 AM Invited
Molecular Dynamics Simulation of Barnacle Cement: *Ya-Pu Zhao*[1]; Jun Yin[1]; [1]Chinese Academy of Sciences, State Key Lab. of Nonlinear Mech. (LNM), Inst. of Mech., Beijing 100080 China

Barnacle cement is an underwater adhesive that is used for permanent settlement. Its main components are insoluble protein complexes which have not been fully studied. In present article, we chose two proteins of barnacle cement for study, 36-KD protein and Mrcp-100k protein. In order to investigate the characteristics of above two proteins, we introduced the method of molecular modeling. And the simulation package GROMACS was used to simulate the behavior of these proteins. In this article, we mainly focused on two properties of these two proteins: structure stability and adhesive ability. First we simulated the structure stability of two proteins in vacuum, then the stability of 36-KD protein in solutions, in water and seawater environment was investigate. We found that the stability varies in the different environment. Next, to study adhesive ability of two proteins, we simulated the process of peeling the two proteins from the substrate (graphite). Then, we analyzed the main reason of these results. We fo und that hydrogen bond in proteins play an important role in the protein stability. Of course there are also other factors such as van der Waals (vdW) interactions and electrostatic interactions. In the process of the peeling, we used Lennard-Jones 12-6 formula to calculate the vdW interactions and found that it is vdW interactions that work effectively between proteins and substrate. At last, we introduced some theories to explain the results, compared the theoretical results with the results obtained by molecular dynamics (MD) simulations.

12:10 PM
Three-Dimensional (3D) Microstructure Visualization and Finite Element Modeling of the Mechanical Behavior of Heterogeneous Materials: Vasudevan Ganesh[1]; Rajen S. Sidhu[1]; *Nik Chawla*[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA

The mechanical behavior of materials is inherently controlled by microstructure. In particular, heterogeneous materials, consisting of two or more components or phases, have complex microstructures. This makes modeling of the mechanical behavior a challenge. We have developed a three dimensional (3D) approach to (a) constructing a "virtual microstructure" in 3D by serial sectioning technique, and (b) finite element modeling using the 3D microstructure as a basis. In this talk we will explore the fundamentals of the 3D virtual microstructure modeling methodology. This methodology was used to study the deformation behavior of two important systems, (i) SiC particle reinforced metal matrix composites, and (ii) Sn-3.5Ag solder alloys. The role of second phase fraction, morphology, and aspect ratio on deformation was quantified and will be discussed. Results from the microstructure based 3D simulations were found to be in good agreement with the experimental observations, indicating the importance and effectiveness of 3D microstructure-based simulations.

Microstructural Processes in Irradiated Materials: RPV Embrittlement and Oxide Dispersion Strengthened Alloys

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday AM Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Steve Zinkle, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA; Graeme J. Ackland, University of Edinburgh, Sch. of Physics, Edinburgh, Scotland EH9 2LZ UK

8:30 AM
Precipitation in Neutron Irradiated Copper Free RPV Steels: *G. Robert Odette*[1]; Mike K. Miller[2]; K. F. Russell[2]; Brian D. Wirth[3]; [1]University of California, Dept. of Mechl. Engrg., Santa Barbara, CA 93106 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6136 USA; [3]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA

The formation of Mn-Ni-Si phases that would produce large and unanticipated hardening and embrittlement in irradiated Cu-free reactor pressure vessel (RPV) steels was predicted more than a decade ago. Since large incubation doses were expected, these features were dubbed "late blooming phases" (LBP); however, LBP have proved to be an elusive quarry. Recent small angle neutron scattering, atom probe tomography and combined electrical resistivity Seebeck coefficient measurements have now provided unequivocal evidence of LBP in Cu-free alloys containing 1.6%Mn, 1.6%Ni, 0.25%Si and 0.005 to 0.040%P irradiated at 270°C at intermediate flux to ≅ 0.025 dpa. All techniques showed the Mn-Ni-Si rich features, with nominal volume fractions up to ≅ 0.6%, in the 0.005%P alloy; however, solute clusters were not observed in the atom probe tomography examinations of the alloy with 0.040%P. Hardening ranged from ≅ 160 to 190MPa, consistent with the estimated precipitate volume fractions.

8:50 AM
The Effects of Irradiation, Annealing and Reirradiation on an A533B RPV Steel: *Michael K. Miller*[1]; Randy K. Nanstad[1]; Mikhail A. Sokolov[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The mechanical properties, number density, size, and composition of precipitates in an A533B RPV steel have been characterized through two irradiation and annealing cycles. Atom probe tomography re-

vealed that irradiation (fluence = $5x10^{23}$ m^{-2}) (E>1MeV) produced a high number density of copper-, manganese-, nickel- and silicon-enriched precipitates. After annealing (168 h at 460°C), their number density significantly decreased. After irradiation (fluence = $0.85x10^{23}$ m^{-2}), annealing and re-irradiation (fluence = $0.85x10^{23}$ m^{-2}), a high number of copper-enriched precipitates was observed. A low number density of some large copper-rich precipitates was observed after the second annealing treatment. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy.

9:10 AM
Investigation of Irradiation-Induced Hardening in Low-Alloy Steels by STEM-XEDS Spectrum Imaging: *Masashi Watanabe*[1]; David B. Williams[1]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Properties and microstructures of materials can significantly be modified after irradiation. Irradiation-induced hardening is frequently observed in low-alloy steels. This phenomenon can be due to the formation of ultra-fine solute-enriched "clusters/precipitates." Previously, X-ray mapping using a scanning transmission electron microscope (STEM) equipped with an X-ray energy dispersive spectrometer (XEDS) has been employed and 2 to 3 nm Ni-enriched "precipitates" have been detected in the matrix. In this study, further quantitative STEM-XEDS mapping has been performed by using a spectrum imaging (SI) technique, which stores a whole spectrum at an individual pixel. In order to enhance weak signals from the fine precipitates, multivariate statistical analysis (MSA) has been applied to the measured SI data and the presence of Mn and Cu has been confirmed besides Ni in the fine precipitates. The combination of SI with MSA can be the ideal approach for investigation of fine features in complex microstructures.

9:30 AM
On the Correlation Between Matrix Damage and Cu-Precipitation: *Abderrahim Almazouzi*[1]; [1]SCK.CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

Within the framework of the european PERFECT project, binary Fe-Cu model alloys are used to assess the contribution of matrix damage and Cu-precipitates to the total strength hardening of the material. Positron annihilation techniques in conjunction with tensile testing are used to identify the effect of the microstructure on the materials behaviour. Both thermal aging and neutron irradiation have been performed in order to separate and quantify the obstacles as function of temperature, time, dose and flux.

9:50 AM
Solute Interaction with Point Defects in a Fe Under Irradiation: A Combined Ab Initio and Kinetic Monte Carlo Approach: *Edwige Vincent*[1]; Charlotte S. Becquart[1]; Christophe Domain[2]; [1]Université des Sciences et Technologies de Lille, Lab. de Métall. Physique & Génie des Matériaux, UMR 8517, Bat. C6, Villeneuve d'Ascq Cédex F-59655 France; [2]EDF-R&D, Dépt. MMC, Les Renardières, Moret sur Loing cédex F-77818 France

Solute Cu plays a major role in the embrittlement of pressure vessel steels under radiation. In RPV steels and dilute FeCu alloys, the Tomographic Atom Probe has revealed the formation of Cu atmospheres under neutron flux. More recently the role of other solutes such as Ni, Mn and Si which are also within the atmospheres have been put forward. It is thus very important to characterise the interactions of these solutes with radiation induced point defects in order to understand the elementary mechanisms behind the formation of these atmospheres. We have investigated by ab initio calculations based on the density functional theory the interactions of point defects and solute atoms in dilute FeX alloys (X = Cu, Mn, Ni or Si). The different possible configurations of small solute clusters, solute-vacancy complexes and solute-dumbbell complexes have been studied. Their formation and binding energies are discussed, as well as the migration energies for the most interesting configurations. These data have been used to derive a parameterisation to couple the diffusion of point defects with solute atoms, in order to simulate the evolution of solute atoms by Kinetic Monte Carlo. First results will be presented and compared to some experimental observations.

10:10 AM Break

10:40 AM Invited
Nano-Mesoscopic Structural Control in 9Cr-ODS Ferritic/Martensitic Steels: *Shigeharu Ukai*[1]; [1]Japan Nuclear Cycle Development Institute, Oarai Engrg. Ctr., Sys. Engrg. Tech. Div., 4002, Narita, Oarai-machi, Higashi-Ibaraki-Gun, Ibaraki-Prefecture 311-1393 Japan

The equi-axial grain structure of 9Cr-oxide dispersion strengthened (ODS) steels with a composition of 9Cr-0.13C-2W-0.2Ti-0.35Y2O3 is controlled by the alpha and gamma phase transformations. The appropriate selection of titanium and excess oxygen contents induces residual alpha grain formation, which is network-like shape and contains the ultra fine and dense Y2Ti2O7 type complex oxide particles with 1.5 nm size. The thermodynamic model calculation quantitatively revealed the condition of the residual alpha grain formation. In addition, for 9Cr-ODS steels containing the residual alpha grains, the critical cooling rate to induce martensitic phase transformation is as high as 10,000 K/h, due to one micron size of prior gamma grains. Prevention of martensitic phase transformation should suppress sliding among packet grain boundaries. It is concluded that excellent high temperature strength of 9Cr-ODS steels is significantly enhanced by the formation of network-like residual alpha grains containing ultra fine Y2Ti2O7 particles and packet-free grain boundaries.

11:20 AM
Precipitation and Stability of Nanometre Particles in ODS Alloys: Philippe Pareige[1]; *Mike Miller*[2]; David T. Hoelzer[2]; Emmanuel Cadel[1]; Roger E. Stoller[2]; [1]CNRS, ERT, Inst. des Matériaux, Ave. de l'Univ., St. Etienne du Rouvray BP12 76801 France; [2]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6136 USA

The microstructures of commercial mechanically-alloyed oxide dispersion strengthened alloys (MA957 and 12YWT) have been characterized in the as-received condition, after annealing up to 20h at 1300°C and after ion irradiation (Fe ions at 300°C up to 0.5 dpa for 12YWT). A comparison of the behaviour of these two materials before and after ageing is reported. Studies were performed by three dimensional atom probes that give access to information on the microstructure at the atomic scale. It has been revealed, that the Ti-,Y- and O- enriched particles are stable under thermal ageing or irradiation in the 12YWT. A slight coarsening of the particles is observed in the MA957 material after 24h at 1300°C and a high oxygen content was measured in the ferrite matrix. The lower molybdenum content in MA957 was found to be less effective in trapping oxygen than the higher tungsten content in 12YWT alloy.

11:40 AM
Molecular Dynamics Simulation of Primary Irradiation Defect Formation in Fe-Cr Alloys: *Jae-Hyeok Shim*[1]; *Brian D. Wirth*[1]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720 USA

Ferritic-martensitic Fe-Cr alloys represent a technologically important class of candidate materials for fusion first wall and blanket structures facilities. These alloys will experience severe irradiation environments including the creation of atomic displacements in high-energy displacement cascades and damaging concentrations of helium and hydrogen. We present the results of molecular dynamics simulations to investigate high energy displacement cascade evolution and the properties of point defect clusters in an Fe-10%Cr alloy. Finnis-Sinclair potentials for Fe and Cr are used to describe the interatomic behavior. A previously published Fe-Cr cross-potential, was slightly modified to fit the heat-of-mixing and lattice constant data of these alloys. Displacement cascade simulations with PKA energies of 20 and 40 keV were performed. The morphology, energetics and mobility of primary irradiation defects were characterized in terms of PKA energy and temperature, and compared to the cascade evolution and defect production in pure Fe.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Experimental Methods for Determining Diffusion Mechanisms

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Tuesday AM Room: 3007
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Yongho Sohn, University of Central Florida, Dept. of Mechl. Matls. & Aeros. Engrg., Orlando, FL 32816-2455 USA; Richard D. Sisson, Worcester Polytechnic, Ctr. for Heat Treat Excellence, Worcester, MA 01609 USA

8:30 AM Invited
Effects of Composition on Kinetics of Intermetallic Layer Growth for Soldered Copper Alloys: *Evan K. Ohriner*[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

The formation of intermetallic compounds on a number of copper alloys soldered with a variety of tin-containing solders has been studied for the temperature range of 150 to 250 C for times of up to 4000 hours. Tin-containing intermetallic layers exhibit parabolic layer growth under all conditions studied. Pure copper and Cu-5Sn-0.2P behave similarly, forming layers of Cu_3Sn and Cu_6Sn_5. A series of Cu-Ni-Sn alloys with nickel contents ranging from 6 to 23% form only a $(Cu,Ni)_6Sn_5$ intermetallic. The parabolic rate constant for layer growth is very sensitive to nickel content of the alloy, increasing rapidly to peak rate with additions of up to 9% nickel and then decreasing to a minimum rate with the highest nickel contents. The intermetallic layer thickness constant varies by a factor of 30 among the nickel-containing alloys. This unusual phenomenon is discussed.

9:00 AM
A Novel Double-Layered Titanium Boride Coating on Titanium: Kinetics of Boron Diffusion and Coating Development: *Nishant M. Tikekar*[1]; K. S. Ravi Chandran[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Increasing the surface performance of titanium and its alloys critically depends on finding an effective coating concept that can prevent galling, seizure and contact deformation of titanium surfaces. While there have been several externally applied coating concepts for titanium, none have been quite reliable. In this study, we performed solid-state diffusion experiments in a variety of boron-containing mediums to explore the possibility of making novel titanium boride coatings on titanium. The coating consisted of a double layer that involved a titanium diboride (TiB_2) top layer and a titanium monoboride (TiB) sublayer, which was primarily made of long, pristine TiB whiskers growing into titanium. Maximum thicknesses of about 10 μm TiB_2 and about 40 μm TiB whisker layer have been achieved by careful control of diffusion kinetics. The kinetics of diffusion was studied and was found to be governed by the ratio of the ingredients of the powder pack, the temperature and the time of the diffusion process. A two-layer diffusion model was developed to illustrate the nature of coating formation.

9:25 AM
Diffusion Mechanism in Two-Phase Intermetallic Titanium Aluminde Alloys: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsch., Geesthacht D-21502 Germany

Intermetallic titanium aluminides exhibit attractive thermo-physical properties, which give them the potential for extensive use as lightweight structural components. Engineering alloys are multiphase assemblies with complex constitution and microstructures. In the present paper the diffusion mechanisms occurring in these material will be investigated. Particular emphasis is placed on the effect of off-stoichiometric deviations, which produce a significant chemical disorder due to the formation of antisite defects. These defects gives rise to fast diffusion via antistructural bridges, which is particularly effective at low homologous temperature. The implication of this diffusion mechanism on the structural and mechanical properties of TiAl-alloys will be investigated. The major areas of the study involve: phase transformations, static and dynamic strain ageing, and creep.

9:50 AM
Grain Boundary Diffusion of Fe and Co in High Purity Iron: *Akiko Inoue*[1]; Koichi Takasawa[1]; Hiroyuki Nitta[2]; Junichi Koike[1]; Yoshiaki Iijima[3]; [1]Tohoku University, Matls. Sci., 02 Arakamaki Aobaku, Sendai, Miyagi 980-8579 Japan; [2]Tohoku Univerisity, Inst. for Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [3]Iwate University, Matls. Sci. & Tech., 3-18-8 Ueda, Morioka, Iwate 020-8551 Japan

It has been well known that the bulk self-diffusion in iron depends on the magnetic spin ordering. This study focuses on the influence of it on the grain boundary diffusion. The radioisotopes of ^{59}Fe and ^{57}Co were electroplated on the mirror-like surface of a high purity iron(C:0.7, S:1.0, N:0.7, O:2.0, P:1.0 mass ppm). The specimens were annealed under 10^{-4} Pa in the temperature range 553-1173K. A serial sectioning method was employed. At lower temperature range, the type C kinetics regime was applied. The grain boundary self-diffusion coefficient, D_{gb}^{Fe}, obtained in the present work was much larger than those by other authors. The activation energy of D_{gb}^{Fe} in the paramagnetic α-Fe was obtained to be 55.7kJ/mol which was only 0.22 of that for the bulk self-diffusion. Below the Curie temperature the decrease in both D_{gb}^{Fe} and D_{gb}^{Co} is remarkably larger than that in the bulk diffusion coefficients.

10:15 AM Break

10:30 AM
Interdiffusion in the Iron-Rich Part of the Ternary Fe-Cr-Al System: Experimental and Simulation Studies: *Guenter Borchardt*[1]; [1]TU Clausthal, Metall., Robert-Koch-Strasse 42, Clausthal-Zellerfeld 38678 Germany

Interdiffusion experiments were carried out on model alloys FexCryAlz with 0.57 x 0.80, 0.06 y 0.32, 0.01 z 0.29 in the temperature range 800°C T 1300°C. From the resulting concentration profiles the two main and two cross-interdiffusion coefficients required for the description of ternary diffusion were determined. For the calculation of the ternary diffusivities, a recent numerical approach was used in which the four ternary interdiffusion coefficients are determined over a selected composition range from a single diffusion couple experiment. The on-diagonal coefficients on both sides of the Matano interface were determined in the high and low Al and Cr concentration part of the diffusion couples. They show an Arrhenius behaviour with activation enthalpies between 158-203 kJ/mol for Cr-high regions, 144-228 kJ/mol for Cr-low regions, 171-221 kJ/mol for Al-high regions and 170-223 kJ/mol for Al-low regions.

10:55 AM
Precipitation-Strengthened Al-Sc-Ti Alloys Studied by Three-Dimensional Atom-Probe Microscopy: *Marsha E. van Dalen*[1]; David C. Dunand[1]; David N. Seidman[1]; [1]Northwestern University, Matls. Sci. & Engrg. Dept., 2220 N. Campus Dr., Evanston, IL 60208 USA

Currently, most precipitation-strengthened aluminum alloys are limited to usage at relatively low temperatures, because of the rapid coarsening and/or dissolution of their precipitates. Al-Sc alloys represent an exception, because they contain nanosize, coherent Al_3Sc precipitates (L12 structure) with low coarsening rates. In the present study, titanium was added as a ternary alloying element because it diffuses more slowly than scandium in aluminum and it has a high solubility in Al_3Sc. Al-Sc-Ti alloys are cast, solutionized and aged at temperatures in the range of 300-450°C to form nanosize, coherent $Al_3(Sc,Ti)$ precipitates within coarse aluminum grains. The Ti additions are found to decrease the coarsening kinetics as compared to Al-Sc alloys. The diffusion coefficient of Ti in Al is so small, however, that only a tiny concentration of Ti is detected in the precipitates by three-dimensional atom-probe microscopy. The resulting creep properties of the Al-Sc-Ti alloys are also discussed.

11:20 AM
Diffusion in Al-Ni-Ce Melts: *Axel Griesche*[1]; Michael-Peter Macht[1]; Günter Frohberg[2]; [1]Hahn-Meitner-Institute, Matls., Glienicker Str. 100, Berlin 14109 Germany; [2]Technical University Berlin, Inst. for Matl. Scis. & Tech., Hardenbergstr. 36, Berlin 10623 Germany

We investigate the influence of the melt's structure and the influence of thermodynamic forces on diffusion in the liquid state of ternary Al-based alloys. The long-capillary method was used to measure

self- and interdiffusion in liquid Al-Ni-Ce alloys at temperatures above liquidus. The interdiffusion coefficients Dik of all element pairs i-k were determined for a mean composition of Al87Ni10Ce3 at 1273 K and for a mean composition of Al77Ni20Ce3 at 1373 K. The self diffusion coefficients of Ni and Ce in liquid Al87Ni10Ce3 were measured by use of the penetration of enriched stable 62Ni- and enriched stable 142Ce-isotopes. Convective contributions to the mass transport were detected by measuring the time dependence of the diffusion coefficients and by direct observation with in-situ x-ray radioscopy. The concentration profiles were analyzed after solidification by means of energy-dispersive x-ray spectroscopy (EDS) in the case of chemical diffusion and by means of inductively coupled plasma mass spectrometry (ICP-MS) in the case of self diffusion. The chemical potentials of the elements in the melt were calculated using the commercial software Pandat. The influence of thermodynamic forces on diffusion is discussed in the framework of Darken's approach, which connects e.g. for a (solid) binary system the interdiffusion coefficient Dik with the self diffusion coefficients D'i and D'k by the equation Dik=(Nk·D'i+Ni·D'k)·F with F the thermodynamic factor representing a force due to gradients of the chemical potential and Ni and Nk the mole fractions of both elements. The influence of the structure of the melt on diffusion is discussed together with results of diffusion experiments from literature done by quasi-elastic neutron scattering and done by molecular dynamics simulations.

Neutron Diffraction Characterization of Mechanical Behavior: Deformation II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Tuesday AM Room: 3004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mark A.M. Bourke, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA; Ersan Ustundag, Iowa State University, Dept. of Matls. Sci. & Engrg., Ames, IA 50011 USA

8:30 AM Invited
Modeling Grain Interactions in Polycrystals with Crystals Discretized with Finite Elements: *Paul R. Dawson*[1]; Tong-Seok Han[1]; [1]Cornell University, Sibley Sch. of Mechl. & Aeros. Engrg., 196 Rhodes Hall, Ithaca, NY 14853 USA

Neutron diffraction data provide detailed information on the lattice strains during loading of polycrystalline solids that offer insight into the characteristics of load sharing among crystals that comprise the aggregate. Such data are particularly useful when combined with finite element modeling of polycrystals in which the influence of crystallographic neighborhood can be explicitly included in a simulation. This presentation summarizes the comparisons between diffraction experiments and finite element simulations of a two-phase (iron-copper) alloy. During loading into the fully plastic regime, the alloy demonstrates several stages in load sharing as each phase makes the transition from elastic to elastic-plastic behavior. Simulation results compare well with lattice strain histories for several combinations of scattering vector and crystal plane. From the simulations, the nature of the grain interactions that lead to the various stages can be explained.

8:50 AM
Effects of Grain Size on the Micromechanics of Deformation in Ni: *J. W.L. Pang*[1]; R. R. Rogge[2]; R. L. Donerberger[2]; W. Liu[3]; G. E. Ice[1]; [1]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, MS6118, Oak Ridge, TN 37831 USA; [2]National Research Council Canada, Steacie Inst. for Molecular Sci., Chalk River Lab., Chalk River, Ontario K0J 1J0 Canada; [3]University of Illinois, Frederick Seitz Matls. Rsch. Lab., 104 S. Goodwin Ave., Urbana, IL 61801-2902 USA

Capability of synergistic combination of neutron and synchrotron diffraction to provide the information for crystal plasticity modeling is presented. A series of Ni tensile samples of different grain sizes were measured by both techniques to investigate deformation behavior on the microscopic and mesoscopic length scales. Samples of grain size ranging from hundreds to few micrometers have been measured at different deformation levels with neutron diffraction. Results indicate that the trends in the microstrain evolution are similar for all grain sizes; however the strain magnitudes are generally larger for samples with smaller grain sizes. The deformed samples were then examined by the 3D X-Ray Crystal Microscope at the Argonne Photon Source. Variations in strains and misorientations within grains, subgrains and grain boundaries were determined. The experimental results were compared with the finite element deformation model.

9:10 AM
Evolution of Intergranular Stresses During In Situ Straining of IF Steel with Different Grain Sizes: *João Quinta da Fonseca*[1]; Pete S. Bate[1]; [1]University of Manchester, Sch. of Matls., Grosvenor St., Manchester, Greater Manchester M1 7HS UK

The heterogeneous nature of plastic deformation of polycrystalline metals at the microscopic scale gives rise to intergranular stresses, which influence fatigue, stress corrosion cracking and also skew residual stress measurements made by diffraction. Grain size is known to have a significant effect on the plastic deformation of metals, but the effect of grain size on the development of intergranular stresses has yet to be investigated. This article presents diffraction measurements obtained during in-situ uniaxial tensile straining of interstitial free (IF) steel with different grain sizes. These are compared to predictions made using crystal plasticity finite element modelling (CPFEM). As well as comparing mean elastic strains by tracking peak position, changes in peak width are correlated with slip activity and predicted spreads in elastic strains.

9:30 AM
Deformation Behavior of Fe-Cu Alloy Composites With a Bimodal Grain Size Distribution: *Jin-woo Jeon*[1]; Hahn Choo[1]; Peter K. Liaw[1]; Guojiang Fan[1]; [1]University of Tennessee, Matl. Sci. & Engrg., 318 Dougherty Hall, Knoxville, TN 37996 USA

Strengthening of an alloy is usually achieved at the expense of the ductility. However, recently developed alloys with so-called "bimodal grain size distribution" exhibit a unique mechanical properties, i.e., high strength with appreciable ductility. To investigate the micromechanics responsible for the unique mechanical behavior of the bimodal-grain-sized (BGS) alloys, immiscible Fe-Cu alloy composites were fabricated by sinter-forging of a mixture of ball-milled ultrafine Fe powder and coarse Cu powder. The microstructure and deformation behavior of the BSG Fe-Cu alloy composites were investigated using x-ray diffraction, transmission electron microscopy, tensile test, and nanoindentation test. Furthermore, in-situ tensile loading measurements were performed using neutron diffraction to investigate the intergranular strain evolution and load partitioning between the ultrafine (Fe) and coarse (Cu) grains.

9:50 AM Invited
Constitutive Behavior of Ferroelectrics: *Ersan Ustundag*[1]; Robert C. Rogan[1]; S. Maziar Motahari[1]; Mark R. Daymond[2]; [1]Iowa State University/Ames Laboratory, MSE Dept., 2220 Hoover Hall, Ames, IA 50011 USA; [2]ISIS Facility, Rutherford-Appleton Lab., Chilton, Didcot OX11 0QX UK

Ferroelectric materials exhibit a unique response to electromechanical loading and can be used as both sensors and actuators. This presentation will present recent results from in-situ uniaxial compression experiments on various Pb(Zr,Ti)O3 or PZTs using neutron diffraction to determine their constitutive behavior. PZTs near the edge of the morphotropic phase boundary as well as single phase (tetragonal and rhombohedral) specimens were investigated. Then a new self-consistent micromechanics model will be presented. This model considers different domain variants and attempts to estimate strain and texture (or domain switching) evolution during the loading of ferroelectrics. Finally, the diffraction data will be compared to the predictions of this model.

10:10 AM
In-Situ Tensile Loading Study of Nanocrystalline Nickel: *Xun-Li Wang*[1]; A. D. Stoica[1]; J. Almer[2]; C. T. Liu[3]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, Oak Ridge, TN 37831-6474 USA; [2]Argonne National Laboratory, Advd. Photon Source, 9700 Cass Ave., Argonne, IL 60439 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, Oak Ridge, TN 37831 USA

The deformation behavior in nanocrystalline nickel was investigated using high energy synchrotron radiation. The first five full diffraction rings were recorded allowing a complete grain orientation analysis of diffraction line shift and broadening. The evolution of

lattice strains was determined during the continuous tensile loading up to failure, as well as during the loading-unloading cycles. For a sample with a grain size of 15 nm, the fracture strength was found to be 1.3 GPa and the maximum recorded elongation was 3.8%. Besides the characteristic lattice strain behavior, the diffraction lines do not show any irreversible broadening after unloading, since the broadening accumulated during the plastic deformation is released upon unloading. In nanocrystalline materials the peak broadening due to strain heterogeneity is overshadowed by the grain size contribution. However, the quite high accuracy in peak profile analysis allows us to conclude that, in nanocrystalline nickel, the immobile dislocations are not accumulating during the plastic deformation, contrast to the coarse grained materials. This research was sponsored by Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

10:30 AM Break

10:50 AM Invited
Atomistic Mechanism of Plastic Deformation in Metallic Glasses: *Takeshi Egami*[1]; [1]University of Tennessee/Oak Ridge National Laboratory, MSE/Physics, 208 S. Coll., 1413 Cir. Dr., Knoxville, TN 37996 USA

While it is well established that mechanical deformation of crystalline materials occurs through the motion of dislocations, the atomistic mechanism of deformation in metallic glasses is poorly understood. It is often phenomenologically described in terms of free volume model, it is becoming clear that the reality of "free volume" is not vacancy-like as originally imagined, but is more collective involving a large number of atoms. On the other hand experimentally it has been observed that homogeneous anelastic deformation results in bond-orientational anisotropy. We propose an atomistic mechanism of plastic deformation in metallic glasses based upon stress-induced bond exchange mechanism. Even though the each element of space used for deformation is much smaller than the atomic size, thus not vacancy-like as free volume, because of the long-range stresses involved the total activation energy is large, comparable to that in crystalline solids. The implication of this model on ductility will be discussed.

11:10 AM Invited
Formation of Free Volume During Mechanical Deformation: *Matthew J. Kramer*[1]; Bulent Biner[1]; Dan J. Sordelet[1]; [1]Iowa State University, Ames Lab., 37 Wilhlem, Ames, IA 50011 USA

The atomic scale deformation in bulk metallic glasses has received considerable attention recently. At low temperature, deformation is concentrated in localized shear bands. At temperatures approaching the glass transition temperature up to the crystallization temperature, deformation appears to be homogeneous. Using high energy synchrotron radiation (~100 keV), we investigated the changes in the total scattering function and shifts in the average bond lengths to attempt to quantify the changes in free volume associated with the mechanical deformation. Room temperature indentation on Vitroloy showed a small decrease in the free volume ranging from 0.1 to 0.23%. High temperature creep experiments at 250 and 400 MPa showed about 0.35% increase in free volume. Experimental results will be compared to molecular dynamic calculations.

11:30 AM Invited
Icosahedral Order in Undercooled Metallic Liquids and the Influence on the Nucleation Barrier: *Kenneth Franklin Kelton*[1]; [1]Washington University, Dept. of Physics, Campus Box 1105, One Brookings Dr., St. Louis, MO 63130 USA

Over a half-century ago, Frank argued that liquid metals can be undercooled because of the development of icosahedral short-range order (ISRO) in the liquid. The existence of this ISRO (though often distorted) is supported by recent in-situ neutron and x-ray scattering studies of undercooled liquids that are levitated by electrostatic and electromagnetic methods. In a TiZrNi alloy, the developing ISRO favors the transformation of the liquid to a metastable icosahedral quasicrystal phase, instead of the stable tetrahedrally-coordinated crystal C14 Laves phase, demonstrating a clear connection between the nucleation barrier and the local structure of the liquid and verifying Frank's hypothesis. The consequences of this coupling between the order parameter characterizing the structure of the liquid and that for nucleation are discussed. Supported by NASA under contract NAG 8-1682, and by the NSF under grant DMR 03-07410.

11:50 AM Invited
The Mechanical Behavior of Ceramic Membranes in Oxygen Partial-Pressure Gradients: *James W. Richardson*[1]; Yaping Li[1]; Evan R. Maxey[1]; [1]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave, Argonne, IL 60439 USA

Dense ceramic components with mixed conduction properties and high oxygen permeability are important as membranes for oxygen separation and solid oxide fuel cell applications. Many of the most promising are perovskite-derived oxides, due to their structural stability over large ranges of chemical composition and oxygen vacancy concentration. Membranes are typically operated at elevated temperatures (800-1000°C) and exposed to large oxygen partial pressure (pO_2) gradients. A number of factors limit the lifetime of a membrane, including chemical decomposition, phase transformation and mechanical instability associated with internal strain generated by lattice parameter gradients. In-situ neutron diffraction, with a large beam, high penetrating power and sensitivity to scattering from oxygen, is extremely effective at characterizing ceramic oxide membranes in operational conditions. Studies of materials in the La-Sr-Fe-Co-O system under static reducing conditions show dramatic lattice expansion ($\Delta a/a$ up to $6x10^{-3}$) as a response to Co/Fe ionic radius changes. Measurements from membranes exposed to large oxygen partial pressure gradients (e.g., $pO_2 = 10^{-1}$ and 10^{-20} on opposing sides) - with corresponding structural gradients - across thin (~1mm thick) membrane tubes provide structural representations integrated across the structural gradients. As expected, the average structure has composition and lattice dimension intermediate between the two extremes represented by the surface environments. Unexpected, though, is the small distribution of composition and lattice dimension, with average near the conditions at the oxidizing surface. The implications for mechanical stability of ceramic membranes and importance of surface properties will be discussed. The Intense Pulsed Neutron Source at Argonne National Laboratory is funded by the U.S. Department of Energy under Contract W-31-109-ENG-38.

Neutron Scattering in Materials Research: Diffraction, Phases, and Micromechanics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Tuesday AM Room: 3022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brent Fultz, California Institute of Technology, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109-2136 USA

8:30 AM Welcome and Introduction: Brent Fultz and Michael Atzmon

8:35 AM Invited
Neutron Diffraction in Engineering Research: *Ersan Ustundag*[1]; [1]Iowa State University/Ames Laboratory, MSE Dept., 2220 Hoover Hall, Ames, IA 50011 USA

Neutron powder diffraction offers unique opportunities in engineering research by allowing in-situ studies of material deformation. In addition to phase information, one can also collect data on texture and lattice strain as a function of stress, temperature and sample environment. The diffraction data are then complemented with micromechanics modeling for full interpretation and to obtain the in-situ constitutive behavior of the material. The latter is very difficult to deduce from ex-situ tests and is crucial for predicting the long term performance in service. The recent construction of dedicated engineering diffractometers such as SMARTS and ENGIN-X has elevated the engineering neutron diffraction field to a new level of sophistication. Stresses exceeding 3 GPa, temperatures above 1500°C and numerous environments are now accessible. This presentation will describe recent work on bulk metallic glass composites, structural ceramics and metal matrix composites. It will also offer insight into exciting future developments.

9:05 AM Invited
Neutron Diffraction Studies of Mechanical Behavior: *Xun-Li Wang*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, Oak Ridge, TN 37831 USA

Since the 1980's, neutron diffraction has been used to study the mechanical behavior of materials. Much of the research in the early days was strongly oriented towards mechanical engineering, involving the determination of residual stress distribution in components and the use of these data in design and life-time predictions. Opportunities for fundamental research began to emerge when it became evident that some of the experimental data could not be understood within the framework of continuum theory and simple thermal-mechanical simulations. I will use recent experiments to illustrate what today's instruments are capable of, and their limitations. Examples will include fatigue behavior, deformation in nano-structured materials, and annealing-induced cracking in intermetallic composites. A number of new instruments are being built world-wide, increasingly adding new capabilities. The VULCAN diffractometer at the SNS is a new generation of diffractometers dedicated for users in materials science and engineering communities. Scientific opportunities with VULCAN will be discussed. This research was supported by U.S. Department of Energy, Basic Energy Sciences, Division of Materials Science and Engineering, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:35 AM
Finite Element Analysis for a Distributed Neutron Scattering Data Analysis Framework: *Cahit Can Aydiner*[1]; Ersan Ustundag[1]; [1]Iowa State University, Matls. Sci. & Engrg., 2220 Hoover Hall, Ames, IA 50011-2300 USA

Integration of data analysis and simulation tools used in neutron diffraction (ND) experiment evaluation offers unique opportunities in ND research. In this study, a scheme for integrating finite element analysis (FEA) to accompany engineering ND experiments is presented using the ABAQUS package. This scheme relies on modularizing the three stages of FEA: preprocessing (model definition), simulation and postprocessing. For the former, most commonly encountered samples and loading procedures in engineering ND experiments are used to form a model library. On the other hand, the advanced user is allowed to integrate a custom model into the framework. A similar module library and extension capability is provided for postprocessing. In addition, a user-defined subset of the model parameters are linked to a generic inverse solver that optimizes these parameters with the experimental output. This approach will be presented in conjunction with a new effort to perform distributed analysis of ND experiments.

10:05 AM Break

10:25 AM
Low Temperature Transformation of NiAlM Alloys: *Ling Yang*[1]; Xun-Li Wang[2]; Chain T. Liu[3]; Jaime A. Fernandez-Bacad[4]; James W. Richardson[5]; [1]University of Cincinnati, Cheml. & Matls. Sci., Cincinnati, OH USA; [2]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA; [4]Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN USA; [5]Argonne National Laboratory, Intense Pulsed Neutron Source, Argonne, IL USA

A systematic study of NiAlM (M=Ni, Fe, Co) alloys is carried out using neutron diffraction. The result shows that when Al atoms are partially replaced by transitional metal atoms ($Ni_{60-x}M_xAl_{40}$), there is a phase transition at very low temperature(~20K), while alloys with transitional metal atoms at Ni sites ($Ni_{50-x}M_xAl_{50}$) maintain a stable B2 structure down to 10K. Clearly, the addition of transitional metal atoms (Ni+M) at Al sites destabilizes the B2 structure and the transition depends on the fraction of (Ni+M) on Al sites. However, the observed transition does not seem to be related to magnetism, and the new phase does not match any of the known martensite. Instead, we found that the new peaks can be well indexed with a slightly distorted double-lattice superstructure. These observations suggest that the new phase is formed by re-ordering of the local atoms, which is fascinating to occur at such low temperatures.

10:45 AM
Evolution of Nanocluster, Dislocation and Grain Structure in Nanostructured Ferritic Alloy MA957 at Elevated Temperatures: *Matthew J. Alinger*[1]; G. Robert Odette[1]; Hirotatsu Kishimoto[2]; [1]University of California, Dept. of Matls., Santa Barbara, CA 93106 USA; [2]Kyoto University, Inst. of Advd. Energy, Gokasho, Uji, Kyoto 611-0011 Japan

So-called Nanostructured Ferritic Alloys (NFAs) containing 12-14Cr and a high density of nanoscale clusters (NCs) of Y-Ti-O exhibit superior creep strength and potential for high resistance to radiation damage. The coarsening-oxide transformation kinetics of the nm-scale precipitates in MA957 were characterized for anneals for various combinations of times from 1/3 to 480h and temperatures from 1150 to 1400°C. Small angle neutron scattering (SANS) was used to quantify the NC evolution, and TEM was used to observe the corresponding changes in the dislocation and grain structure and the formation of oxide phases. The NC coarsening and transformations can be approximately described by a pipe diffusion-type kinetics mechanism, with a time exponent of ≅ 1/5 and a high effective activation energy of ≅ 660 kJ/mole. This very high activation energy is believed to be due to the very low solubility of Y.

11:05 AM
A Neutron Diffraction Study of Phase Transformation by Tracking Texture Evolution with Temperature in Ti-6Al-4V: *Dhriti Bhattacharyya*[1]; G. B. Viswanathan[1]; S. C. Vogel[2]; D. J. Williams[2]; V. Venkatesh[3]; H. L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, LANSCE, Los Alamos, NM USA; [3]TIMET, R&D, 8000 W. Lake Mead Dr., Henderson, NV 89015 USA

The two phase (alpha/beta) Ti alloy Ti-6Al-4V is considered to be a workhorse alloy for its many applications in these diverse fields. One of the major aspects of this alloy which is not yet well understood is, whether the beta that forms at high temperatures is nucleated freshly from the alpha phase or whether it grows from the beta preexisting at room temperature. Another important, and as yet unresolved, question is whether the alpha phase maintains the Burgers Orientation Relationship (BOR) with the beta phase even after cold work and recrystallization. A unique way to address these issues is to monitor the development of texture in this microstructure as a function of temperature. The present study attempts to do this by carrying out, for the first time, high temperature in-situ texture measurements of TI-6Al-4V using the HIPPO instrument at LANSCE. This instrument enables texture measurements by neutron diffraction at various temperatures with the sample located inside a vacuum furnace. The textures of the alpha and the beta phases measured at room temperature, 800°C and 1020°C provided fresh evidence which indicate definite answers to the questions posed above.

11:25 AM
Orientation Distributions of Diamond Particles in Polycrystalline Diamond Synthetic Cells: *Jun-Yun Kang*[1]; Suk Hoon Kang[1]; Kyu Hwan Oh[1]; Hu-Chul Lee[1]; [1]Seoul National University, Sch. of Matls. Sci. & Engrg., Shinrim 9-dong, Kwanak-gu, Seoul 151-744 Korea

The orientation distributions of diamonds in two types of synthetic cells were examined. From the orientation distribution function (ODF) calculated from EBSD data, it was concluded that the diamonds in layered cell exhibited weak texture of <111> parallel to the stacking direction. And from the neutron diffraction spectrum of powdered cell, the texture indices of diamond {111}, {220} and {311} planes were calculated as 1.18, 0.89 and 0.90, respectively, which implied that the orientation distribution of the diamonds approached random distribution. In layered cell, there exists concentration gradient of carbon around growing diamonds along the stacking direction. By contrast, in powdered cell, diamonds are surrounded by homogeneous carbon solution. This discrepancy was considered to lead to the different orientation distributions of diamonds in the two cells. The favored alignment of {111} planes perpendicular to the stacking direction in layered cell was thought to originate from their lowest surface energy.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Aging, Crystallographic Texturing and Characterization of Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Tuesday AM Room: 3016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Iver E. Anderson, Ames Laboratory/Iowa State University, Matls. & Engrg. Physics, Ames, IA 50011 USA; Lawrence P. Lehman, Binghamton University, Dept. of Physics, Binghamton, NY 13902 USA

8:30 AM Invited
Void Suppression in Thermal Aging of Tin-Silver-Copper-X Solder Joints: *Iver Eric Anderson*[1]; Joel L. Harringa[1]; Sung K. Kang[2]; Bruce A. Cook[1]; [1]Ames Laboratory (USDOE), Iowa State University, Matls. & Engrg. Physics, 222 Metals Dvlp. Bldg., Ames, IA 50011 USA; [2]IBM T. J. Watson Research Center, Microelect. Packing Tech., 1101 Kitchawan Rd., Rte. 134, PO Box 218, Yorktown Hgts., NY 10598 USA

Recent work demonstrated the strategy of modifying a strong (high Cu) Sn-Ag-Cu (SAC) solder alloy with a substitutional alloy addition (X=Co, Fe) for Cu to retain solder joints with strength and ductility after aging at 150°C for up to 1000 h. This is critical to Pb-free assembly of portable electronics with potential for drop impact failure of interconnects. The SAC + X solder alloy joints appear to suppress the interdiffusion of Sn and Cu through Sn-Cu intermetallic layers to inhibit the formation and linkage of voids at the Cu substrate/Cu3Sn interface and, thus, to prevent embrittlement on aging. Other recent work identified Cu as the fast diffusing species that leads to voiding/embrittlement, albeit at lower aging temperatures. Void distributions in the Cu/solder interface region were analyzed on aging (at 150°C) in SAC and SAC + X solder joints to gain understanding of the limiting diffusion species and mechanism. Supported by ISU Research Foundation and USDOE-BES (W-7405-Eng-82).

9:00 AM
Microstructure and Shear Strength Evolution of Sn-Ag-Cu Solder Bumps During Aging at Different Temperatures: Dezhi Li[1]; *Changqing Liu*[1]; Paul P. Conway[1]; [1]Loughborough University, Wolfson Sch. of Mechl. & Mfg. Engrg., Loughborough, Leicestershire LE11 3TU UK

Flip chip devices with Sn-3.8Ag-0.7Cu solder on electroless Ni UBM were studied after aging at both 80°C and 150°C. The shear strength of the solder bumps was tested and the microstructure evolution was studied by SEM with EDX and EBSD technique. When the chips were aged at 80°C, a (Cu,Ni)6Sn5 IMC layer grew very slowly during the aging and the increase of the IMC thickness was not significant. Also, no Kirkendall voids were found under SEM examination. However, when the chips were aged at 150°C, the increase of the IMC thickness became very clear. Some Kirkendall voids appeared even after the chips were aged at 150°C for only 2 days. During the aging, the (Cu,Ni)6Sn5 IMC changed from a scallop-like formation to a faceted shape. The shear tests showed that the shear strength of solder bumps dropped at some point during the aging and then almost kept at a consistent value during the later aging periods. All the solder bumps were found to fracture in the bulk solder.

9:20 AM
Microstructure Evolution of Gold-Tin Eutectic Solder on Cu and Ni Substrates: *Jui-Yun Tsai*[1]; C. W. Chang[1]; C. E. Ho[1]; Y. L. Lin[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matl. Engrg., No 300, Jung-da Rd., Chungli City Taiwan 320

The microstructures of Au20Sn/Ni and Au20Sn/Cu that had been reacted in different bonding conditions first, and then aged were studied. An Sn/Au/Ni sandwich structure (2.5/3.75/2 μm) and an Sn/Au/Ni sandwich structure (1.83/2.74/5.8 μm) were deposited over the Si wafer, respectively. The overall composition of the Au and Sn layers corresponded to the Au20Sn binary eutectic (wt.%). The microstructures of Au20Sn solders on Ni and Cu were controlled by the bonding conditions. When the reaction condition was 290°C for 2 min, the microstructure of and Au20Sn/Ni and Au20Sn/Cu was both a two-phase (Au5Sn and AuSn) eutectic microstructure. The difference between Au20Sn/Cu and Au20Sn/Ni is that (Au, Ni)Sn formed next to Ni and (Au, Cu)5Sn formed next to Cu. When the bonding condition was at 240°C for 2 min, an layered microstructure was produced (AuSn/Au5Sn/Ni and AuSn/Au5Sn/Cu). The thermal stability of the as-bonded samples were also studied. Many interesting microstructure evolutions were observed. In general, the thermal stability for samples on Ni was better than that of on Cu when the bonded samples were subjected to long-term aging at 240°C.

9:40 AM
Interfacial Reaction Between 42Sn/58Bi Solder and Electoless Ni-P/Immersion Au UBM During Thermal Aging: *Moon Gi Cho*[1]; Kyung Wook Paik[1]; Hyuck Mo Lee[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., Kusung-Dong 373-1, Yusung-Gu, Taejon 305-701 Korea

The interfacial reaction between the 42Sn/58Bi solder and the electoless Ni-P/immersion Au UBM has been investigated before and after thermal aging, with a focus on formation of intermetallic compound (IMC), interfacial reaction rate, and bump shear strength. The immersion Au layer with a thickness of 0 (bare Ni), 0.1 and 1 μm was plated on the electroless Ni-5~6at.%P (6 μm) layer. Then, 42Sn/58Bi solder balls were fabricated on UBM by screen-printing and reflow. The IMC layer composed of Ni3Sn4 grains was formed at the join interface after reflow. On aging at 125°C, a ternary IMC phase was observed on the Ni3Sn4 layer in case of Au plating, which was identified as (Au,Ni)Sn4. Its thickness reached 7 μm and higher after aging for 1000 hr, and the Ni-P UBM was consumed by more than 1.2 μm. The thick (Au, Ni)Sn4 IMC layer deteriorated the integrity of the solder joint and thus the shear strength of the solder bump decreased by about 30% compared with non-aged joints.

10:00 AM Invited
Formation and Growth of IMC During Thermomechanical Fatigue of Sn-Based Solders: J. G. Lee[1]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824 USA

Sn-based solder joints exhibit significant aging from thermomechanical fatigue (TMF) treatments with longer dwell times at elevated temperature extreme. In the TMF cycles employed in this study the total accrued aging time at the high temperature extreme of 150°C could be about 2000 hours for about 1000 TMF cycles. Formation and growth of IMC phases in ternary and quaternary eutectic Sn-Ag based alloys with Cu and (Cu+Ni) additions due to such treatments, and their relative importance in the damage accumulation that results in deterioration of properties will be discussed. Work supported by the National Science Foundation under grant NSF DMR-0081796 and NSF DMI-0339898.

10:20 AM Break

10:30 AM
Distinct Reaction Morphologies of Two High Lead Solders, 90Pb10Sn and 95Pb5Sn, on Cu UBM During Solid-State Aging: Jin-Wook Jang[1]; Lakshmi N. Ramanathan[1]; Jong-Kai Lin[1]; *Darrel R. Frear*[1]; [1]Freescale Semiconductor, FMTC, 2100 E. Elliot Rd., MD EL725, Tempe, AZ 85284 USA

The solid-state reaction of two high lead solders, 90Pb10Sn and 95Pb5Sn, on Cu UBM were examined. Upon reflow, the Cu3Sn intermetallics formed on Cu UBM for both solder alloys, but solid-state aging produced significantly different reaction morphologies. For 90Pb10Sn solder, the Cu3Sn intermetallics continued to grow during solid-state aging at 170°C for 1500 hrs whereas 95Pb5Sn solder showed very limited intermetallic growth and the subsequent spalling of Cu3Sn from Cu UBM was observed. Such a difference is explained by a two-step mechanism: the Sn diffusion from bulk solder to the solder/Cu3Sn interface and the subsequent intermetallic formation by interdiffusion of Cu and Sn atoms. It was postulated that the competition between

these two kinetic factors leads to such a big morphological difference. The spalling phenomenon of Cu3Sn intermetallics in 95Pb5Sn solder was due to the loss of chemical adhesion between the Cu3Sn intermetallics and Cu UBM. Thermodynamic interpretation of spalling phenomenon showed that the interfacial free energy without spalling is greater than that with spalling after solid-state aging.

10:50 AM
Cyclic Twin Nucleation in Tin Based Alloys: *Lawrence P. Lehman*[1]; Yan Xing[1]; Ju Wang[1]; Kara Mather[1]; Thomas R. Bieler[2]; Adwait U. Telang[2]; Eric J. Cotts[1]; [1]Binghamton University, Dept. of Physics, Sci. II, PO Box 6000, Binghamton, NY 13902-6000 USA; [2]Michigan State University, Dept. Cheml. Engrg. & Matls. Sci., 2527 Engrg. Bldg., E. Lansing, MI 48824-1226 USA

Most Pb free solders are based on tin, with small additions of silver and copper. The mechanical and physical properties of these solders depend upon the slip and recovery/creep properties of the tin microstructure. The anisotropic nature of tin's mechanical properties means that the number and relative orientation of the tin grains in a solder joint will strongly influence its mechanical response. SnAgCu solder joints are often comprised of essentially one tin grain, or at most, a few dominant orientations. Twinning during Sn solidification is common, resulting in highly preferred, ~60° misorientations between neighboring Sn grains. We explore the source, crystallography and growth of cyclic twinned microstructures in tin based solders. Models of the geometry of twinning on different crystallographic planes can account for different growth morphologies, and different physical manifestations of cyclic twinning.

11:10 AM
Microtextural Analysis of Lead Free Solder Alloys: *Vineet Kumar*[1]; Zhigang Zak Fang[1]; Jin Liang[2]; Nader Dariavach[2]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [2]EMC2 Corp., 176 South St., Hopkinton, MA 01748 USA

There is a thrust to develop lead free solder alloys, having better or at least similar properties to conventional tin-lead solder alloys. Tin based eutectic and near-eutectic compositions similar to Sn-4Ag-1Cu are most promising alloys. However, there is still a lack of comprehensive understanding of the microstructure of these materials, hence their dependence on processing variable. In order to achieve best possible properties a comprehensive physical metallurgical analysis is required. In this direction, for alloy Sn-3.8Ag-0.7Cu, microstructural and microtextural analysis was done using Scanning Electron Microscope (SEM) and Electron Back-Scattered Diffraction (EBSD) technique. Composition and crystallographic details for different phases were measured and it was found that the intermetallics had a different composition from that of reported in literature. Orientation of different microstructural components was found. Misorientation relationship between different microstructural constituents including copper substrate was found. It is found that intermetallics have a definitive misorientation relationship with tin matrix and copper substrate.

11:30 AM
Effect of Cu on Microstructure and Grain Boundary Character in Sn-3.0Ag-Cu Solder Balls Solidified at 1/s: Adwait U. Telang[1]; *Thomas R. Bieler*[1]; Yan Xing[2]; Larry P. Lehman[2]; Eric J. Cotts[2]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]Binghamton University, Physics, Matls. Sci. Prog., Binghamton, NY 13902 USA

The solder alloy composition and cooling rate is known to have a large effect on microstructure of lead free solder joints. However, the mechanisms by which alloy composition influence solidification microstructures are unknown. In this study, 1 mm diameter solder balls having 3.0 wt% Ag with Cu levels of 0, 0.27, 0.59, 0.86, 1.1, and 1.4 wt% Cu were remelted and solidified at a cooling rate of 1/s, and examined in cross polarized light microscopy. With increasing Cu content, the size of the crystalline domains increased from about 50 microns with no Cu, to about 250 microns with 1.4 Cu. Using orientation imaging microscopy, grain orientation maps and misorientation statistics will be presented to identify the grain boundary character, and from this, the effect of Cu on possible solidification mechanisms will be discussed.

11:50 AM
Novel Characterizing Technique in Phase Distribution with Sample Preparation for Application in Solder Joint, Wire Bonding Chip and Li-Ion Battery Assembly: *Hung-Kai Chen*[1]; Shih-Hai Li[1]; *Jenq-Gong Duh*[2]; [1]National Tsing Hua University, Dept. of Engrg. & Sys. Sci., 101 Sec.2 Kunag-Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Dept. of Matl. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Characterization of solder joint microstructure is essential in the evaluation of microelectronic packaging reliability. Analyzing technique and sample preparation play important roles in the material characterization. This study demonstrates several novel approaches in the microstructure evaluation of solder joint. By utilizing the precision etching and coating technique, the interface of the IC packaging chip between the solder and under bump metallization (UBM) would be observed much more precisely and the intermetallic compounds could be revealed in details under field-emission scanning electron microscope (FE-SEM). For the interface of Sn-Pb/Ni/Cu, the intermetallic compound, defined as Ni_3Sn_4 and Cu_6Sn_5 by electron probe microanalyzer (EPMA), would form between the nickel and the solder. By adjusting the operating variables, such as etching energy, etching period, incident angle between the ion beam and sample, the time for sample preparation was significant reduced. For an example, when the cross-section surface of Sn-Pb solder specimen was perpendicular to the incident ion beam, the best quality of the SEM micrographs could be obtained either at 2.5 keV with an etching time for 7 min or at 4.5 keV for 3 min. In addition, the diffusion phenomenon of zinc in ZnO-coated $LiCoO_2$ powder and the redistribution of boron in LiBO-coated $LiMn_2O_4$ powder could be clearly revealed and evidenced by field-emission electron probe microanalyzer (FE-EPMA) with special sample preparation technology.

TUESDAY AM

Phase Transformations Within Small-Size Systems: Phase Separation, Precipitation and Displacive Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee
Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Tuesday AM, February 15, 2005 — Room: 3002, Location: Moscone West Convention Center

Session Chairs: David N. Seidman, Northwestern University, Matls. Sci. & Engrg., Evanston, IL 15229-3180 USA; Vijay K. Vasudevan, University of Cincinnati, Cheml. & Matls. Engrg., Cincinnati, OH 45221-0012 USA

8:30 AM Invited
Effect of Surface Stress on the Coherent Phase Equilibria of Nanoparticles: *Joo-Youl Huh*[1]; William C. Johnson[2]; James M. Howe[2]; [1]Korea University, Div. of Matls. Sci. & Engrg., 5-1, Anam-Dong, Sungbuk-Ku, Seoul 136-701 Korea; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., PO Box 400745, Charlottesville, VA 22904-4745 USA

As motivated by the recent observations on the stabilization of an ordered phase ($L1_2$) in Ag-Cu-Ag nanoparticles at temperatures more than 250°C above the bulk ordering temperature, we investigated the influence of particle size on coherent phase equilibria of a binary, spherical particle by considering the alteration of elastic fields caused by surface stress. The coexisting phases in a spherical particle are assumed to be configured as concentric shells. In the stress-free state, the binary system exhibits a miscibility gap ($\alpha_1 + \alpha_2$) at high temperatures and a eutectoid transition to form an ordered phase ($L1_2$) at a low temperature. Phase diagram constructions show that, depending on the particle size and materials parameters, the interaction between surface stress and misfit strains can stabilize either the $\alpha_1 + \alpha_2$ two-phase state by suppressing completely the $L1_2$ phase formation or the $\alpha_1 + L1_2$ two-phase state until it transforms to a single-phase solid solution at high temperatures. This talk will also discuss possible effects of compositional strains on the phase stability of nanoparticles.

9:05 AM
Phase Transformations Within Nanoparticles of Aluminum Alloys: Jixiong Han[1]; Martin J. Pluth[1]; Kazuo Furuya[2]; Jainagesh A. Sekhar[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Cheml. & Matls. Engrg., 401 Rhodes Hall, ML 0012, Cincinnati, OH

45221-0012 USA; [2]National Institute for Materials Science, Nanocharacterization Lab., 3-13 Sakura, Tsukuba, Ibaraki 305-0003 Japan

Phase transformations within nanoparticles of Al alloys were studied. Nanoparticles of binary Al-Cu and Al-Zn alloys were synthesized by plasma ablation of precursor ingots and the structure of these particles as well structural changes in these on aging at temperatures between 65-190°C for times to 100h studied by electron diffraction, nanoprobe energy dispersive x-ray spectroscopy and HRTEM. The particles were supersaturated fcc state in both cases, but displayed a variation in the individual particle composition when compared with the precursor bulk alloys. A 3-5 nm thick oxide layer was present around all the particles. On aging the Al-Cu nanoparticles, a precipitation sequence consisting of nearly pure Cu precipitates to θ' to the equilibrium θ was observed, with all three forming only along the outer oxide-particle interior interface. The structure of θ' and its interface with the Al matrix was characterized in detail. In the Al-Zn alloy, a spinodal structure was noted in the as-synthesized nanoparticle, which coarsened on aging into a fine scale structure composed of f.c.c twin-related platelets within which were contained platelets with a hcp structure. This morphology led to relatively complicated diffraction effects, which were analyzed in detail. Nearly-pure Zn precipitates, with an hcp structure, also formed along the oxide-particle interface and consumed the spinodal structure with time. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. In addition, the synthesis of and precipitation behavior in ultrafine (5-25 nm) Al-Cu nanoparticles will be presented. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

9:30 AM
Microstructure and Mechanical Behavior of Fe-20Ni-25Mn-25Al: *Ian Baker*[1]; Markus W. Wittmann[1]; James Hanna[1]; Paul R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Engrg., 8000 Cummings Hall, Hanover, NH 03755 USA; [2]University of New South Wales, Elecron Microscope Unit, Sydney, NSW 2052 Australia

An alloy of composition (in atomic percent) Fe-20Ni-25Mn-25Al was cast and its microstructure examined in both the as-cast condition and after anneals for one hour at various temperatures. The as-cast microstructure, which appears to form by spinodal decomposition, consists of alternating, coherent ~50nm wide B2 and b.c.c. plates aligned along <100>. The microstructure is remarkably resistant to coarsening below 1000 K, but precipitation of a f.c.c. phase occurs in the b.c.c. plates at higher temperatures. Mechanical testing revealed a yield strength of 1.3-1.5 MPa from 300-600 K and a strength greater than 200 MPa above 1073K. Room temperature hardness testing was also performed after various anneals. This presentation will attempt to correlate the observed microstructure with the mechanical properties, and will briefly discuss the effects of changes in composition on the microstructure and mechanical properties. Research supported by NIST grant 60NANB200120.

9:55 AM Break

10:10 AM Invited
The Chemical Evolution of Gamma Prime Precipitates in Ni-Al-Cr Base Alloys on a Subnanoscale to Nanoscale: *David N. Seidman*[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; Ronald D. Noebe[2]; [1]Northwestern University, Matls. Sci. & Engrg., Cook Hall, 2220 Campus Dr., Evanston, IL 60208-3108 USA; [2]NASA, MS 49-3, Glenn Rsch. Ctr., 21000 Brookpark Rd., Cleveland, OH 44135 USA

The temporal evolution of the gamma (FCC) and gamma prime (L12 structure)phases in Ni-Al-Cr alloys, with additions of W or Re is followed on a sub- to nanoscale employing three-dimensional atom-probe (3DAP)microscopy. The emphasis in this presentation is on the chemistry within nanoscale gamma prime precipitates. It is demonstrated experimentally that, for example, in a ternary Ni-Al-Cr alloy the compostion of the gamma prime precipitates evolve temporally and they do not follow the compositions given by tie line, whereas in contrast the compostion of the gamma matrix follows the tie line. This is consistent with the Kuehmann-Voorhees model of coarsening in a ternary alloy. Additionally, 3DAP microscope evidence is presented, which shows the existence of concentration gradients within the gamma prime precipitates that evolve toward their equilbrium values. Thereby proving that transient composition profiles exist before the quasi-steady state profiles are achieved.

10:45 AM
Application of the Cluster/Site Approximation to the Calculation of Coherent Interphase Boundary Energy: *W. W. Cao*[1]; J. Zhu[1]; F. Zhang[2]; W. A. Oates[3]; M. D. Asta[4]; Y. A. Chang[1]; [1]University of Wisconsin, Dept. of Matl. Sci. & Engrg., Madison, WI 53706 USA; [2]CompuTherm LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [3]University of Salford, Inst. for Matls. Rsch., Salford M5 4WT UK; [4]Northwestern University, Dept. of Matl. Sci. & Engrg., Evanston, IL 60208 USA

The coherent interphase boundary (IPB) energies are calculated as a function of temperature thermodynamically using the Cluster/Site Approximation (CSA) for binary fcc-based alloy systems. CSA calculated results are compared with those obtained from the tetrahedron-octahedron approximation of the Cluster Variation Method (TO-CVM). We have demonstrated that the CSA offers comparable accuracy as the TO-CVM in calculating the IPB energies. Moreover, the CSA-calculated IPB energies for (Al)/Al_3Li in Al-Li and γ / γ' in Ni-Al are also in accord with experimental data. One advantage of CSA is that it is computational less demanding.

11:10 AM
Differential Role of Nanoscaled Oxide Dispersoids (Y2O3 vs Al2O3) in the High Temperature Structural Stability of NiCr Alloys: *Dheepa Srinivasan*[1]; P. R. Subramanian[2]; Reed R. Corderman[2]; [1]GE India Technology Centre, Matls. Rsch. Lab., EPIP-II, Whitefield Rd., Bangalore, Karnataka 560 066 India; [2]GE Global Research, Ceram. & Metall. Tech., PO Box 8, K1-MB265, Schenectady, NY 12301 USA

Microstructural stability studies were conducted on nanoscale yttria and alumina-reinforced NiCr alloys that were fabricated by electron-beam physical vapor deposition (EB-PVD). The nanoscale yttria dispersoids were far more effective in contributing to the structural stability of the NiCr alloys as compared to the alumina particles. The fine yttria particles share a coherent interface with the NiCr matrix and exert a Zener pinning force on the matrix grains to inhibit rapid grain growth at elevated temperatures. Coarsening of yttria takes place by diffusion of the metallic species through the NiCr matrix, whereas the nano sized alumina particles undergo a hierarchy of phase transformations (gamma-Al2O3 to delta-Al2O3) leading to rapid coarsening. Phase selection at the nanoscale has been examined on the basis of free energies of formation of the metastable vs stable phases at the respective size ranges. The thermal stability of these alloys, as mapped via microstructural examination, has been correlated to room temperature hardness. Further, the different mechanisms responsible for strengthening (Hall-Petch, Orowan and solid solution strengthening) have been examined. The impact due to the most dominant mechanism is brought out for the two types of dispersoids.

11:35 AM
Observations of Reverse Martensitic Transformations in NC Pearlitic Steel: *Yu. Ivanisenko*[1]; I. MacLaren[2]; R. Z. Valiev[3]; H.-J. Fecht[1]; [1]Forschungszentrum Karlsruhe, Inst. für Nanotech., Karlsruhe 76021 Germany; [2]University of Glasgow, Dept. of Physics & Astron., Glasgow G12 8QQ UK; [3]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., Ufa 450000 Russia

Unusual phase transformations in nanocrystalline (NC) materials, such as extension of solid solubility and alteration of phase boundaries, etc. may be related with the shift of thermodynamic equilibrium in a small system due to strong increase of interfacial energy and interfacial stresses. At the same time an important part can play also kinetic factors during preparation of NC structure, such as cooling rate, or high stresses and dislocation density at severe deformation. Here we report about observations of a shear ferrite-austenite transformation taking place at severe deformation of NC pearlitic steel, something which never occurs in conventional deformation of coarse-grained iron and steels. Orientation relationship between parent ferrite and new austenite are either Kurdjumov-Sachs or possibly Nishiyama-Wassermann, i.e. the same as those observed for temperature-driven martensitic transformations in Fe and steels. A mechanism of reverse martensitic transformation is discussed in terms of sliding of transforming partials 1/6<1-10>(110) in bcc lattice.

Shape Casting — The John Campbell Symposium: Solidification

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM Room: 2008
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Srinath Viswanathan, Sandia National Laboratory, Albuquerque, NM 87185-1134 USA

8:30 AM
The Role of the Eutectic in Porosity Formation in Al-Si Foundry Alloys: *Arne K. Dahle*[1]; Stuart D. McDonald[2]; Liming Lu[1]; Kazuhiro Nogita[2]; [1]University of Queensland, CRC for Cast Metals Mfg., Matls. Engrg., Brisbane, Qld 4072 Australia; [2]University of Queensland, Matls. Engrg., Brisbane, Qld 4072 Australia

It is now well-established that three different eutectic solidification patterns can operate in Al-Si foundry alloys. Furthermore, the selection of each solidification mechanism can be manipulated through additions of typical modifier elements, such as Sr and Na, but also less common elements. The spatial distribution of evolving eutectic is very different for each eutectic solidification pattern, therefore resulting in large differences in permeability in the last stages of solidification of the castings. This paper presents a coherent approach to solidification mechanisms in Al-Si alloys and the relationship to the porosity formation in this important alloy system.

8:55 AM
Mechanism of Eutectic Solidification of Aluminum-Silicon Alloys: *Sumanth Shankar*[2]; *Makhlouf Makhlouf*[1]; [1]Metal Processing Institute - Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609 USA; [2]McMaster University, Mechl. Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canda

A mechanism is presented to explain the formation of the eutectic phases in Al-Si hypoeutectic alloys. The mechanism is supported with results of non-equilibrium thermal analyses, and microstructure evidence obtained from optical, scanning and transmission electron microscopy, as well as selected area electron diffraction analyses and elemental x-ray mapping, in addition to results of high temperature rheological measurements performed on Al-Si alloy samples of precisely controlled chemistry.

9:20 AM
Chemical Modification of the Morphology of the Eutectic Phases in Hypoeutectic Aluminum-Silicon Alloys: Sumanth Shankar[1]; *Makhlouf Makhlouf*[2]; [1]McMaster University, Mechl. Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada; [2]Metal Processing Institute - Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609 USA

A mechanism is presented to explain the chemical modification of the morphology of the eutectic phases in Al-Si hypoeutectic alloys. The mechanism is supported with results of non-equilibrium thermal analyses, and microstructure evidence obtained from optical, scanning and transmission electron microscopy, as well as selected area electron diffraction analyses and elemental x-ray mapping, in addition to results of high temperature rheological measurements performed on Al-Si alloy samples of precisely controlled chemistry.

9:45 AM
An Overview of Heterogeneous Nucleation Mechanisms in Al Alloys: *Brian John McKay*[1]; Peter Schumacher[1]; [1]University of Leoben, Lehrstuhl für Gießereikunde, Franz-Josef Str. 18, Leoben, Styria Austria

The benefits of grain refining wrought and foundry Al alloys are widely acknowledged. To induce grain refinement, TiBAl and TiCAl master alloys are commonly added to the melt prior to casting. Understanding the nucleation mechanisms of these refiner particles is important with respect to optimizing current commercial casting practices and improving future refining potency. However, using conventional solidification casting techniques, heterogeneous nucleation is difficult to investigate as α-Al grain growth obscures the nucleation sites. A metallic glass technique has been successfully applied allowing the nucleation mechanisms to be examined using Transmission Electron Microscopy (TEM). The technique freezes the crystalline growth of α-Al at an early stage in a glass and allows the study of the orientation relationship between nucleant and nuclei using diffraction and chemical analysis. The effects of Si and Zr which at higher concentrations can have a detrimental effect on the ultimate grain size (UGS) achieved by the grain refiner addition were examined. It has been found that the chemical composition of the refiner particles and their interfaces play an important role in the nucleation mechanism, which has implications for current casting practices.

10:10 AM Break

10:20 AM
Nucleation of Primary Al_5FeSi in an Al-11.6Si-1.5Fe-0.37Mg-0.29Mn Alloy: *David N. Miller*[1]; Liming Lu[1]; Arne K. Dahle[1]; Graham B. Schaffer[1]; [1]University of Queensland, Div. of Matls. Engrg., Brisbane, Queensland 4072 Australia

Recent research published by Cao and Campbell[1] has provided strong evidence to support the theory that the iron containing 'sludge' phases, Al_5FeSi and $Al_{15}(FeMn)_3Si_2$, nucleate on oxide films entrained in aluminium casting alloys. This is evidenced by the presence of crack-like defects within these iron intermetallics. In an attempt to verify the role of oxide in iron intermetallic formation, experiments have been conducted under conditions of low melt agitation and significant agitation to alter the oxide levels of cast samples with primary Al_5FeSi formation. The results are in general agreement with the theory of Cao and Campbell where the crack-like defects are only evident in Al_5FeSi particles from alloys that are agitated during solidification, ie. conditions that lead to the likely entrainment of oxide films. More detailed electron microscopy studies have been carried out into the nature of the oxide-Al_5FeSi system. [1]Cao, X. and J. Campbell, Metallurgical and Materials Transactions, 2003. 34A(7): p. 1409-1420.

10:45 AM
Observation of Hot Tearing Crack Propagation: Cameron Davidson[2]; *David Michael Viano*[1]; Liming Lu[1]; David StJohn[3]; [1]University of Queensland, Div. of Matls., Sch. of Engrg., St. Lucia, Queensland 4072 Australia; [2]CSIRO Manufacturing & Infrastructure Technology, PO Box 883, Kenmore 4069 Australia; [3]University of Queensland, CRC for Cast Metals Mfg., UDP No. 055, Brisbane, QLD 4072 Australia

Hot tears are generally thought to occur at high solid fractions between 0.95 and 0.99. While the general mechanisms of hot tearing are understood, i.e. the inability of liquid to feed strain imposed on the mushy material, work continues on improving the understanding of the mechanisms at play. A hot tear test rig that measures the temperature and load imposed on the mushy zone during solidification has been successfully used to study hot tearing in Al-Cu alloys. The mould has been modified to incorporate a window above the hot spot region allowing visual observation of hot tear formation and growth. Combining information from visual observation with load and temperature data has led to a better understanding of the mechanisms of hot tearing. It was found that hot tearing started at extremely low loads. The development of load with time was surprisingly insensitive to the presence, or not, of a crack.

11:10 AM
Prediction Hot Tearing Tendency for Multicomponent Aluminum Alloys: *Xinyan Yan*[1]; Jen C. Li[1]; [1]Alcoa, Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Ctr., PA 15069 USA

It is of practical importance to be able to predict the hot tearing tendency for multicomponent aluminum alloys, as hot tearing is one of the most common and serious defects occurred during casting of commercial aluminum alloys that are essentially all multicomponent systems. For many years, the main criterion applied to characterize the hot tearing tendency of an alloy system was based on solidification interval. However, this criterion cannot explain the susceptibility-composition relation between the limits of the pure base metal and the eutectic composition. Clyne and Davies [T. Clyne and G. Davies, Brit. Found., 74(1981), 65] correlated the susceptibility/composition relationship in binary systems based on the concept of the existence of critical time periods during the solidification process when the structure is most vulnerable to cracking. The Scheil equation was used in their model with constant partition coefficient and constant liquidus slope estimated from the phase diagram. In the present study, we followed Clyne and Davies' general idea, and directly coupled the Scheil solidification simulation with phase diagram calculation via PanEngine, a multicomponent phase equilibria calculation interface, and extended the model to higher order systems. Experimental results of hot tearing tests on some ternary and quaternary systems will be presented. The predicted hot tearing tendencies correlated very well with the experimental results of multicomponent aluminum alloys.

11:35 AM
Influence of Temperature and Alloying Elements on Fluidity of Al-Si Alloys: *Marisa Di Sabatino*[1]; Sumanth Shankar[2]; Diran Apelian[3]; Lars Arnberg[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., A.Getz vei, 2B, Trondheim 7491 Norway; [2]McMaster University, Dept. of Mechl. Engrg., Hamilton, Ontario L8S 4L7 Canada; [3]Worcester Polytechnic Institute, Metal Procg. Inst., Worcester, MA 01609 USA

The goal of the work is to study the influence of casting temperature and four alloying elements: Mg, Ti, Fe and Sr, on fluidity of Al-7wt.% Si alloys. Fluidity of the alloys was measured using a fluidity mould produced by N-Tec Ltd., U.K. The experiments were designed using three orthogonal L8 Taguchi matrices. Each of the four alloying elements and the casting temperature was an independent variable with two levels. Three interactions between the variables were identified and analyzed. The two levels of Mg were 0.03wt.% and 0.45 wt.%; Ti levels were 0 and 0.2wt.%; Sr at 0 and 0.023wt.%; and Fe levels were 0.06wt.% and 0.24wt.%. Superheats were 70°C and 130°C over the respective liquidus temperatures of the experimental alloys. The main effect of each of the independent variables on the fluidity was quantified and ANalysis Of VAriance (ANOVA) was performed on the experiment matrix. The results were verified and validated to ensure robustness of the experiment design. In addition to the Taguchi design of experiments, fluidity evaluations were carried out on five melt systems: pure Al, Al-17wt.%Si, 356, 390 and 520. The results of the Taguchi design of experiments show that casting temperature had the most pronounced influence on fluidity of the molten metal. Among the alloying elements chosen, only Mg had an appreciable effect on fluidity. Increasing Mg in the melt from 0 to 0.45wt.% showed a decrease in fluidity of the molten metal. The results of the fluidity experiments on the additional five melt systems show that silicon has a significant effect on fluidity. The variation of fluidity among families of Al-Si alloys is more pronounced than the variation within a particular family of alloy such as 356, which implies that minor changes in composition within a family of Al-Si alloy does not influence fluidity significantly.

Superalloys and Coatings for High Temperature Applications: Oxidation Behaviour - I

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Tuesday AM Room: Nob Hill A/B
February 15, 2005 Location: San Francisco Marriott

Session Chairs: Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; Roger C. Reed, University of British Columbia, Dept. of Metals & Matls. Engrg., Vancouver, BC V6T 1Z4 Canada

8:30 AM Invited
Desk Top TBC Spallation and Interfacial Hydrogen Embrittlement of Alumina Scales: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls., 21000 Brookpark Rd., 106-1, Cleveland, OH 44135 USA

Plasma sprayed 8YSZ coatings were deposited on PWA 1484 single crystal superalloys, without bond coats, and oxidized in 1100°C furnace tests, using 1-hr and 100-hr cycles. Depending on the alloy sulfur content, lives ranged from about 200 to 2000 hr. Often failure did not occur immediately upon cooldown but only after a considerable period at room temperature or upon water immersion. Also, Rene-N5 was oxidized at 1150°C for 1000 1-hr cycles. Scale spallation, before and after water immersion, was monitored by weight change, macrostructure, and acoustic emission. While exhibiting excellent cyclic oxidation resistance, exposure to humidity at room temperature often produced additional interfacial spallation events, occurring in rapid succession or bursts and continuing for hours. Using electrochemical hydrogen charging techniques, cathodic polarization in 1N H2SO4, at -2V and only 1 mA, caused massive scale delamination in a matter of minutes, with minimal N5 dissolution. Conversely, anodic polarization, at +2V and 100 mA, produced no delamination, but only gradual chemical dissolution. A new phenomenon, moisture-induced hydrogen embrittlement of the scale-metal interface, is proposed to explain these results.

9:00 AM Invited
Beneficial Aspects of Pt+Hf-Modified γ'-Ni_3Al+γ-Ni Alloys and Coatings for High-Temperature Oxidation and Corrosion Resistance: *Brian Gleeson*[1]; Daniel J. Sordelet[1]; [1]Iowa State University, Matls. Sci. & Engrg., 2220 Hoover Hall, Ames, IA 50011-2300 USA

Many high-temperature alloys and coatings rely on the formation of a continuous and adherent thermally grown oxide (TGO) scale of α-Al_2O_3 for extended resistance to degradation. For instance, the durability and reliability of thermal barrier coating (TBC) systems in gas turbines are critically linked to the oxidation behavior of the alumina-forming β-NiAl-based bond coat. It has been found that certain unique alloy compositions based on the Ni-Al-Pt-Hf system, yet sufficiently low in aluminum content to be free of β-NiAl, are excellent candidates for the development of novel bond coats for significantly improved TBC reliability and durability. Specifically, it has been found that γ'-Ni_3Al+γ-Ni alloys modified with 10-30 at.% Pt and up to 2 at.% Hf form highly adherent, slow-growing TGO scales during both isothermal and cyclic oxidation at high temperature (maximum temperature studied was 1200°C). Moreover, the thermodynamic activity of aluminum in the alloys is below that in Ni-base superalloys used for areo-engine turbine applications. As a consequence, and in complete contrast to the typically used β-based bond coat compositions, aluminum diffuses from the substrate alloys to the novel γ'+γ coatings. Thus, the novel coating compositions offer the advantages of (1) forming a slow-growing and adherent TGO scale of α-Al_2O_3, (2) not depleting in aluminum due to coating/substrate interdiffusion during service, and (3) being compatible with superalloy substrate in terms of phase constitution and, hence, coefficient of thermal expansion. This paper will review recent findings on the oxidation, hot-corrosion and interdiffusion behaviors of Pt+Hf-modified γ'+γ alloys and coatings.

9:30 AM Invited
Oxidation Behavior of Ni-Al-Hf-(Pt or Pd) Bond Coating Compositions: *Bruce A. Pint*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, MS 6156, Oak Ridge, TN 37831-6156 USA

Interdiffusion problems for conventional Pt-modified aluminide coatings can limit their long-term durability as the Al content in the coating decreases. Recent work has suggested that two-phase aluminide coatings with less than 25% Al may be more compatible with single-crystal superalloys. The oxidation resistance of this class of coatings is critically dependent on a precious metal addition to improve selective oxidation of Al and inhibit Ni-rich oxide formation. The oxidation behavior of model Ni-22.5Al+Hf alloys containing either Pt or Pd are being investigated at 1000°-1200°C in order to better understand the role of composition on oxidation resistance of this class of coatings.

10:00 AM Invited
Cyclic Oxidation Behavior of Ru-Containing Single Crystal Superalloys: *Q. Feng*[1]; B. Tryon[1]; T. K. Nandy[1]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 3062 H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Single crystal superalloys containing Ru as a new alloying addition have recently been of interest since Ru additions may further improve high temperature properties. Cyclic oxidation experiments were conducted on a series of experimental high Ru-containing single crystal superalloys as a function of temperature (900-1200°C) and alloying content. The experimental results indicated that high levels of Ru (3.5-9 at.%) and Cr (8 at.%) additions exhibit good oxidation behavior, equivalent to the second generation single crystal alloys. But high Ru containing alloys without Cr additions displayed poor oxidation resistance. The oxides present and the morphology of oxidized surface were characterized by X-ray diffraction and scanning electron microscopy (SEM). The possible oxidation mechanisms will be discussed.

10:25 AM Break

10:45 AM
Effects of Applied Stresses on Oxidation of Superalloys: *Bryan Randall Barnard*[1]; Peter K. Liaw[1]; Ray A. Buchanan[1]; Peter F. Tortorelli[2]; Bruce A. Pint[2]; Weiju Ren[2]; Dwaine L. Klarstrom[4]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 427-B Dougherty

Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6156 USA; [4]Haynes International, Inc., Tech. Engrg., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The purpose of this research is to examine the effects of applied stresses on the elevated-temperature oxidation behavior of superalloys. Elevated-temperature corrosion is a leading cause of failures among high-temperature structural components. Superalloys, particularly those that contain large amounts of chromium, are often the chosen materials for these components. This is because chromium forms oxide scales that protect the alloy from further damage due to elevated-temperature corrosion. These oxide scales have been studied extensively. However, the effects that applied stresses have on oxide layers have not been examined as thoroughly, and, thus, are the subject of this research. Preliminary results comparing the isothermal oxidation behavior of Haynes 75® and Haynes 230® alloys with and without an applied (creep) load will be presented for temperatures of 700 and 800°C. A model for predicting the oxidation behavior under various conditions of load and temperature will be developed, based upon the results of testing.

11:10 AM Invited
Oxidation and Partitioning Characteristics of Advanced Ni-Base Superalloys with Ru and Pt: L. Zhang[1]; *S. Tin*[1]; M. K. Miller[2]; S. S. Babu[2]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge, Cambridgeshire CB2 3QZ UK; [2]Oak Ridge National Laboratory, Microscopy, Microanalysis, Microstruct. Grp., Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, Oak Ridge, TN 37831 USA

The structural properties and temperature capabilities of advanced Ni-base single crystal superalloys can potentially be extended by utilizing Pt-group metal additions. Recent investigations have shown single crystal Ni-base superalloys containing additions of both Pt and Ru exhibit unusually good high temperature oxidation characteristics. Partitioning characteristics of these elements within the constituent phases of the microstructure have been investigated using atom probe and high resolution transmission electron microscopy. Results of cyclic oxidation tests on bare and aluminized specimens are presented. Environmental implications of incorporating Pt-group elements in Ni-base superalloys will be discussed.

11:35 AM
Oxidation Characteristics of the Third Generation Superalloy CMSX-10: *Ainul Akhtar*[1]; Roger C. Reed[1]; [1]University of British Columbia, Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The oxidation resistance of the single crystal superalloys is an important consideration, since it is not always the case that coatings can be employed, for reasons of practicality and cost. Here, the oxidation of bare CMSX-10 is studied. Since the Cr content is relatively low, it is found that the oxidation behaviour differs from earlier alloys in some important respects. Although an external scale of NiO containing spinels is formed, transformation into the beta phase preceeds the creation of an internal oxidation zone which contains (Ni,Co)Ta2O6 and (Ni,Co)WO4. We have found no evidence of alumina formation. The kinetics of oxidation are interesting: oxidation at higher temperatures can be slower than at lower temperatures, and it is postulated that this is due to the formation of the aluminide phase.

12:00 PM
Yttrium Doping for Oxidation Resistance of 4th Generation Ni-Base Superalloys: *Atsushi Sato*[1]; Kyoko Kawagishi[2]; Kenji Nishida[3]; Toshiharu Kobayashi[2]; Hiroshi Harada[2]; Hachiro Imai[1]; [1]Shibaura Institute of Technology, Dept. of Matls. Sci. & Engrg., 3-9-14, Shibaura, Minato-ku, Tokyo 108-0023 Japan; [2]National Institute for Materials Science, High Temp. Matls. Grp., 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047 Japan; [3]National Institute for Materials Science, Surface Analy. Grp., 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047 Japan

The 4th generation Ni-base single crystal (SC) superalloys with platinum group metals, e.g., Ru, have superior creep strengths and recessive oxidation resistance. In this study, yttrium doping was applied to a 4th generation Ni-base SC superalloy, TMS-138, to improve the oxidation resistance. TMS-138 base samples with yttrium contents ranging from 0 to 1000 wppm were examined by a 1100°C cyclic oxidation test. The weight change was found to be smallest with a sample containing 100 wppm yttrium. Microstructure observation with this sample showed that yttrium sulfide particles were dispersed in the gamma/gamma prime matrix, whereas other additional yttrium compounds were observed in samples with higher yttrium contents. Thus, it was considered that the right amount of yttrium capable enough of removing sulfur from the matrix to form sulfides improved the adhesiveness of oxide scales to the matrix and consequently, the oxidation resistance.

Surface Engineering in Materials Science - III: Nanocoatings

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Tuesday AM Room: 2022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. & Mechl., Oviedo, FL 32765-7962 USA; John J. Moore, Colorado School of Mines, Dept. of Metall. & Matls. Engrg., Golden, CO 80401 USA

8:30 AM Invited
Nanocomposite Coatings: Structure and Properties: *Jeff T. De Hosson*[1]; [1]University of Groningen, Appl. Physics, Nijenborgh 4, Groningen 9747 AG The Netherlands

This contribution deals with fundamental and applied concepts in nano-structured coatings, in particular focusing on the characterization with high-resolution (transmission) electron microscopy. Both balanced and unbalanced magnetron-sputtering systems were used to deposit nc-TiC/a-C nanocomposite coatings with hydrogen-free DLC matrix. The contents of Ti and C in the coatings were varied in the whole range of interest (5~45 at.%Ti) by changing the configuration of the targets. The size and distribution of the nanoparticles were determined with High-Resolution (HR) and Energy-Filtered (EF) Transmission Electron Microscopy (TEM); their size varies between 2 and 20 nm diameter and particle clustering was observed. Electron microscopy was further employed to determine the chemistry of the a-C matrix (sp3/sp2 ratio), through Electron Energy Loss Spectroscopy (EELS). It was demonstrated that both TiC particle size and a-C chemistry could be directly controlled by change of deposition parameters such as applied bias and deposition pressure.

8:55 AM
Interfacial Phenomena in Carbon Nanotube Reinforced Al-Based Composite Coatings Deposited by Air Plasma Spray and High Velocity Oxy-Fuel Techniques: *Arvind Agarwal*[1]; *Tapas Laha*[1]; [1]Florida International University, Mechl. & Matls. Engrg., 10555 W. Flagler St., EC 3464, Miami, FL 33174 USA

Employment of carbon nanotubes (CNT) as reinforcement in metal matrix composite coatings potentially improves the fracture toughness, wear resistance and hardness. In the present effort, CNT reinforced Al-based composite coatings have been deposited by atmospheric plasma spray and high velocity oxy-fuel spray deposition techniques. The distribution of CNT reinforcement in the composite coatings has been investigated by scanning electron microscopy, X-ray diffraction, Raman spectroscopy and transmission electron microscopy. The characterization confirms presence of physically and chemically stable carbon nanotubes in both the composite coatings. Besides, TEM investigation has been carried out to understand the interfacial phenomena. The effect of high temperature and rapid solidification processing on the interfacial reactions, as well as on the wettability of aluminum on carbon nanotubes has been studied. Appreciable wetting of CNT by molten aluminum has been noticed in the composites.

9:10 AM
Role of Surface Treatment of Carbon Fibers on Properties: *G. V. Prabhu Gaunkar*[1]; S. P. Sharma[1]; S. C. Lakkad[1]; [1]IIT Bombay, Dept. of Metallurgl. Engrg. & Matls. Sci., Powai, Mumbai 400076 India

A process of modifying surface characteristics of fibers by incorporating nano and micro constituents has been developed. Carbon fibers of different strength levels and subjected to surface modification treatments have been used to make polymer matrix composites. Role of

surface modification of fibers on mechanical properties. Damage tolerance under impact loading has been studied using ultrasonic C scan technique and scanning electron fractography. Surface modification treatments are found to give significant improvements in mechanical properties and well as daamage tolerance under impact loading.

9:25 AM
Nano-Coatings on Carbon Structures for Interfacial Modification: *Rajasekhar Venkata Pulikollu*[1]; Pratik Joshi[1]; Sharmila Mitra Mukhopadhyay[1]; [1]Wright State University, Dept. of Mechl. & Matls. Engrg., 209 Russ Engrg. Ctr., 3640 Col. Glenn Hwy., Dayton, OH 45435-0001 USA

Microwave plasma has been used to deposit 1-5 nanometer thin films on surfaces of carbon foam and nano-fibers, and the resulting changes in interfacial properties are investigated. In this presentation, interfacial issues related to carbon-polymer, and carbon-metal composites will be discussed. Two types of coatings: One for enhancing the surface reactivity and another for surface inertness has been compared with uncoated carbon structures. It is observed that the stress-strain behavior, as well as fracture paths of foam-epoxy and nanofiber-epoxy composites is significantly altered by the nano-coatings. Additionally, the influence of these films on the microstructure and growth mechanism of metallic (copper) thin films on graphite has been investigated, and will be presented. The significance of these nano-coatings to formation of metal-matrix and polymer-matrix composites will be discussed.

9:40 AM Break

10:10 AM Invited
Nanostructured Coatings: Properties and Fundamentals: *Enrique J. Lavernia*[1]; [1]University of California, Coll. of Engrg., John D. Kemper Hall of Engrg., One Shields Ave., Davis, CA 95616 USA

The application of nanocrystalline materials used as powder feedstock for thermal spraying in recent years has been mainly facilitated by the wide range of powder sources available, including: vapor condensation, solution precipitation, combustion synthesis, sol-gel processing, thermochemical synthesis, and mechanical alloying/milling. The resultant thermal sprayed coatings have been shown to exhibit unique and often enhanced physical and mechanical performance properties in comparison to the coatings produced by current technology. Improvements in physical have been documented for several metallic and cermet based nanostructured coatings. However, the behavior of a nanostructured material during thermal spraying is rendered complex by factors such as morphology of feedstock powders; thermal stability of nanostructured powders; and thermal and momentum behavior of nanostructured powder. Optimization of chemistry, morphology and coating thickness, for example, should lead to the attainment of physical performance heretofore unattainable with conventional coatings. The present paper is to provide an overview of recent advancements in the field of high performance nanostructured coatings, paying particular attention to underlying fundamental issues. Examples of several metallic and cermet coatings and bulk samples will be used to demonstrate the influence of the morphology of nanostructured powders on performance.

10:35 AM
The Effect of the Surface Nanocrystallization-and-Hardening (SNH) Process on the Fatigue Behavior of a 316 Stainless Steel: *J. W. Tian*[1]; W. Yuan[1]; G. Y. Wang[1]; D. E. Fielden[1]; J. Jeon[1]; H. Choo[1]; J. C. Villegas[2]; L. L. Shaw[2]; P. K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-0001 USA; [2]University of Connecticut, Metall. & Matls. Engrg., Storrs, CT 06269 USA

Using the surface-nanocrystallization-and-hardening (SNH) process, a nano-structured layer was found on the surface of the 316 stainless steel disk, while the coarse grain below the surface remained unchanged. A grain-size gradient from the surface nano-layer to the interior coarse grain, a micro-hardness gradient, and a residual compressive to tension stress distribution were introduced through the cross section of the samples. Microstructural features of the cross section near and below the surface were investigated by means of the X-Ray diffraction (XRD), scanning-electron microscope (SEM), and transmission-electron microscope (TEM). The microhardness of the cross section was measured by the Vicker's-hardness tester and nanoindentation, and the residual stresses distributions were determined by XRD. Four-point-bend-fatigue experiments were employed to study the fatigue behavior of the specimens. The results show that the nano-structured layer on the surface has high resistance to the fatigue-crack initiation, and the interior coarse grain can retard the growth of fatigue cracks, both of which evidently improved the fatigue lives of the specimens. However, with the further increase of the processing time, fatigue properties become worse because of the surface roughness and/or surface damage. The present work is supported by the National Science Foundation under DMR-0207729 with Dr. K.L. Murty and Dr. J. Akkara as the contract monitors.

10:50 AM
Nano-Engineered Multiwall Carbon Nanotube-Copper Composite Thermal Interface Material for Efficient Heat Conduction: *Quoc Xuan Ngo*[1]; Brett A. Cruden[2]; Alan M. Cassell[2]; Gerard Sims[2]; Jun Li[2]; M. Meyyappan[2]; Cary Y. Yang[1]; [1]Santa Clara University, Ctr. for Nanostruct., 500 El Camino Real, Santa Clara, CA 95050 USA; [2]NASA Ames Research Center, Ctr. for Nanotech., Moffett Field, CA 94035 USA

Efforts in integrated circuit (IC) packaging technologies have recently been focused on management of increasing heat density associated with high frequency and high density circuit designs. While current flip-chip package designs can accommodate relatively high amounts of heat density, new materials need to be developed to manage thermal effects of next-generation integrated circuits. Multiwall carbon nanotubes (MWNT) have been shown to significantly enhance thermal conduction in the axial direction and thus can be considered to be a candidate for future thermal interface materials by facilitating efficient thermal transport. This work focuses on fabrication and characterization of a robust MWNT-copper composite material as an element in IC package designs. We show that using vertically aligned MWNT arrays reduces interfacial thermal resistance by increasing conduction surface area, and furthermore, the embedded copper acts as a lateral heat spreader to efficiently disperse heat, a necessary function for packaging materials. In addition, we demonstrate reusability of the material, and the absence of residue on the contacting material, both novel features of the MWNT-copper composite that are not found in most state-of-the-art thermal interface materials. Electrochemical methods such as metal deposition and etch are discussed for the creation of the MWNT-Cu composite, detailing issues and observations with using such methods. We show that precise engineering of the composite surface affects the ability of this material to act as an efficient thermal interface material. A thermal contact resistance measurement has been designed to obtain a value of thermal contact resistance for a variety of different thermal contact materials.

11:05 AM
The Effect of Gas Pressure on the Structure and Tribological Performances of Nanometer Diamond-Like Carbon Films Prepared by Plasma-Based Ion Implantation: Liao Jiaxuan[1]; *Liu Weimin*[2]; Xu Tao[2]; Yang Chuanren[1]; Chen Hongwei[1]; Fu Chunlin[1]; *Leng Wenjian*[1]; [1]University of Electronic Science and Technology of China, Sch. of Microelect. & Solid-State Elect., Chengdu 610054 China; [2]Chinese Academy of Sciences, State Key Lab. of Solid Lubrication, Lanzhou Inst. of Cheml. Physics, Lanzhou 730000 China

Nanometer diamond-like carbon (DLC) films were prepared on Si wafers by plasma-based ion implantation at 18kV for 30min. The effect of gas pressure ranging from 0.5Pa to 2.0Pa on the DLC films has been investigated. Dry sliding tribological experiments against alumina balls have also been carried out on a ball-on-disc tester. The results show that the films range from 30nm to 70nm in thickness, and are firmly adhered to Si substrate owing to a C-implanted layer. A critical gas pressure of 0.5Pa corresponds to no films but a C-implanted layer. The DLC films exhibit an average roughness of less than 0.50nm, a content of sp3 bonds of more than 50% and an improvement in tribological performances. Meanwhile, an increasing gas pressure corresponds to an increasing film thickness, a decreasing content of sp3 bonds and an improvement in tribological performances.

Texture and Microstructure in Thin Films and Coatings: Coatings

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Tuesday AM Room: 3010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Bae-Kyun Kim, McGill University, Mining, Montreal, Quebec H3A 2B3 Canada

8:30 AM
Texture and Microstructure in Oxide Films Formed During Oxidation of Pure Iron: *Bae-Kyun Kim*[1]; *Jerzy A. Szpunar*[1]; [1]McGill University, Mining, Metals & Matls., 3600 Univ. St., Montreal, Quebec H3A 2B3 Canada

In order to improve the quality of steel surface, it is important to understand descaling process that take place in hot rolling. For better understanding of the scale removal, iron oxide texture, microstructure and structure of interphones should be characterized. Electron backscattered diffraction (EBSD) become an important tool for studying local texture and microstructure of materials. The orientation imaging microscopy (OIM) based on the EBSD technique can be used to examine transformation of texture and microstructure during high temperature oxidation. OIM can also be used to study oxide defects, which may affect the cracking of the oxide scale. In order to simulate industrial hot rolling, high temperature oxidation tests of pure iron were performed in the tube furnace up to 950°C with continuous heating under atmospheric pressure. The iron oxide microstructure can be described using OIM maps representing the image quality (IQ) and the inverse pole figure (IPF) of the cross-sectional area of oxidized iron. The three different iron oxides phases, namely the wüstite (FeO), magnetite (Fe3O4), and hematite (Fe2O3), are distinguished and the characteristics of oxides developed during different oxidation treatment are compared. Additionally, interfacial structure between different iron oxide layers is analyzed in relation to nucleation of microcracks and mechanism of oxide removal from the substrate.

8:50 AM
The Effects of Substrate Bias, Substrate Temperature and Pulse Frequency to the Microstructures of CrN Coatings Deposited by Pulsed DC Magnetron Sputtering: *Jyh-Wei Lee*[1]; Shih-Kang Tien[2]; Chih-Hsiung Lin[2]; Jenq-Gong Duh[2]; [1]Tung Nan Institute of Technology, Dept. Mechl. Engrg., #152, Sec.3 Pei-Shen Rd., Shen-Ken, Taipei County 222 Taiwan; [2]National Tsing Hua University, Dept. Matls. Sci. & Engrg., #101, Sec.2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

The chromium nitride coatings have been deposited by the bipolar symmetric pulsed DC magnetron reactive sputtering process at different pulse frequency, substrate bias power and substrate temperature. The substrate bias was applied with constant pulse frequency of 50 KHz. The surface and cross sectional morphologies of the coatings were analyzed by the field emission scanning electron microscopy. Phase analysis of the coatings was performed by the XRD. For the CrN coatings deposited with pulse frequency of 2 and 20 KHz, the preferred orientation of coatings changed from (111) to (200) as substrate temperature and bias increased. The deposition rate of the CrN films decreased with increasing pulse frequency, whereas the substrate temperature and bias showed almost no influence. It was concluded that the pulse frequency, substrate bias and temperature played important roles to the texture, microstructure and surface roughness of the CrN coatings deposited by the pulsed DC magnetron sputtering.

9:10 AM
Simulation of Texture Development in Oxide Films: *Hualong Li*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Mining, Metals & Matls., 3600 Univ. St., Montreal, Quebec H3A 2B2 Canada

A methodology that can be used to simulate the texture formation in oxide formed on metal substrate is proposed. Graphically enhanced discrete computer simulation incorporates Orientation Imaging Microscopy (OIM) experimental data to characterize the texture and microstructure of the substrate that used as an input to simulation. It also allows user to generate input data based on X-ray measurements of texture. The abundant information contained in OIM measurement allows the computer model to incorporate many structural characteristics of polycrystalline materials such as, texture, grain boundary character, grain shape and size, phase composition, chemical composition, stored elastic energy, and residual stress. The input data to the simulation characterize the interfacial energy between the oxide and substrate, state of the stress in oxide film and oxide diffusivity in the bulk and also the grain boundary diffusion coefficients. These data can be obtained from the experiments or from the first principle calculations. Graphical representation allows the user to monitor each simulation step of the structural transformation of oxide film and to perform virtual experiments. In this paper, we will present the results of simulation of oxide layer in Zr and Ni alloys.

9:30 AM
Significant Improvements in Ni-B Coatings for Metal Alloys: *Yancy W. Riddle*[1]; Ed McComas[1]; Wynn Atterbury[2]; [1]Universal Chemical Technologies, Inc., 7825 SW Ellipse Way, Stuart, FL 4997 USA; [2]UCT Defense, 7825 SW Ellipse Way, Stuart, FL 34997 USA

Significant improvements to the properties associated with early generations of Ni-B coatings for metal alloys have been made. When compared to other industrial metal coatings such as hard chrome new Ni-B coatings can outperform. Unfortunately many engineers and scientists refer to the properties of early generation Ni-B coatings during investigative or comparative studies. This paper serves as a technical and scientific update of Ni-B coatings based on an extensive research program. These new Ni-B coatings are of major industrial importance due to improved properties of; hardness, lubricity, ductility, coefficient of friction, wear resistance, corrosion resistance, environmental friendliness, safety, processing, thermal compatibility, and more. A wide variety of carbon and stainless steels, aluminum alloys, copper alloys, titanium alloys, and other metal alloys can be successfully coated by these new Ni-B coatings. Included with this paper are comparative performance data between old and new generation Ni-B coatings reinforced by microstructural evidence and examples of successful application in industry.

9:50 AM
Crystallographic Texture of EB-PVD TBCs on Stationary Flat Surfaces in a Multiple Ingot Chamber: Jeremy Bernier[1]; G. Levan[2]; Sudha Bose[2]; Md. Maniruzzaman[1]; *Richard D. Sisson*[1]; [1]Worcester Polytechnic Institute, Mfg. & Matls. Engrg., Mechl. Engrg. Dept., 100 Inst. Rd., Worcester, MA 01602 USA; [2]Pratt & Whitney, Matls. & Process Engrg., E. Hartford, CT USA

The crystallographic texture of EB-PVD TBCs (7 wt% Y2O3) deposited in a two ingot chamber has been experimentally determined by comparing pole figure data with measured column growth angles. It was found that the coating deposited on a flat surface directly above an ingot exhibited <220> texture. Coatings deposited between the ingots and off of the centerline exhibited <311> texture>. Coating deposited at the far corners of coating chamber revealed a <110> fiber texture or <311> single crystal type texture. The microstructures of specimen cross sections reveal that column growth angle and vapor incidence angles are in reasonable agreement and that the columns grow towards the closest ingot. These results are discussed in terms of substrate temperature and vapor incidence angles.

10:10 AM Break

10:40 AM
The Metallurgy of High-Strength and Thermally Stable Nanostructured Ni-Mn Alloys: *Albert Alec Talin*[1]; [1]Sandia National Laboratories, 7011 East Ave., Livermore, CA 94550 USA

Electroplated NiMn alloy is a promising material for applications requiring high strength, thermal stability and low residual stress. In this paper we report on the variation of crystallite diameter, crystallographic texture, and yield strength of NiMn electrodeposits as a function of current density and Mn content. Over the concentration range of 0 wt. % to 1 wt. %, Mn reduces the average crystallite size from hundreds of nanometer to ~20 nm, dramatically alters the texture dependence on current density, and increases the yield strength from ~ 300 MPa to over 1 GPa. We also show how small additions of Mn solute affects the recrystallization temperature and texture of Ni, and how these results can be understood in terms of the spatial distribution of Mn in Ni, obtained using three dimensional atom probe technique.

11:00 AM
Texture and Corrosion Resistance of Electrodeposited Tin Coatings: *Shixue Wen*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Due to its non-toxicity, good corrosion resistance and excellent solderability, tin is widely used in food packaging and electronic industries. Tinplate, mild steel coated with tin, is a material of great economic importance. Tin electrodeposition on mild steel substrates using stannous sulfate and sulfuric acid was studied under different current densities, temperature and organic additives. The macrotexture of the tin deposits was measured using an x-ray texture goniometer. The orientation imaging microscopy (OIM) was used to measure the orientations and sizes of individual grains. The corrosion resistance of electrodeposited tin coatings was measured using a standard corrosion cell kit according to the ASTM standard G3. It was found that tin coatings with three different texture, (110), (100) and (301) fiber texture can be produced by electrodeposition. At a temperature 20ºC and a current density of 50A/m2, a fibre (110) was obtained. As the current density increased to 100 and 200 A/m2, the texture of tin coatings was changed to (100) fibre. With a different organic additive, tin coatings with (100) fibre and (301) fibre textures were obtained at the current densities of 200A/m2 and 50A/m2 respectively at 20ºC. An increase in current density leads to a decrease in grain sizes. At the same current density, the grain sizes of tin coatings increase with increased temperature. The influence of temperature (20, 40, 60 and 80ºC) on texture is relatively negligible. The corrosion resistance of tin coatings increases with a decrease in grain size. The corrosion resistance of tin coatings with (301) fibre was 1.5 times higher than that of the tin coating with (100) fibre texture and the corrosion current density was 0.2092 µA/cm2 for the former sample and 0.3011µA/cm2 for the latter sample. The results suggest that texture and microstructure (grain size) play an important role in controlling corrosion rate of tin based coatings. It also can be concluded that texture's influence on corrosion resistance is much higher than that of grain size.

11:20 AM
Microstructure and Mechanical Properties Evaluation of Chromium Nitride/Tungsten Nitride Superlattice Coatings: Fan-Bean Wu[1]; *Jyh-Wei Lee*[2]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Matls. Sci. Ctr., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan; [2]Tung Nan Institute of Technology, Dept. Mechl. Engrg., #152, Sec.3 Pei-Shen Rd., Shen-Ken, Taipei County 222 Taiwan

Chromium nitride and tungsten nitride superlattice coatings have been fabricated by a dual-gun rf magnetron reactive sputtering system. The superlattice period of the coatings varies in the range 6 to 24 nm. The microstructures of the CrN/WNx coatings were evaluated by the field emission scanning electron microscopy and transmission electron microscopy. Elemental distributions of the coatings were revealed by the Auger electron spectroscopy depth profiling analysis. Nanoindentation technique was employed to evaluate the mechanical properties including hardness and Young's modulus. The phase identification indicated that the coatings were composed of the CrN and W2N phases. The CrN/WNx superlattice coatings exhibited a high hardness over 30 GPa, which was superior to that of the single CrN coating. The nanolayered structure that confined the grains of the nitrides in the nano range was beneficial to the enhancement of the mechanical performance of the multilayer coating.

11:40 AM
Effect of Process Current Density and Temperature on Electrochemical Boriding of Steel in Molten Salts: *Guldem Kartal*[1]; Servet Timur[1]; [1]Istanbul Technical University, Metall. & Matls. Sci. Engrg., ITU Chmst-Metall. Faculty, Maslak, Istanbul 34469 Turkey

In this study, the boriding of steels by molten salt electrolysis in borax-base electrolyte at various current densities (50-700mA/cm2), was investigated. The influence of the electrolysis parameters (current density) on thickness and morphology are present. Different borided phases can form depending on the amount of the diffused boron from surface to matrix as follows: FeBx(x>1)?¨FeB?¨Fe2B?¨Fe3B?¨FeyB(y>3)?¨Fe. Determination of optimum current density for the boriding of DIN EN 10130-99 DC04 low carbon steel are 1 hour and at constant bath temperature (900°C), and % 10 NaCl + % 90 Na2B4O7 bath composition.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Tuesday AM — Room: 3003
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: David E. Laughlin, Carnegie Mellon University, MSE, Pittsburgh, PA 15213 USA; Peter W. Voorhees, Northwestern University, MSE, Evanston, IL 60208 USA

8:30 AM Opening Remarks

8:35 AM Invited
Elastic Stability and the Limits of Strength: *John William Morris*[1]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining, Berkeley, CA 94720 USA

The upper limit of strength (the "theoretical strength") has been an active subject of research and speculation for the better part of a century. The subject has recently become important, for two reasons. (1) Given recent advances in ab initio techniques and computing machines, the limits of strength can be calculated with considerable accuracy, making this one of the very few problems in mechanical behavior that can actually be solved. (2) Given recent advances, the limits of strength are being approached in some systems, such as hardened or defect-free films, and their relevance is becoming recognized in others. The present paper discusses results from recent research on the limits of strength. Topics include: criteria for elastic stability, the inherent nature of {100} cleavage in bcc metals, the source of strength in steel, resistance to cleavage in fcc metals, the difference between "hard" and "soft" carbonitrides, and the possibility of measuring ideal strength with nanoindentation.

9:00 AM Invited
Linking Phase Field Method to Ab Initio Calculations: Modeling Dislocation - Precipitate Interactions in Real Alloys: Chen Shen[1]; *Y. Wang*[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

In an effort to understand effects of microstructural characteristics on deformation mechanisms observed in Ni-based superalloys, we incorporate directly information from ab initio calculations into phase field model of dislocations. This is accomplished by formulating appropriate invariant forms of the crystalline energy, which relates explicitly to the generalized stacking fault (GSF) energy. The use of ab initio GSF energy in the phase field model allows for quantitative studies of core structures of both stationary and moving dislocations in individual phases as well as at interphase interfaces, and the effects of size and spatial distribution of gamma prime particles on deformation mechanisms. The work is supported by the National Science Foundation.

9:25 AM
Phase-Field Modeling of ä?-Precipitation at Dislocations in Al-Li Alloys: *Y. H. Wen*[1]; A. W. Zhu[2]; G. J. Shiflet[2]; [1]UES Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Virginia, Dept. Matls. Sci. Engrg., Charlottesville, VA 22904 USA

A phase-field model is formulated to study the precipitation process at the presence of various dislocations. The evolution of the particular morphology of ä? precipitates are demonstrated to be associated with interaction between the stress fields around edge dislocations and the particle in Al-Li alloys. The results are compared with TEM observation where the ä? heterogeneous precipitation occurs through isothermal aging with small undercoolings. Work completed under Air Force Contract # F33615-01-C-5214.

9:40 AM
Dislocation-Induced Crossover Scaling During Spinodal Decomposition: *Mikko P. Haataja*[1]; Jennifer Mahon[2]; *Nikolas Provatas*[2];

Francois Leonard[3]; [1]Princeton University, Mechl. & Aeros. Engrg., Olden St., Princeton, NJ 08544 USA; [2]McMaster University, Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [3]Sandia National Laboratories, Livermore, CA 94551 USA

In this talk we address the role of mobile dislocations on the phase separation process of a binary alloy. We employ a phase-field model which explicitly incorporates the dynamics of the composition and dislocation fields in two spatial dimensions, coupled through their elastic fields. We show both analytically and numerically that the effects of mobile dislocations on domain coarsening kinetics can be expressed via a crossover scaling function. We also discuss experimental ramifications of such a crossover scaling function.

9:55 AM
The Influence of Interstitial Oxygen and Peak Pressure on the Shock Loading Behavior of Zirconium: *E. K. Cerreta*[1]; G. T. Gray[1]; R. S. Hixson[2]; R. A. Rigg[2]; D. W. Brown[1]; B. L. Henrie[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, DX-2, Los Alamos, NM 87545

The pressure of the a-w phase transition in zirconium has been quantified as a function of interstitial content. The pressure increases with increasing interstitial oxygen content and for the high purity (HP) material occurs at 7.1GPa. Increasing the interstitial oxygen content increases the number of octahedral sites occupied; this is postulated to increase the pressure for the phase transformation. Deformation behavior and substructural evolution of as-annealed HP zirconium under quasi-static conditions has been compared to its response following shock prestraining at 5.8 and 8 GPa. The reload stress-strain response of HP zirconium shock prestrained to 8GPa was found to exhibit enhanced hardening when compared to its quasi-static constitutive behavior. The reload yield behavior of zirconium specimens shocked to 5.8 GPa did not exhibit enhanced shock hardening.

10:10 AM Break

10:35 AM Invited
Modeling of Cyclical Phase Transformations in Mechanical Alloying: *Jong K. Lee*[1]; [1]Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI 49931 USA

As a driven process, mechanical alloying synthesizes a mixture of equilibrium and non-equilibrium phases at a different temporal sequence. In many instances, the relative phase fractions are apparently invariant after long milling times. The situation is termed dynamic equilibrium. Some recent works, however, show evidence of cyclical phase transformations taking place during mechanical alloying. Cyclical phase transformations resemble dynamic equilibrium in the sense that two phases are simultaneously present, but the phase fractions vary. A brief thermodynamic and kinetic account is first discussed to establish the criteria for cyclical transformations. A two-dimensional molecular dynamic work is then followed to demonstrate cyclical phase transitions between an equilibrium and a non-equilibrium phase during mechanical alloying. Model binary nanocrystals, made of Lennard-Jones atoms, are studied to display cyclical transitions between an equilibrium rhombus and a non-equilibrium square phase under periodic, small-shear loading conditions.

11:00 AM Invited
Elastic Inhomogeneities and Microstructural Evolution: *Perry H. Leo*[1]; [1]University of Minnesota, Aeros. Engrg., 107 Akerman Hall, 110 Union St. SE, Minneapolis, MN 55408 USA

The importance of elastic effects on microstructural evolution during diffusional phase transformations has been well documented in the past ten years. In particular, Professor Khachaturyan and his coworkers have made remarkable advances in the theory and simulation of elastic effects on particle behavior and multiparticle interactions. In this talk, we present some work extending these ideas to show how elastic inhomogeneity in cubic materials affects the elastic interactions. We go beyond the ideas of 'soft' and 'hard' precipitates to consider cases where some elastic constants may be higher in the precipitate than the matrix, while other constants are lower. We document the specific role of the elastic constants on particle interactions and on certain behavior of individual particles, such as particle splitting.

11:25 AM Invited
Spontaneous Patterning of the Chemical Order Field in L12 and L10 Ordered Alloys Subjected to Energetic Irradiation: *Pascal M. Bellon*[1]; Jia Ye[1]; [1]University of Illinois, Matls. Sci. & Engrg., 1304 W. Green St., Urbana, IL 61801 USA

Dense displacement cascades produced by irradiation with energetic particles lead to the formation of disordered zones in chemically ordered alloys. At temperatures below the order-disorder transition, these disordered zones, whose sizes range from a few to several nanometers, are thermally annealed out. Under sustained irradiation, the competition between these two dynamics drives the system into various steady states. Kinetic Monte Carlo simulations and analytical modeling are employed to identify these steady states in binary alloys that form L12 and L10 ordered phases at equilibrium. Besides the expected long range ordered and disordered steady states, a new state is discovered, where the microstructure is comprised of well ordered domains of finite size. A two-stage reordering of the disordered zones is at the origin of the dynamical stabilization of these patterns. The present results indicate that ion-beam processing could be used to synthesize ordered nanocomposites with tunable sizes.

11:50 AM
Evolution of Deformation and Recrystallization Textures in Cold-Rolled CP-Ti - Experiments and MC Simulation: Y. B. Chun[1]; S. L. Semiatin[2]; *S. K. Hwang*[1]; [1]Inha University, Sch. of Matls. Sci. & Engrg., 253 Yong Hyun-Dong, Nam-Gu, Incheon 402-751 S. Korea; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The development of deformation texture in CP-Ti during cold rolling (10%~90%) and the evolution of the recrystallization texture during subsequent heat treatment were determined experimentally using XRD and EBSD and modeled using Monte-Carlo computer simulations. For a low- to-medium level of deformation (up to 40%), twinning governed the deformation and gave rise to a normal basal texture. For higher levels of deformation (up to 90%), however, the main deformation mechanism was slip, and a bi-modal basal texture with peaks at j1=0', F=35', j2=30' was developed. Recrystallization annealing suppressed the cold-rolling texture and introduced instead two new texture components (j1=15', F=35', j2=35' and j1=0', F=35', j2=0'), whose intensities significantly increased during the grain-coarsening stage of recrystallization and grain growth. From the EBSD analysis, it was found that grains with the major recrystallization-texture components had considerably larger grain sizes than others. A two-dimensional Monte-Carlo simulation was conducted to trace the evolution process of the major recrystallization-texture components. The fact that the intensification of the recrystallization texture occurred during the grain-coarsening stage, found by EBSD-mapping method, was also confirmed by the simulation. The present results suggest that the heterogeneous grain-size distribution during the recrystallization process was responsible for the evolution characteristics of the recrystallization texture in CP-Ti.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Grain Boundary Properties and Severe Plastic Deformation

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Tuesday AM Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: T. G. Nieh, University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA; Sergey Dobatkin, Russian Academy of Sciences, A.A. Baikov Inst. of Metall. & Matls. Sci., Moscow 119991 Russia; Sean R. Agnew, University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; Zenji Horita, Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan

8:30 AM
Fatigue Behavior of Ultra-Fine Grain Copper: Kai Zhang[1]; Adam Chenoweth[1]; Giancarlo Izzi[1]; Abhishek Lahoti[1]; *Julia R. Weertman*[1]; [1]Northwestern University, Matls. Sci. & Engrg. Dept., 2220 Campus Dr., Evanston, IL 60208 USA

Fatigue experiments have been carried out on ultra-fine grain copper prepared by cryogenic rolling. Samples are tested in the pull-pull mode. Results in the form of S-N curves will be presented, along with characterization studies of changes in the surface features and internal structures caused by the cyclic deformation. This research is supported by US DOE grant DE-FG02-02ER 46002.

8:45 AM

Microstructures and Mechanical Properties After Processing by High-Pressure Torsion: *Zenji Horita*[1]; Genki Sakai[1]; Kaoru Kishikawa[1]; Yoshimi Hisatsune[1]; Terence G. Langdon[2]; [1]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

In this study, severe plastic strain was imposed through the process of high-pressure torsion (HPT). An Al-3%Mg-0.2%Sc alloy and pure Cu were subjected to HPT to examine the effect of applied pressure, number of revolutions and position of the sample on the microstructures and the mechanical properties. The results show the hardness increases with increasing applied pressure or with increasing numbers of revolutions but these increases tend to become saturated and the saturation is more easily attained in the outer area than in the central area. Microstructural observations revealed that the outer part consists of fine grains having high-angle grain boundaries with a grain size of ~0.17 μm for the Al-3%Mg-0.2%Sc alloy. This alloy exhibited superplastic ductilities of ~500% when tensile specimens were taken at points away from the central area.

9:00 AM

Significantly Increased Strength and Rate Sensitivity Due to Grain Refinement or Twinning at the Nanoscale: *Ming Dao*[1]; Lei Lu[2]; Ruth Schwaiger[3]; Subra Suresh[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 8-139, Cambridge, MA 02139 USA; [2]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsch., Shenyang 110016 China; [3]Forschungszentrum Karlsruhe, Inst. for Matls. Rsch. II, Karlsruhe 76133 Germany

Indentation and tensile experiments performed on electrodeposited nanocrystalline Ni showed significantly increased rate sensitivity in addition to marked improvement in strength comparing with experiments on ultrafine crystalline Ni. A physically motivated model of Grain Boundary Affected Zone (GBAZ) is proposed to explain the observed experimental phenomenon. The GBAZ model simply assumes that, duo to the presence of a grain boundary, within a distance on the order of 10 lattice parameters, the material is plastically softer than the grain interior material. A simple unit cell finite element model integrating the GBAZ concept is able to capture the correct experimental trends. Taking the assumption that the twin boundaries of nano-sized twins have the similar effect as grain boundaries of nanocryalline materials, the concept of Twin-Boundary Affected Zone (TBAZ) can be integrated into a polycrystalline model to study the significantly increased plastic strength and rate sensitivity in pure Cu with nano-sized twins. Within the proposed physically motivated crystal plasticity framework, the orientation and size dependent plastic behavior parallel (plastically softer) and perpendicular (plastically harder) to the twin boundaries is specifically modeled. Parametric studies showed that the proposed model can correctly capture the significantly increased plastic strength and rate dependence versus a decreasing nanoscale twin thickness. Possible influences on the ductility due to the presence of many nano-sized twins are also discussed.

9:15 AM

Deformation Mechanisms of Nanostructured Materials: *Yuntian Ted Zhu*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanostructured materials deform via mechanisms not accessible to their coarse-grained counterparts. Partial dislocation emission from grain boundaries, stacking faults and deformation twinning may occur in metals such as Al, which does not deform by twinning in its coarse-grained state. In this presentation I'll discuss several deformation mechanisms in nanomaterials as well as their formation conditions.

9:30 AM

Influence of Stacking Fault Energy on Deformation Twinning in Nanocrystalline Cu and Cu Alloys: *Yonghao Zhao*[1]; Xiaozhou Liao[1]; Yuntian Ted Zhu[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Kyushu University, Dept. Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Deformation twinning has been observed in nanocrystalline copper and aluminium although these two face-centered-cubic (fcc) metals usually do not deform by twinning in their coarse-grained state. It has been found that partial dislocation emission from the boundaries of nano-grains is responsible for the formation of deformation twins and this twinning mechanism is different from those operating in coarse-grained materials. The stacking fault energy is known to affect the twinning in coarse-grained materials but its effect on twinning in nanocrystalline materials has not been studied. This paper presents a systematic study of the influence of stacking fault energy on deformation twinning in nanocrystalline copper and copper alloys processed by high pressure torsion.

9:45 AM

Deformation Twinning in Nanocrystalline Stainless Steel Within Adiabatic Shear Bands: *Qing Xue*[1]; Xiaozhou Liao[1]; Yuntian Theodore Zhu[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., G755, Los Alamos, NM 87545 USA

Transmission electron microscopy was used to investigate the nanostructure and the deformation mechanism of adiabatic shear bands in a cold-rolled stainless steel deformed during dynamic forced shear tests, in which the localized deformation occurred under high strain rates and locally high temperature. The development of adiabatic shear localization resulted in continuous refinement of the original coarse grains to nanocrystalline grains. High-resolution transmission microscopy examination showed extremely high density of nanotwins within the nanocrystalline grains at the centers of shear bands, indicating that twining played a significant role in the localized deformation when the grain sizes were at the nano range. The twinning mechanism in nano-scale stainless steel will be discussed.

10:00 AM

Design of Microstructures of Difficult-to-Work Alloys Utilizing Deformation Twinning During Severe Plastic Deformation: *Ibrahim Karaman*[1]; [1]Texas A&M University, Dept. of Mechl. Engrg., MS 3123, Coll. Sta., TX 77843 USA

The works of Prof. Langdon on severe plastic deformation (SPD) of metal alloys have helped understanding of the mechanisms of the SPD stages and often inspired the author in his studies on the SPD of difficult-to-work alloys. This talk will summarize our recent work on severe plastic deformation processing of several structural and smart materials using Equal Channel Angular Extrusion (ECAE). The common characteristic of these studies was the utilization of deformation twinning to modify the microstructure to obtain submicron and nanograins with low CSL boundaries. We will present the common features we have observed in Ti-6Al-4V, AISI 316L stainless steel and NiTi shape memory alloys and challenges and opportunities for twinning induced grain boundary engineering. The unique observation was that significant deformation twinning activity was observed for the first time in Ti-6Al-4V and 316L stainless steel at temperatures as high as 800°C (0.65 Tm!). Possible mechanisms of twin nucleation in these unusual cases will be discussed. ECAE of NiTi led to the observation of highly organized, twin-related nanograins in the high temperature phase which enhance cyclic stability and fatigue resistance of this alloy. Deformation twinning in a B2 intermetallic is an additional mechanism that improves the ductility of these materials. The formation of well-organized twin-related nanograins via severe plastic deformation opens a new opportunity for twinning induced grain boundary engineering in Ti-6Al-4V, austenitic steels and B2 NiTi intermetallics.

10:15 AM

Multiscale Investigations of Grain Refinement Using Severe Plastic Deformation: *Igor V. Alexandrov*[1]; Ruslan Z. Valiev[1]; [1]Ufa State Aviation Technical University, 12 K. Marx, Ufa 450000 Russia

The current work represents the results of combined computer modeling and experiments conducted by Ufa team on different scale (macro-, meso- and micro-) levels. These investigations aimed to study the equal channel angular (ECA) pressing process, reveal the peculiarities and mechanisms of the crystallographic texture development and microstructure refinement. The homogeneity of the obtained texture and microstructure in pure SPD metals depending on the processing parameters has been studied as well. The obtained results help to optimize ECA processing of the bulk nanostructured ingots and to predict regimes of the development of homogeneous structure and texture in pure metals with the different crystal lattice such as FCC copper and HPC titanium. The work was carried out within the framework of project CRDF 10505 Model-driven manufacturing of nanocrystalline structures (project coordinator Dr. I.J. Beyerlein) in cooperation with scientists from LANL (Los Alamos), VNIIEF (Sarov), PTI (St. Petersburg).

10:30 AM Break

10:45 AM
Plasticity and Strength of Nanocrystalline Ni and Ni-W Alloys: *T. G. Nieh*[1]; Jeffrey Wadsworth[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Bethel Valley Rd., No. 1, Oak Ridge, TN 37831 USA

In this paper, both experiments and simulation results from nanocrystalline Ni with a grain size less than 20 nm are presented. The effect of grain size, the type of grain boundary, and alloying on the strength and plasticity will be discussed. Microstructure and impurity distribution in the nano-alloys both before and after deformation are examined using techniques such as atom probe microscopy (APM), electron energy loss spectroscopy (EELs) with a nano-sized probe, and high-resolution TEM with high angle angular dark field (HAADF) imaging capability (i.e. Z-contrast). Fracture process in nanocrystalline alloys will be addressed. MD simulation to show tension-compression strength asymmetry in nanocrystalline Ni will also be presented.

11:00 AM
On Applying Nano-Grained Superplastic Materials to General Residential Seismic Dampers: *Kenji Higashi*[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

Recently the superplastic damping device, which has been using Zn-22Al alloys with an ultra-fine (nano order) grain size, has been put into practical use for a high-rise building. For the purpose of the improvement and mass-production of the damping devices by nano-grained superplastic materials, the investigation about the mechanical properties in the extruded alloys and the capability of superplastic forming was carried out. The extruded alloys, produced by optimum treatments, exhibited lower stress and higher elongation in comparison with the rolled alloys, i.e., the superplasticity at higher strain rate occurred. As the results of FVM analysis, it was verified that the formability of the rectangular samples was inferior to the rod samples because of the high effective strain at the corner. The tensile properties after the forging deteriorated due to the grain growth during the forging, but satisfied the mechanical properties needed to utilize as a damper in practice.

11:15 AM
Modelling Grain Boundary Strengthening in Ultra-Fine Grained Aluminium Alloys: *Erik Nes*[1]; Bjørn Holmedal[1]; Knut Marthinsen[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Alfred Getzv. 2, Trondheim N-7491 Norway

By careful annealing of heavily deformed metals ultra-fine grained materials can be obtained. This phenomenon has been known for long and utilised in the production of special aluminium sheet qualities. It has received new interest with the emergence of the equal channel angular pressing (ECAP) technique, where pioneering works have been performed by T. Langdon and co-workers. In the present work the mechanical properties of aluminium alloys with grain sizes in the range from less than a micron (ultra-fine) to hundreds of microns have been modelled within the framework of the multi-parameter microstructural work hardening model developed by Nes and co-workers. The effect of grain size on the flow-stress and work hardening, including a deviation from the Hall-Petch grain size dependency for ultra-fine grain sizes, is well accounted for by the model. A mechanism is suggested for the sharp yield point and associated Lüder-band elongation relevant for ultra-fine grained AlMg-alloys.

11:30 AM
Nano- and Submicrocrystalline Steels by Severe Plastic Deformation: *Sergey Dobatkin*[1]; [1]Russian Academy of Sciences , A.A. Baikov Inst. of Metall. & Matls. Sci., Leninsky prospekt, 49, Moscow 119991 Russia

The aim of this paper is to consider the features of structure evolution during SPD of steels, thermal stability of the UFG structure processed and its influence on mechanical properties. The investigation have been carried out mainly on low and high carbon steels as well as on austenitic and ferritic stainless steels after SPD by torsion under high pressure and equal channel angular (ECA) pressing. Structure formation dependencies on temperature deformation conditions, strain degree, chemical composition, initial state and pressure are considered. The role of phase transformations for additional grain refinement, namely, martensitic transformation, precipitation and dissolution of carbide particles during SPD and others, is underlined. Mechanical properties of several steels made by various ways of SPD such us multiple all-round forging, multi-axis deformation, accumulative roll-bonding, alternating bending and ECA pressing, are compared in relation to their structure.

11:45 AM
Superplastic Flow in Materials Processed by ECAP: *N. Balasubramanian*[1]; Terence G. Langdon[2]; [1]R V College of Engineering, All India Council of Techl. Educ., Mysore Rd., Bangalore 560 059 India; [2]University of Southern California, Aeros. & Mechl. Engrg. & Matls. Sci., 3650 McClintock Ave., Rm. OHE430G, Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing (ECAP), when carried out optimally, produces submicron grain size, equiaxed grains and a significant fraction of high angle boundaries - conditions for high strain rate and/or low temperature superplasticity. Experimental results on superplastic flow after ECAP processing of various alloys will be reviewed. The stress and grain size exponents in the usual strain rate equation,the activation energy and the role of internal stress will be analyzed with a view to identifying the rate-controlling mechanisms.

12:00 PM
A Look into an Aluminum Nano-Composite Material for Advanced Aerospace Fastener Technology: *Patrick B. Berbon*[1]; Steven G. Keener[2]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]The Boeing Company, 2401 E. Wardlow Rd., MC C078-0533, Long Beach, CA 90807-5309 USA

Every aircraft produced today contains hundreds of thousands of fastened joints. These joints and the fasteners that connect them are perhaps the most common source of failure in aircraft structure. Therefore, it is imperative that advancements in fastener materials and designs be given the utmost consideration and attention to achieve increased joint performance and integrity. This paper presents the results of development efforts relating to an advanced processing technique, namely cryogenic milling, and its effect upon material microstructures and mechanical properties. These metallic nano-composites are found appropriate and important for potential fastener applications. By employing this nano-composite material processing technique in the production of aerospace fasteners, the fasteners' performance characteristics are enhanced and the manufacturing costs are reduced, by elimination of processing steps, e.g., thermal treatment, and/or reduction in unacceptable fastener installations.

12:15 PM
The Influence of Boundary Structure on the Mechanical Property in a Commercial Purity Aluminum: *Pei-Ling Sun*[1]; Ellen Kathleen Cerreta[1]; George T. Gray III[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

A commercial purity aluminum AA1050 was subjected to equal channel angular extrusion (ECAE) route A, the billet is not rotated, and C, rotated 180º, between extrusion passes respectively, to a von Mises strain of 8.4. The resulting microstructures have similar grain sizes but relatively different grain boundary structures. The microstructure processed by route A has ~70% high angle boundaries (HABs) while the route C microstructure has ~38% HABs. It was found that boundary structure plays an important role in the mechanical properties of the material because these boundaries act as a source and sink of dislocations. Mechanical tests conducted on these two microstructures at intermediate and dynamic strain rates at both 77 and 298K are compared with the results of material tested at quasi-static strain rates.

The Role of Technology in the Global Primary Aluminum Industry Today and in the Future

Sponsored by: Light Metals Division
Program Organizer: Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday AM Room: 2000
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

8:30 AM
Presentation 1: *Cynthia Carroll*[1]; [1]President and CEO of Alcan Primary Metal Group, Canada

8:55 AM
Presentation 2: *Truls Gautesen*[1]; [1]President of Hydro Aluminium Primary Metal, Norway

9:15 AM
Presentation 3: *Wayne Hale*[1]; [1]Executive Vice President of Alumina and Aluminium, Sual, Russia

9:35 AM Break

9:45 AM
Presentation 4: *Xiangmin Liu*[1]; [1]Vice President, Aluminum Company of China Limited (Chalco), Beijing, China

10:05 AM
Presentation 5: *Valery Matvienko*[1]; [1]Managing Director, Aluminium Division, Rusal, Russia

10:25 AM
Presentation 6: *Bernt Reitan*[1]; [1]Vice President of Alcoa Primary Products, USA

10:45 AM Break

11:00 AM Panel Discussion
Panel Moderator: James W. Evans, University of California, Dept. of Matls. Sci. & Mineral Engrg., Berkeley, CA 94720 USA

TUESDAY PM

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Processing

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday PM Room: 2009
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mahmoud Y. Demeri, FormSys Inc., Northville, MI 48167-3427 USA; Amit K. Ghosh, University of Michigan, Dept. Matls. Sci. Engrg., Ann Arbor, MI 48109 USA

2:00 PM Invited
Improved and Affordable Hot Forming Technologies: *Amit K. Ghosh*[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109 USA

The need to use lightweight materials in aerospace and automotive applications have constantly created the need to innovate new processing methods to deal with rising manufacturing costs. The use of complex aluminum and magnesium alloys to replace steel, without the loss of design flexibility, has spawned a series of warm and hot forming techniques and new methods of material preparation. In this paper, some latest developments in this area will be reviewed, that help to take advantage of higher forming rate, lower forming temperature and use of complex constitutive behavior of materials inherent in such processing operations. (Work supported by National Science Foundation, DMR Grant.)

2:25 PM
Parametric Analysis for Warm Forming of Aluminum Blanks Using 2D FEA and DOE - Effects of Temperature Distribution, Friction and Speed: Peng Chen[1]; *Muammer Koç*[1]; [1]University of Michigan, S. M. Wu Mfg. Rsch. Ctr. & Dept. of Mechl. Engrg., Coll. of Engrg., Ann Arbor, MI USA

The effects of temperature distribution, speed, holding time and friction on warm forming performance are investigated for 5083-O (Al-Mg) sheet metal blanks. Combined isothermal/non-isothermal FEA with DOE tool is used to predict appropriate warm forming temperature conditions for deep drawing and two-dimensional stamping cases. In the ranges investigated (temperature: 25-250°C; speed: 2.5-5 mm/s; holding time: 1-3 sec; friction coefficient: 0.06-0.2), the formability of Al-5083 alloy is found to be greatly dependent on the temperature of the die and punch. To achieve increased degrees of forming, different temperature levels should be assigned to the corner and body of the die and punch in conjunction with a slow forming speed and combination of die-blank low friction and punch-blank high friction.

2:45 PM
The Effect of Lubrication on QPF Formability: *Paul E. Krajewski*[1]; [1]General Motors, R&D Ctr., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA

Lubrication plays an important role in the ability to manufacture components by quick plastic forming (QPF). This occurs both through facilitating metal flow during deformation but also enabling part release from the die. The present paper provides two examples of using lubrication to prevent necking and thereby decrease the time required to make QPF components. The first example shows how the addition of milk of magnesia to boron nitride can modify friction and be used to reduce necking over sharp entry radii. The second example describes how tailored application of lubrication at entry radii can produce the same effect.

3:05 PM
Effect of Copper Additions on Superplastic Behavior of 5083 Aluminum Alloy: *Sooho Kim*[1]; Ravi Verma[1]; [1]General Motors, R&D Ctr., Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

Four 5083 aluminum alloys with varying copper and manganese concentrations were thermomechanically processed to a thickness of 2 mm. All rolled alloys exhibited highly refined grain structures with average grain sizes below 10 μm. Alloy A3, with the highest copper content (0.78%), showed the highest tensile elongation and lowest flow stress under most test conditions. This alloy exhibited peak strain-rate sensitivity at a strain-rate of 1x10-2 s-1, which is an order of magnitude higher than typical peak strain-rate for a base 5083 aluminum alloy, suggesting potential for faster forming with the copper-modified alloy. In biaxial pan forming tests, only the two copper-containing alloys (A2 and A3) produced fully formed pans. Of the two, A3 showed the least die-entry thinning. TEM characterization revealed that A3 had finer size, higher population density, and more uniform distribution of intermetallic particles than A2. It is believed that the finer particle size distribution of A3 is more effective in pinning grain-boundaries, and in turn is responsible for higher superplastic properties.

3:25 PM
Development of High Performance Alloy Sheets by Strip Casting: *Nack J. Kim*[1]; Sunghak Lee[1]; Sung S. Park[1]; Jung G. Lee[1]; [1]POSTECH, Ctr. for Advd. Aeros. Matls., San 31, Hyojadong, Pohang 790-784 Korea

Recently, strip casting has received a large attention due to its several advantages over conventional solidification processes such as continuous casting and direct chill casting. Strip casting combines solidification and hot rolling into one operation, thereby eliminating or reducing the hot rolling needed for conventionally produced ingots. Strip casting also offers much reduced cooling rates over conventional continuous casting or direct chill casting, resulting in the improvement of microstructural features. Such characteristics of strip casting are ideal for the development of high performance alloy sheets. The present paper discusses our efforts on developing the high performance alloys such as Mg and bulk metallic glass (BMG) alloys by strip casting. The impetus for the present research comes from the fact that there are virtually no wrought Mg and BMG alloys with respectable mechanical properties. The development of these alloys, particularly in sheet form, would greatly expand their applications in transportation industries. Microstructure and mechanical properties of strip cast alloys will be discussed with particular emphasis on the solidification behavior during strip casting.

3:45 PM Break

4:00 PM
Low-Cost Aluminum Tubes for Hydroforming Applications: *Alan A. Luo*[1]; Anil K. Sachdev[1]; [1]General Motors Research & Development Center, Matls. & Processes Lab., 30500 Mound Rd., MC 480-106-212, Warren, MI 48090 USA

Low-cost aluminum tubes made from twin-belt continuous casting sheet 5754-CC were evaluated for hydroforming applications, in comparison with extruded 6063 seamless tubes and seam-welded 5754-DC tubes (made from direct-chill cast aluminum sheet). The results show that the low-cost 5754-CC tubes offer acceptable dimensional tolerances and formability upon bending and hydroforming, as well as good mechanical properties after hydroforming. The microstructural evolution of the three materials during bending and hydroforming were closely investigated.

4:20 PM
Damage Tolerance and Durability of Glass Fiber Reinforced Aluminum Laminates for Aircraft Structures: *Guocai Wu*[1]; Jenn-

Ming Yang[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095 USA

Glass fiber reinforced aluminum laminate (GLARE) is a new class of fiber metal laminates (FML) consisting of alternating layers of thin aluminum sheets and unidirectional or biaxial reinforced adhesive prepregs of high strength glass fibers. Due to its outstanding fatigue resistance, high specific static properties, excellent impact resistance, good flame resistance and corrosion properties, it offers the aircraft structural designer a damage-tolerant, light-weight and cost-effective solution for advanced transport structural applications. More recently, GLARE has been selected for the upper fuselage skin structures of Airbus A380. However, the full potential of GLARE as aircraft primary structures has not been fully explored yet. More research and testing are necessary to generate adequate data to facilitate greater utilization of GLARE. One of the important structural integrity issues and concerns is the damage tolerance and durability for airworthiness of GLARE for aircraft structures. This paper presents the investigation results of damage tolerance and durability assessments of newly developed GLARE with cross-plied S-2 glass prepregs. An abroad impact testing program was performed, and the post-impact residual strength and fatigue behavior were extensively investigated to evaluate the damage tolerance of GLARE compared to the monolithic aluminum alloys. The crack propagation, damage progression and detection under static and dynamic loading after impact were investigated with microscopy, X-ray radiography, and by chemically removing outer aluminum layers.

4:40 PM
The Effect of Welding Parameters on the Microstructure and Microhardness of Resistance Spot Welded Galvannealed Steel Sheets: *Cherqueta Romeca Claiborn*[1]; Viola L. Acoff[1]; [1]University of Alabama, Metallurgl. Engrg., 129 Bevill Bldg., 126 7th Ave., Box 870202, Tuscaloosa, AL 35487-0202 USA

Resistance spot welding is used extensively in the automotive industry for joining low carbon steel components in high-volume and high rate production. However, spot welding often results in inconsistent weld quality. This study examines the effect of welding parameters on the microstructure and microhardness of resistance spot welded galvannealed steel. The specimens were welded at various percent currents while holding all other parameters constant. Light microscopy, microhardness testing, nanoindentation measurements, scanning electron microscopy, and x-ray diffraction were used to characterize the welded samples. Microhardness measurements taken across the weld nugget of each sample showed an increase in the microhardness values as the percent current increased. The purpose of this study is to investigate the cause of the observed increase in microhardness.

5:00 PM
Microstructural Evolution During Hemming: *John E. Carsley*[1]; [1]General Motors Corp., R&D, MC 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA

Aluminum sheet is typically more difficult to hem than steel due to edge cracking. The difference has been attributed to the susceptibility of aluminum to strain localization during the hemming process. Recently developed techniques such as roller hemming, which have been used by automakers to assemble aluminum and steel components, have been shown to improve hemming of aluminum. The present study investigates microstructural evolution during both conventional and roller hemming to understand the effect of strain path on fracture during hemming/bending operations.

Alumina and Bauxite: Influences of Alumina on Smelter Performance

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2005
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Tony Bagshaw, AMIRA International Ltd., W. Perth, Western Australia 6872 Australia

2:00 PM
SGA Requirements in Coming Years: *Stephen J. Lindsay*[1]; [1]Alcoa, Inc., Primary Metals Div., 300 N. Hall Rd., MS S-01, Alcoa, TN 37701-2516 USA

Refineries report various physical and chemical properties of Smelting Grade Alumina, SGA, on Certificate of Analysis data sheets. Without strong understanding of customer needs such posted properties can fall short of the true needs of smelters and downstream customers. An example is smelting efforts to improve excess fluoride control in pot bath, but with no specification or target for the variability of Na2O content. Downstream customers for aluminum conductor products require excellent electrical conductivity, but this does not always translate itself into alumina shipping limits for properties such as Cr2O3, MnO or V2O5. The author presents these and other examples for consideration during the joint sessions between Alumina & Bauxite and Aluminum Reduction Technology.

2:25 PM
Effects of the Vessel Loading and Unloading on Quality Variations (Particle Size) of Alumina: *Carl Behrens*[1]; [1]Hydro Aluminium Primary Metals, Alumina, Bauxite & Energy, PO Box 2560, Porsgrunn N-3907 Norway

Hydro Aluminium's smelter system is frequently using alumina from 10-15 different sources. In the feed back from the smelters, some sources are characterized as more variable than others. Also some individual shipments from otherwise well reputed qualities show occasionally variable behaviour when processed at the smelter. The sources characterized as variable, manifest them selves in higher pot instabilities such as settings for process control systems (transport systems, pot controls etc.) as well as inferior performance. The variability in smelter performance is most likely attributed to variations in particle size distribution (PSD), but an open mind is kept on this aspect. Segregation processes taking place in the silo systems used along the logistic chain in general generate the variations in PSD. Hydro Aluminium has initiated a number of studies focusing on how quality variations pass through the logistic chain from the alumina refinery to the individual pots in the pot room. The work presented in the present paper focus on the segregation processes taking place during the loading and unloading of alumina vessels. The variability is expressed as the standard deviation of a number of samples collected evenly over the entire shipment. The system is characterized as segregating when the standard deviation increases from the input to the output. In the opposite case, when the standard deviation is suppressed through the system, it is characterized as homogenizing. The present work demonstrates how the loading and un-loading operations of alumina vessels contribute to a reduction of the quality variations by a factor of 2-4 (ratio: Sin:Sout). Consequently the maritime transport system contributes to a homogenisation of the quality.

2:50 PM
Alumina Phase Distribution, Structural Hydroxyl and Performance of Smelter Grade Aluminas in the Reduction Cell: *James B. Metson*[1]; Margaret Hyland[2]; Tania Grouts0[1]; [1]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand; [2]University of Auckland, Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand

Recent developments in the phase analysis of smelter grade aluminas have shown that aluminas with nominally similar specifications differ significantly in their phase compositions, particularly in the relative amounts of the transition phases. These differences will affect alumina performance in the smelting cell, for example the potential for the alumina to generate HF. The transition phases are the likely source of residual structural hydroxyl, which recent experimental studies have linked to HF generation in the cell. It is of interest to understand the role of alumina structure, and especially the distribution of residual structural hydroxyl, which is strongly influenced by calcination conditions. In laboratory prepared gamma alumina the residual hydroxyl is thought to be located on the exterior of the alumina structure and at grain boundaries, but for smelter grade aluminas, the very rapid calcination conditions must influence this -OH distribution. Structural analysis studies on a range of SGAs suggest different capacities for HF generation which are not closely tied to the industry specifications for these materials.

3:15 PM
Adsorption/Entrainment of Fluoride in Smelting Grade Alumina: Surface Chemical Speciation and Adsorption Mechanism: *Neal R. Dando*[1]; [1]Alcoa, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

Smelting grade alumina (SGA) is employed to scrub vapor-phase HF from the exhaust gases of aluminum smelters using either fluidized bed or injection scrubbers. As aluminum plants continue to increase production rates, the efficiency of the fluoride recovery process comes under elevated scrutiny, owing to the considerable cost of fume treatment centers. This elevates the need for an improved understanding of

the factors impacting fluoride evolution and recovery. Comparative characterizations of the short-range bonding and chemical speciation of lab and plant reacted smelting grade aluminas were employed to refine our understanding of the mechanism of fluoride adsorption on SGA surfaces. We have been able to identify and measure the relative populations of at least 6 different amorphous-state adsorbed and entrained fluorides in plant-reacted ore. The relative concentrations of these species reflect dry-scrubber operating conditions and pot-tending practices.

3:40 PM Break

3:55 PM
Exposures of Concern for Developing Occupational Asthma During Production of Primary Aluminium: *Y. Thomassen*[1]; N. P. Skaugset[1]; D. Ellingsen[1]; L. Jordbekken[1]; H. Notø[1]; [1]National Institute of Occupational Health, PO Box 8149, DEP., NO-0033 Oslo Norway

The quality of workroom atmospheres in potrooms have traditionally been monitored by measuring workers exposures to "total" dust and gases including particulate fluorides, hydrogen fluoride, sulphur dioxide and coal tar pitch volatiles (e.g. PAH's). Bronchial hyperresponsiveness (potroom asthma) is despite exposures below international occupational exposure limits (OEL's) for the contaminants mentioned above, still frequently occurring among workers. During recent years a high number of suspected potroom asthma and asthma like symptom cases have been reported by the Norwegian aluminium industry to the Labour Inspection Authority. Recently, a dose-response relationship has been suggested for fluoride exposures, but whether fluorides are the causative agent, co-agent or simply markers for causative agent(s) for potroom asthma, remains to be determined. To better understand the complex workroom composition in aluminium potrooms and the temporal variability in worker's exposure, new air monitoring strategies with the use of aerosol samplers for the health related aerosol fractions are required and has been included in an ongoing project (HAPPA) for the Nordic aluminium industry . Aerosol mass, total and water soluble fluorides, and beryllium in the respirable/thoracic/inhalable fractions in addition to HF and SO2 are measured in 10 different potrooms representing 6 smelters and Søderberg and Prebake-technologies. The use of personal direct reading instruments for SO2 and aerosols is used to obtain information about exposure variability. These exposure indicators may allow more complete description of workroom air contamination situation in Al-potroom. Preliminary results from this monitoring project will be presented.

4:20 PM
Ultrafine Particles at Workplaces of a Primary Aluminium Smelter: *Y. Thomassen*[1]; W. Koch[2]; D. Ellingsen[1]; N. P. Skaugset[1]; W. Dunkhorst[2]; L. Jordbekken[1]; P. A. Drabløs[3]; [1]National Institute of Occupational Health, PO Box 8149 DEP, N-0033 Oslo Norway; [2]Fraunhofer Institute of Toxicology and Experimental Medicine, Nikolai- Fuchs-Str. 1, D-30625 Hannover Germany; [3]Karmøy Plant Norsk Hydro, Håvik Norway

This study was aimed at measuring number concentration, number size distribution, particle morphology and ultrafine particles directly at the source during the process of changing anodes, and at locations representing typical mean exposure situations to aged smoke in plant site of an Al-smelter. The average "background" total number concentration is 20000 [particles/cm³] in Prebake area with concentration peaks an order of magnitude higher related to anode change operations. Søderberg technology produces more pollution with average concentrations of the order of 80000 [particles/cm³]. Time averaged size distribution exhibits a bimodal structure with a peak at or below 10 nm and a second peak at larger size (50-100 nm). Airborne particles show a variety of morphological structures including long thin fibers, chain like agglomerates and different forms of crystallites. Only a small fraction of the particles associated with the 10 nm peak of the number size distribution could be found on TEM-pictures indicating that these particles might be volatile. This study shows existence of elevated number concentrations of nanoparticles at workplaces in the primary aluminium industry. Main emission source of this particle size fraction in Prebake is open bath during anode changing. Nanoparticles were measured directly at the source but could also be identified as episodes of high number concentrations in the general "background" air in the Prebake production hall.

4:45 PM
Origins and Effects of Potroom Dust: *Margaret M. Hyland*[1]; Mark P. Taylor[1]; [1]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand

Dust is defined as the fine particulate material that escapes confinement and causes a nuisance or a hazard in the workplace. This paper examines the origins of fine particulates and their impact on process stability and efficiency. This impact is complicated by the fact that it may not be observed on every cell in the line, because of the material's tendency to segregate in handling systems and is further confounded because once a disturbance has been set up in a cell, process conditions work against the containment of particulate and gaseous emissions. Recent work has also demonstrated that the fine fraction of crushed anode cover can have a dramatic effect on potline process stability and dust release. A study is now underway to investigate the origins of potroom dust, and the factors which control its generation and release into the environment. The role of alumina, both directly and through the disruption of reduction cells in the potline is of particular interest. Progress in understanding the origin of dust in the potrooms is reviewed, in light of these investigations.

5:10 PM
Alumina Dissolution and Sludge Formation Revisited: *Rudolf Keller*[1]; [1]EMEC Consultants, 4221 Roundtop Rd., Export, PA 15632 USA

Alumina added to the bath of an aluminum reduction cell does not immediately sink into and through the bath, because of its low bulk density (aside from temporarily freezing some bath). Bath penetrates into the bulk alumina, and in this process, g-alumina transforms into a-alumina and agglomerates are formed. Such agglomerate pieces may sink through the bath to the metal-bath interface. Provided their size and density are sufficient, accumulations of agglomerates may sink through the metal to form sludge at the carbon-metal interface. There can be conditions, however, where they remain at the bath-metal interface. Possible impacts of such accumulations are discussed along with the current distribution in the presence of bottom sludge and the formation of aluminum below the sludge.

5:35 PM
The Model of Dissolving and Heating of Alumina During its Supplying Through PF in the Program "Virtual Cell": *Alexander Berezin*[1]; [1]RUSAL Engineering & Techonology Center, Krasnoyarsk 660011 Russia

PFS (Point Feeding System) Control algorithms, taking into account parameters of bath alumina dissolving are necessary for increasing of cell efficiency, in particular for reducing of anode effects and cell output increasing. Modeling of dissolving process dynamics and alumina heating during its intake through the PFS is necessary for the definition of these algorithms. This work represents mutual solution of the following goals: -Modeling of the process of alumina dissolving on the basis of experimental data taking into account different qualities of alumina. -Modeling of thermal particles interaction and alumina agglomerates with the bath. -Modeling of time and alumina agglomerates settlement quantity at the interface bath-metal. The models are realised in the program "Visualised cell". Test calculations are fulfilled. The results of technical data alteration (cell voltage, bath temperature, overheating, alumina concentration, settlement quantity etc.) are presented during the work of PF.

Aluminum Reduction Technology: Cell Development & Operations - Part 2

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2003
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Claude H. Vanvoren, Alcan, Primary Metal Grp., Voreppe 38341 France

2:00 PM
A Case Study on Life Cycle Analysis of Copper Anode Bar: *Jyoti Mukhopadhyay*[1]; Y. V. Ramana[2]; Rajnish Kumar[2]; [1]Jawaharlal Nehru Aluminium Research Development and Design Centre, Amravati Rd., Wadi, Nagpur, Maharashtra 440 023 India; [2]Hindalco Industries Limited, R&D, PO Renukoot, Renukoot, Uttar Pradesh 231 217 India

During aluminium production, frequent failure of copper anode bar was observed. The average life cycle of the bar was found to reduce from 50 cycles to 20 cycles during a defined time span. While reduction, it was observed that both stiffener and the bar were continuously exposed to flue gases. Failures in the vicinity just above the MS stiffener take place due to gradual reduction in cross-sectional area of the

Cu-bar as a result of oxidation, pitting and crack formation. Microstructural analysis also reveals that the presence of pits/voids are also responsible for such failure. In fact, the failure was attributed mostly due to the intergranular mode. In order to prevent such failures, a few important remedial measures were undertaken. Among them, stringent quality control of the copper metal before and after the fabrication of bar as well as also increasing the height of the stiffener could well be explored.

2:25 PM

Application of Aluminum-Copper Bonded Sheet in Aluminum Reduction Cells: *Kayron F. Lalonde*[1]; Mark D. Ohlswager[2]; [1]Alcoa, Inc., Primary Metals, PO Box 472, Rockdale, TX 78664 USA; [2]Elkem Aluminium, Hoffsveien 65B, PO Box 5211 Majorstuen, Oslo No-0303 Norway

Use of aluminum-copper bonded sheets on the anode riser joint connection of prebake aluminum reduction cells has eliminated the occurrence of high voltage drops across these joints. The sheets were installed between the solid aluminum leaves of the anode buswork and the copper tabs of the anode riser-flexibles bolted to the moveable anode bus. Prior to bimetal sheet installation, half of the riser joints would increase in voltage over time, as much as 350 millivolts, requiring expensive disconnecting, cleaning and remaking of the joint connections. Since the bimetal sheet installation began two years ago, all new joints have remained at normal voltage, resulting in a significant reduction of the voltage drop external to the cell.

2:50 PM

The Impact of Anode Cover Control and Anode Assembly Design on Reduction Cell Performance - Part 2: *Evan W. Andrews*[1]; Mark P. Taylor[2]; Greg L. Johnson[1]; Ian Coad[1]; Geoff P. Brookes[1]; [1]Boyne Smelters, Boyne Island, Queensland Australia; [2]University of Auckland, The Light Metals Rsch. Ctr., New Zealand

Reduction line amperages have been increasing around the world in recent years as companies seek to increase the return from existing assets. The key aspects of anode cover quality and anode hanger design on the cell top heat balance were exemplified in Part 1 of this work, published in TMS Light Metals 2004. The anode assembly design criterion, which is now most critical for the stability of the cell is the dissipation of sufficient heat from the assembly and the cover to maintain component temperature and overall cell heat balance. This paper completes last year's work, with presentation of an operational anode cover control loop that has permitted tighter control of the heat balance in cells. The importance of the anode assembly design on the cell operating heat balance window and reduction of anode problems is also considered, especially for smelters approaching their upper amperage and heat balance limits. Plant data is presented to demonstrate and support the practical aspects of the work.

3:15 PM

Fluoride Evolution/Emission from Aluminum Smelting Pots: Impact of Ore Feeding and Cover Practices: *Neal R. Dando*[1]; Robert Tang[2]; [1]Alcoa, 100 Techl. Dr., Alcoa Ctr., PA 15668 USA; [2]Alcoa, Smelting Div., Eastalco Works, Frederick, MD 21703 USA

Dry scrubbers use smelting grade alumina (SGA) to capture vapor-phase fluoride from the exhaust gases of aluminum smelters. The reacted SGA from the scrubbers is then fed back to the pots to recover the lost fluoride. A range of smelter-owned factors such as pot chemistry, operating practice, pot-tending practice, and ore feeding dramatically affect the dynamics of this recovery process. This presentation will discuss real-time measures of the impact of ore feeding practices and tending practices on fluoride emission and evolution from aluminum smelting pots. The dynamics of fluoride release from reacted SGA directly fed to the pots was studied by varying the rate and dynamics of the ore feed cycle. Of particular interest is the relative magnitude of pot-tending practices on emission and evolution.

3:40 PM Break

3:55 PM

Anode Cover Material and Bath Control: *Siegfried Wilkening*[1]; Pierre Reny[2]; Brian Murphy[2]; [1]Hydro Aluminium T & P, PO Box 2468, Bonn 53014 Germany; [2]Aluminery Alouette Inc., Case postale 1650, Sept Iles, Quebec G4R5M9 Canada

The various types of recycled bath material and anode cover material are defined in clear terms. The basic properties of primary anode cover material are described and discussed as well as their testing and control in the laboratory. Further important aspects are dealt with, such as the handling, crushing, grinding, classification and storage of recycled bath materials, followed by the preparation and control of a suitable and consistent anode cover material in modern potlines. Examples of the equipment generally used in bath treatment centres, or which may be employed in the future, are given.

4:20 PM

CFD Modeling of the Fjardaal Smelter Potroom Ventilation: *Jon Berkoe*[1]; Philip Diwakar[1]; Lucy Martin[1]; Bob Baxter[1]; Mark Read[1]; Patrick Grover[2]; Don Ziegler[2]; [1]Bechtel M&M, N. America, 1500, rue Université, Bureau 910, Montréal, Québec H3A 3S7 Canada; [2]Alcoa, 6603 W. Broad St., Richmond, VA 23230 USA

The Fjarðaál Smelter potline buildings are designed to achieve ventilation of heat and fugitive contaminants using a system based on natural air circulation. The design of the potline buildings is based on Alcoa's Aluminerie Deschambault smelter facility, located in Deschambault Quebec, Canada. The Fjarðaál Smelter Project potline buildings are longer, with reduced spacing between buildings and are situated on a sloping site adjacent to a fjord located in eastern Iceland. The Fjarðaál Project is faced with additional unique factors like local terrain, high wind speed, and multiple approach wind directions - that make the predictability of the ventilation system performance more complex. To help guide the ventilation design, computational fluid dynamics (CFD) modeling was employed. The CFD analysis utilized state-of-the-art capabilities to capture in detail the velocities, temperatures, pressures, and pollutant concentrations inside and outside the buildings. The model was validated against smoke tests conducted at the Deschambault smelter. The model demonstrated that the ventilation system performance is relatively unaffected by the terrain and winds, implying that the claustra wall design is very effective. Pressure gradients can cause locally non-uniform flow patterns in the potroom but these effects are manageable within the system's performance constraints.

4:45 PM

Egyptalum Experience in Operating End to End Prebaked Cells with Over 200 kA: M. M. Ali[1]; M. El-Ghonimy[1]; F. M. Ahmed[1]; Z. A. Bassiony[1]; *S. M. El-Raghy*[2]; [1]Aluminium Company of Egypt (Egyptalum), Nage-Hammadi Egypt; [2]Cairo University, Faculty of Engrg., Cairo Egypt

Egyptalum believes in continuing development. Improvements in Soderberg cells have resulted in increase of current efficiency from project design (84%) to today's level of more than 88%. A major milestone in development was the conversion of Soderberg cells into prebaked cells. This achievement has been reported in JOM in May 2003. It has gone through three phases, namely: familiarization, mathematical models and actual design, fabrication and operation. As expected, early stages of operation of the home-made 200 kA prebaked cells faced some difficulties. Overcoming these difficulties was followed by converting Soderberg line #5 into prebaked cells. Another goal was to increase the operating current. This development was achieved through 6 steps: 203 in the year 1997 up to 210 in the year of 2003. In each step, unsteady operation needed careful changes in operating parameters. Thus, many factors have been changed to match the new situation. Cell stability was one of these factors to be checked each time. Today at Egyptalum, 210 kA prebaked home-made cells are operating at a steady state showing satisfactory norms. Current efficiency reached 95%; energy consumption is 13870 k w hr/ton Al; also carbon consumption has come below 410 kg/ton Al.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Applications of Carbon-Based and Inorganic Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Tuesday PM Room: 3018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Apparao M. Rao, Clemson University, Physics, Clemson, SC 29634-0978 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany; Seung H. Kang, Agere Systems, Allentown, PA 18109 USA

2:00 PM Opening Remarks

2:05 PM Invited
Novel One Dimensional Nanostructures: *Meyya Meyyappan*[1]; [1]NASA, Ctr. for Nanotech. - Ames Rsch. Ctr., MS 229-3, Moffett Field, CA 94035 USA

Carbon nanotubes (CNTs) exhibit a combination of remarkable mechanical properties and unique electronic properties and thus offer significant potential for a wide range of applications. In this talk, our recent results on growing very well-aligned, individual, freestanding nanotubes by plasma CVD and development of bio and chemical sensors will be described. It is possible now to grow vertically oriented, well-aligned nanowires of semiconducting materials such as silicon, germanium and high temperature oxides. These inorganic nanowires exhibit very interesting electronic and optical properties compared to their two dimensional thin film cousins and are being investigated for device, laser and sensor applications. Growth of ZnO, InO and other nanowires, characterization and development of vertical transistors will be discussed.

2:35 PM Invited
Rational Design and Fabrication of High-Quality Inorganic Semiconductor Nanostructures: *L. Bock*[1]; [1]Nanosys, Inc., 2625 Hanover St., Palo Alto, CA 94304 USA

Nanosys synthesizes inorganic semiconductor nanostructures in the form of nanorods, nanowires and more complex shapes, such as cones, teardrops and tetrapods. Using computer modeling and synthetic methods, we rationally design inorganic semiconductor nanostructures with desired composition, size, shape, crystal structure, doping and surface chemistry characteristics, and successfully fabricate these materials every time. In addition to controlling the composition and structure of these nanomaterials, we can even change the composition as they are grown, forming nano heterostructures containing nano-sized heterojunctions. These junctions can be made atomically sharp, and defect-free; allowing the production of complex, high-performance electronics integrated directly within a single nanostructure. In contrast to traditional microelectronics, elements such as p-n diodes, p-i-n diodes, LEDs, bipolar transistors, etc. can be constructed chemically from the bottom up as the nanostructures are grown.

3:05 PM Invited
Recent Progress in Carbon Nanotube Field Emission Display: *Jong Min Kim*[1]; In Taek Han[1]; YongWan Jin[1]; Jun Hee Choi[1]; Jung Hee Lee[1]; Jae Eun Jung[1]; Young Joon Park[1]; Deuk Seok Chung[1]; Sang Hyun Park[1]; Hang Woo Lee[1]; [1]Samsung Advanced Institute of Technology, Matls. Lab, Keyheung NongSeoRi San 14-1, YongIn, KyoungKi 449-712 Korea

Field Emission Display (FED) having carbon nanotube (CNT) emitters has been fabricated for several years in Samsung. The phase shift from metal electron emitter to CNT has given us great success not only in the business field but also in the academic field. In this presentation, the parameters to enhance field emission property of CNT will be discussed. The study and precise control of CNTs in the form of paste and CVD grown thin films gave us much informations to use CNTs as a better electron emitter. Four components in CNT paste are CNT, frit, filler, and vehicle. The pretreatment of CNT, using proper frit and vehicle, and modifying frit resulted 10 times larger emission current and half value of operating bias. The patterning process of CNT paste has been developed and the results of hole filling process will be addressed. The CNTs are also directly grown on the FED structure by CVD method. The emission properties of CVD grown multiwalled CNT are similar with those of physically synthesized single wall CNTs. Finally our recent large screen demonstration image of FED will be shown.

3:35 PM Break

4:00 PM Invited
High Performance Nanotube/Fiber Field Emitters for Microwave Applications: *K. B.K. Teo*[1]; E. Minoux[2]; O. Groening[3]; L. Gangloff[2]; J.-P. Schnell[2]; D. Dieumegard[2]; F. Peauger[2]; R. G. Lacerda[1]; P. Legagneux[2]; D. G. Hasko[4]; G. A.J. Amaratunga[1]; W. I. Milne[1]; [1]Cambridge University, Dept. of Engrg., Cambridge UK; [2]Thales Research and Technology, France; [3]Federal Laboratories for Materials Testing and Research, EMPA Thun, Switzerland; [4]University of Cambridge, Microelect. Rsch. Ctr., UK

Multiwalled carbon nanotubes/fibers (CN) are pursued here as field emission electron sources because of their whisker-like shape, high aspect ratio, high conductivity, thermal stability and resistance to electromigration. Despite these favourable properties, it is often difficult to integrate this material into devices because of the lack of control in the synthesis of this material. We report the use of plasma enhanced chemical vapour deposition to provide ultimate control of the CN structure such as their alignment, individual position, diameter, length and shape. The CN produced from this process exhibit excellent structural uniformity, with typical standard deviation in the diameter and height of 4.1% and 6.3% respectively. Using such well controlled structures, we show it is possible to predict the electrical characteristics of the CN emitters as an individual electron source or as an array. Thus, it is now possible to build designer cathodes with predictable characteristics, and also to determine optimised arrangements for these cathodes. We demonstrate high current densities of 1A/cm^2, under DC and 1.5GHz direct modulation, from CN cathodes. These CN cold cathodes offer considerable weight and size savings over conventional hot cathode cathodes used in microwave applications (eg. SATCOM, radar).

4:30 PM Invited
Electron Filtering by Multiply-Connected Carbon Nanotubes and Field Emission of Double-Wall Carbon Nanotubes: *Jisoon Ihm*[1]; [1]Seoul National University, Sch. of Physics, Seoul Korea

We have investigated the electron transport in multiply-connected metallic carbon nanotubes within the Landauer-Buttiker formalism. Quasibound states are coupled to the incident Π* states and give rise to energy levels of different widths depending on the coupling strength. In particular, donor-like states originated from heptagonal rings are found to give a very narrow level. Interference between broad and narrow levels produces Fano-type resonant backscattering as well as resonant tunneling. Over a significantly wide energy range, almost perfect suppression of the conduction of Π* electrons occurs, which may be regarded as filtering of particular electrons. We have also performed first-principles pseudopotential calculations for the field emission from carbon nanotubes by solving the time-dependent Schroedinger equations under high applied voltage. Both capped and open-ended geometries are considered. Carbon atoms bridging two walls are assumed to exist in the open-ended double-wall tubes. The outer wall screens the external field very effectively in general. Double-wall nanotubes with at least one metallic wall are shown to produce more stable emission current than single-wall nanotubes. Implications to the performance of the actual field emission display developed in the industry are discussed.

5:00 PM
The Effect of Structural Change and Ni Doping on Hydrogen Storage Properties of Carbon Nanotubes: *Jai-Young Lee*[1]; Hyun-Seok Kim[1]; Kyu-Sung Han[1]; Min-Sang Song[1]; Min-Sik Park[1]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. & Engrg., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

Hydrogen storage properties in carbon nanotubes(CNTs) were investigated from the view points of not only physical hydrogen molecules adsorption in nano-hole but also chemical hydrogen adsorption on graphite surface. CNTs with closed or open capped were studied through hydrogen thermal desorption technique equipped with gas chromatograph. The precise analysis on thermal desorption spectra on CNTs with closed structure showed that hydrogen gas was released at three major temperature ranges such as 100-230K, 290-350K, and 600-625K, where the evolved hydrogen amount were about 1.65wt%, about 0.64wt%, and about 0.03wt%, respectively. However, in case of

open CNTs, the evolution peak around 290-350K was highly developed (about 1.9wt%). And Ni-doping(dispersion) effect on hydrogen storage properties were investigated comparatively. The metal nanoparticles were homogeneously dispersed using incipient wetness impregnation method. Hydrogen desorption spectra of MWNTs with Ni nanoparticles showed that about 2.8wt% of hydrogen was released in the range of 340-520K.

Arsenic Metallurgy: Fundamentals & Applications: Removal of Arsenic and its Precipitation from Process Streams II

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Tuesday PM Room: 2014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Shijie Wang, Phelps Dodge Refining Corp, El Paso, TX 79915 USA; Larry G. Twidwell, Montana Tech of University of Montana, Sch. of Mines & Engrg., Butte, MT 59701 USA

2:30 PM
Biological Removal of Arsenic from Tailings Pond Water at Canadian Mine: *Tina Maniatis*[1]; Tim Pickett[1]; [1]Applied Biosciences, PO 520518, 265 Crossroads Sq., Salt Lake City, UT 84152 USA

Applied Biosciences has developed a biological technology for removal of arsenic, nitrate, selenium, and other metals from mining and industrial waste waters. The ABMet® technology was implemented at a closed mine in Canada for removing arsenic from tailings pond water. The system included six bioreactors that began treating water in the spring of 2004. Design criteria incorporated a maximum flow of 567 L/min (150 gallons per minute) and temperatures ranging from 10°C to 15°C. Influent arsenic concentrations range from 0.5 mg/L to 1 mg/L. The ABMet® technology consistently removes arsenic to below detection limits (0.02 mg/L). Data from the bench scale testing, and the full scale system will be presented, as well as regulatory requirements site specific and challenges.

2:55 PM
Arsenic Removal from Contaminated Waters: *Jack Adams*[1]; Ximena Diaz[1]; Jan D. Miller[1]; Terrence Chatwin[2]; [1]University of Utah, Dept. Metallurgl. Engrg., 1335 S. 1460 E., Salt Lake City, UT 84112-0114 USA; [2]University of Utah, Utah Engrg. Experiment Sta., 1495 East 100 S., Salt Lake City, UT 84112 USA

Arsenic is a contaminant at 781 of 1,430 sites identified on the National Priorities List and in mining and mineral processing wastewaters, smelter wastes, and sites for manufacture of semi-conductors, petroleum products, wood preservatives, animal feed additives, and herbicides. Arsenic affects ~4,100 municipal water systems nationwide and is difficult to treat to 10 ppb levels. Adsorptive media can remove up to 99% of arsenic from drinking water at costs for POU/POE applications of $0.20/0.02/gal. Full-scale microbial arsenic removal/stabilization treatment costs of $0.10/1,000 gal have been demonstrated for mining and ground waters to 2ppb levels. Processes using magnetically activated carbon and bacteria/biopolymers; separately and combined (BIOMAC) have been demonstrated to treat high levels of Arsenic III and V to 2 ppb under wide water chemistry ranges. Additional BIOMAC benefits include other heavy metal removal, such as lead, copper, zinc, fluoride, selenium, and improvement in taste and odor.

3:20 PM
Arsenic Removal by Electrocoagulation Technology in the Comarca Lagunera Mexico: *Jose R. Parga*[1]; David L. Cocke[2]; Jesus L. Valenzuela[3]; Ventura Valverde[1]; Jewel A. Gomes[2]; Mehmet Kesmez[2] Hector A. Moreno-Casillas[2]; [1]Instituto Tecnologico de Saltillo, Matls. Sci., V. Carranza 2400, Saltillo, Coahuila 25280 Mexico; [2]Lamar University, Gill Chair of Chmst. & Cheml. Engrg., PO Box 10022, Beaumont, TX 77710 USA; [3]University of Sonora, Chmst., Zona Centro, Hermosillo Sonora, Sonora Mexico

In some parts of the Comarca Lagunera with population of about 2.5 million people, situated in the central part of northern México, chronic arsenic poisoning is endemic and severe adverse effects attributed to Arsenic exposure have been reported. There are several methods available for removal of arsenic from well water in large conventional treatment plants, however a very promising electrochemical treatment technique that does not require the addition of chemicals or regeneration is Electrocoagulation (EC). The EC operating conditions are highly dependent on the chemistry of the aqueous medium, conductivity and pH. In this study, Powder X-ray Diffraction, Scanning Electron Microscope, and Transmission Mössbauer Spectroscopy were used to characterize the solid products. Finally the results of this study suggest that the presence of maghemite and magnetite particles can be used to remove arsenic (III) and arsenic (V) in a field pilot plant scale study yielded 99% removal from groundwater.

3:45 PM
Removal of Arsenic from Soda Matte Process Solutions-An Overview: *Shijie Wang*[1]; [1]Phelps Dodge Refining Corp, 850 Hawkins Blvd., El Paso, TX 79915 USA

Soda Mattes are normally generated by treating the rough copper dross with sodium to produce a high grade copper matte which is actually a mixture of sodium sulfide, lead metal, and lead and copper sulfides. Since about 60% of the arsenic is solubilized during the hydrometallurgical treatment, the solution from the process has to be treated for arsenic removal. In this paper, the treatment scheme is evaluated. A mass balance of the process is presented. The characteristics of the process control and the efficiency of arsenic removal are discussed.

Automotive Alloys 2005: Session I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Tuesday PM Room: 2006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Lexington, KY 40511 USA; Zhong Li, Commonwealth Aluminum, Lexington, KY 40511 USA

2:00 PM
Application of 5182 for Automotive Parts: *Gyan Jha*[1]; W. Yin[2]; [1]Arco Aluminum, 9960 Corp. Campus Ste. 3000, Louisville, KY 40223 USA; [2]SECAT, 1505 Bull Lea Rd., Lexington, KY 40511 USA

Aluminum alloys have been increasingly used in the automotive industry due to their excellent properties,high strength and corrosion resistance. The formability of these alloys is critical for automotive applications. The Forming Limit Diagram (FLD) allows for an opportunity to determine process limitations in sheet metal forming and is used as an input to aid in the analysis of the stamping characteristices of sheet metal materials. This paper will investigate the application of 5182 sheet for Automotive applications by analyzing the of FEA part simulations that have utilized the FLD for 5182.

2:25 PM
Deformation-Induced Surface Roughening in 6022-T4 Al Sheets: *Y. S. Choi*[1]; H. R. Piehler[2]; A. D. Rollett[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15213 USA

The surface roughening behavior of 6022-T4 Al sheets deformed in plane-strain tension was thoroughly studied using multi-scale approaches. Various experimental techniques and analytical tools were used to characterize surface roughening and its evolution. The results showed the strong anisotropy in development of the characteristic roughening patterns, and their interrelations among different scales of the observation windows. The roughening patterns undesirable from an engineering aspect were identified. The through-thickness roughening behavior was also investigated, and the results showed a symmetry in roughening pattern between top and bottom surfaces, depending on the pulling direction, the size of the observation window and the level of the strain. Various efforts were made to clarify causes of surface roughening, particularly focusing on the correlation between the orientation texture and the surface roughness, and some new and useful results will be presented.

2:50 PM
Microstructure and Property Inhomogeneity of Cast Aluminum Wheels: *Robert Shang*[1]; Naiyi Li[2]; William J. Altenhof[1]; Henry Hu[1]; [1]University of Windsor, Mechl., Auto. & Matls. Engrg., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Com-

pany, Rsch. & Advd. Engrg., 2101 Village Rd., Dearborn, MI 48124 USA

The automotive industry has been attracted to the application of aluminum in road wheels, as aluminum wheels with reduced unsprung weight offer better vehicle handling and drivability. In an effort to better understand engineering functionality and performance of aluminum wheels, a study on structure inhomogeneity of cast A356 aluminum wheels has been carried out. Samples for mechanical properties and microstructure analysis were sectioned from various locations of the aluminum wheels. The microstructures have been investigated by means of optical metallography and scanning electron microscopy (SEM). Eutectic phase and intermetallic compounds are identified using energy dispersive X-ray analysis (EDS). The microstructure analysis reveals the dependency of the dendrite arm spacing (DAS) on the section thickness of the sampling regions. Tensile properties were obtained from specimens extracted from the hub, spoke and rim of the wheels. The results indicate that the tensile strength, and in particular, elongation for the sampling locations vary considerably. The property inhomogeneity is primarily attributed to inhomogeneous microstructure in the wheels resulting from the nonuniform cooling occurred during solidification.

3:15 PM

Microstructure Evolution During the Annealing of Cold Rolled AA6111: *Johnson Go*[1]; Matthias Militzer[1]; Warren J. Poole[1]; Mary A. Wells[1]; [1]University of British Columbia, Ctr. for Metallurgl. Process Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The heat-treatable Al-Mg-Si-Cu alloy AA6111 is one of the main aluminum choices for automobile sheet skin due to its excellent combination of paint bake hardening response and high formability. In the commercial processing for this alloy, fine and closely spaced precipitates that developed during hot rolling and subsequent coiling processes can retard the evolution of recrystallized microstructure by suppressing the movement of high angle boundaries. On the other hand, large primary Fe-containing constituent particles can promote recrystallization via particle-stimulated-nucleation (PSN). In an effort to clarify the interactions between these microstructural processes, a series of isothermal annealing experiments have been conducted on cold rolled AA6111 with systematically varied precipitation states. The evolution of microstructure, both in terms of the recrystallized grain size distribution and spatial distribution of second phases are characterized using a variety of experimental techniques including EBSD grain mapping, electron channelling contrast in SEM, optical microscopy and resistivity measurements. The results indicate that the recrystallization kinetics are severely retarded irrespective of prior aging conditions.

3:40 PM

Particle Cracking Damage Nucleation in Wrought Aluminum Alloys: *Y. Mao*[1]; Joel Harris[1]; A. M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

Damage nucleation in numerous ductile Al-alloys involves cracking of brittle inclusions; subsequent void growth around the cracked inclusions and the void coalescence leads to the global fracture. Consequently, it is of interest to quantify particle/inclusion cracking in wrought Al-alloys. In the past, such quantitative microstructure characterization involved manual detection of the cracked particles using optical or scanning electron microscopy, which is quite laborious and inefficient. We present a novel digital image analysis technique for automatic detection of cracked particles and cracks and subsequent measurements of their geometric attributes such as number density, and size, shape, and orientation distributions. The technique is presented through its applications to characterization of cracking of Fe-rich intermetallic inclusions in 7075, 6061, and 5086 Al-alloys.

4:05 PM

Use of EBSD to Quantify the Microstructural Damage in Aluminum Alloys Under Monotonic Loading: *Joseph A. Querin*[1]; J. A. Schneider[1]; Mark F. Horstemeyer[1]; [1]Mississippi State University, Dept. of Mechl. Engrg., Mississippi State, MS 39762 USA

A high degree of success has been achieved in the use of damage modeling for predicting the durability and reliability of cast components. Cast components typically have a high void volume fraction due to porosity. Under the stress triaxiality conditions present in thick walls, the porosity voids grow and coalease. Implementation of this damage modeling technique for predicting durability and reliability in rolled sheet components presents new challenges. Rather than damage accumulation originating at porosity voids, damage accumulation originates within shear bands in deformed aluminum sheet metal. The initial texture and evolving texture in sheet metal affects the shear band formation and is an important factor in material failure. How this texture evolves will be affected by grain size and grain boundary orientations. Electron backscattered diffraction EBSD is useful for characterizing texture, but it may also be used to characterize shear banding and damage progression. Results from our experiments will illustrate how the microstructural changes in monotonically loaded tensile specimens can provide the damage evolution data necessary for predictive modeling of aluminum sheet metal components.

4:30 PM

Microstructure, Texture and Mechanical Properties of Continuous Cast AA5083 and 5182 Aluminum Alloys: *Tony Zhai*[1]; Jin Li[2]; Xiyu Wen[3]; [1]University of Kentucky, Dept. of Cheml. & Matls. Engrg., 177 Anderson Hall, Lexington, KY 40506 USA; [2]Beijing Jiaotong University, Dept. of Civil & Environmental Engrg., Beijing China; [3]University of Kentucky, Ctr. for Al Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA

The recrystallization textures of cold-rolled continuous cast AA 5083 and 5182 aluminum alloys with and without prior heat treatment were investigated by X-ray diffraction. The results showed that the prior heat treatment strongly affected the recrystallization texture of CC AA 5083 aluminum alloy. The recrystallization texture of CC AA 5083 aluminum alloy without prior heat treatment was characterized by a major strong {113}<110> component and a minor R component, while the recrystallization texture of the alloy with prior heat treatment consisted of a weak R component. The tensile properties and their anisotropy of the 5083 and 5182 alloys were also studied.

4:55 PM

Development of Twin Roll Cast AA 6016 for Automotive Applications: *Murat Dundar*[1]; *Ozgul Keles*[1]; Necmi Dogan[1]; Bilal Kerti[1]; Gerhard Anger[2]; [1]Assan Aluminum, R&D, E5 Karayolu 32. km. Tuzla, Istanbul 81700 Turkey; [2]AMAG Rolling GmbH, PO Box 32, Ranshofen A-5282 Austria

Aluminum alloys employed for inner and outer panel applications of automotive industry necessitate alternative solutions to implement some improvements on mechanical properties and formability as well as a cost efficient production. Continuous interest to twin roll casting technology, in recent years, originates from cost effective nature of the casting technique and some unique features of the materials cast in comparison to conventional casting. Very high solidification rate at the roll gap introduces unique micro structural features that will further contribute mechanical properties and formability performance, with the application of appropriate downstream processing route. In present study, detailed micro structural characterization studies were conducted on the as-cast samples of twin roll cast AA6016. Casting parameters were optimized to tailor micro structural features at the surface and through the thickness. Further on results for specific automotive applications as car body sheet and structural sheet will be discussed.

5:20 PM

Processing of AA6082 Profiles for Automotive Applications: *Yucel Birol*[1]; Osman Cakir[1]; Tanya Aycan Baser[1]; Fahri Alageyik[1]; Erdogan Bengu[2]; [1]Marmara Research Center, Gebze, Kocaeli 41470 Turkey; [2]Asas Aluminum, Akyazi, Adapazari Turkey

Among several alloys within the Al-Mg-Si system, AA6082 alloy is regarded as a higher strength alloy and is used for sections requiring tensile strengths exceeding 300 MPa in the automotive industries. This alloy needs to be processed in an optimum way in order to meet the everincreasing market demand for improved performance particularly with regard to their strength. Factors such as alloy chemistry, microstructure or the extrusion process parameters have a big impact on the strength of the final product. For high strength extruded profiles, processing must assure either fine recrystallized grains or a uniform unrecrystallized structure as intermediate partially recrystalized structures with coarse grains particularly near the profile surface, are detrimental to strength, toughness, formability and weldability of these products. The present work was carried out to investigate the effect of Mn content, the quench rate, the solution temperature, the quenching practice and the storage time before artificial ageing on the final properties of 6082 hollow cylindirical profiles for antivibrational elements used in automotive industry where high strength and dimensional stability is critical.

5:40 PM

Properties of Al-Si-Mg Automobile Body Sheet by Twin Roll Casting: *Hideyuki Uto*[1]; Hiroki Esaki[1]; Yoshio Watanabe[1]; Tadashi Minoda[1]; Kaoru Ueda[1]; Kazuhisa Shibue[1]; [1]Sumitomo Light Metal Ind., Ltd., R&D Ctr., 1-12 Chitose 3, Minato-ku, Nagoya, Aichi Prefecture 4558670 Japan

Properties of Al-1.0%Si-0.4%Mg alloy sheets for automobile body by twin roll casting (TRC sheets) were investigated. Additionally me-

chanical, forming and corrosion properties of the Al-Si-Mg alloy containing ferrous impurity from 0.15 to 1% were estimated in order to develop the material recycling technology for body panels. After cold rolling and heat treatment the mechanical properties of TRC sheets are almost the same as that of the sheets by DC casting (DC sheets). At deep-drawing test the forming height of TRC sheets is almost the same. However, both the forming height at stretching test and the bendability for hemming of TRC sheets is lower than that of DC sheets. Moreover, the surface quality of TRC final products was evaluated. As a result it is clarified ripple mark affects filiform corrosion sensitivity.

Beta Titanium Alloys of the 00's: Composites and Processing

Sponsored by: Structural Materials Division, SMD-Titanium Committee

Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Tuesday PM — Room: Salon 10/11
February 15, 2005 — Location: San Francisco Marriott

Session Chairs: John Fanning, TIMET, Henderson, NV 89009 USA; James G. Ferrero, The Perryman Company, Houston, PA 15342 USA

2:00 PM
Mechanical Properties of Laser Deposited Beta Titanium Alloys with TiB Reinforcements: *Davion Hill*[1]; Rajarshi Banerjee[1]; Daniel Huber[1]; Peter C. Collins[1]; Jaimie Tiley[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

In recent years there has been considerable interest in b Ti alloys due to their wide applicability in a number of different areas, for example, in aircraft structural components. In order to increase the stiffness and strength of these alloys, reinforcements such as transition metal borides are considered promising. Therefore, there is substantial interest in the development of metal-matrix composites consisting of borides dispersed in a b Ti matrix. Laser engineered net-shaping (LENS®) is a directed laser deposition process which uses a powder feedstock and appears to be a promising technology for the processing of these metal-matrix composites. Two types of TiB reinforced composites have been studied, one based on the alloy Ti-5Al-5V-5Mo-3Cr-0.5Fe (TIMETAL 5553) and the other based on the alloy TIMETAL 21S with TiB reinforcements. Using a feedstock consisting of a blend of pre-alloyed TIMETAL 5553 (or TIMETAL 21S) and elemental boron powders, these composites have been deposited in a single step via the LENS® process. These as-deposited composites exhibit a refined homogeneous distribution of TiB precipitates within the alloy matrix, a consequence of the rapid solidification rates inherent to the LENS® process. The microstructure of the LENS® deposited composites has been investigated in detail using SEM and TEM based techniques. The room temperature tensile properties and wear resistance of these composites is currently being investigated and will be presented in this paper.

2:25 PM
Processing, Microstructure, and Properties of Beta Ti Alloys Modified with Boron: *Sesh Tamirisa*[1]; Radhakrishna B. Bhat[2]; Jay Tiley[3]; Dan B. Miracle[3]; [1]Ohio University/AFRL, AFRL/MLLMD, 2230 Tenth St., Ste 1, Wright-Patterson AFB, OH 45433-7817 USA; [2]UES Inc., Matls. Procg., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433-7817 USA

The development of next generation beta Ti alloys is expected to involve very attractive combinations of strength-toughness-fatigue resistance at large cross sections, improved and affordable processibility, and higher elevated temperature capability. This paper describes the development of beta Ti alloys modified with small boron additions to achieve the above goals. Two important aerospace alloys, Ti-15Mo-2.6Nb-3Al-0.2Si (TIMETAL21S) and Ti-5Al-5V-5Mo-3Cr (Ti-5553) with various boron levels are considered. The objective of this study is to establish the influence of boron addition on the microstructural evolution and mechanical properties. Ingots of 70 mm diameter and 500 mm length are cast using induction skull melting. Detailed microstructural characterization and tensile property evaluation are conducted. Effect of boron addition on the microstructural stability and properties in the as-cast condition will be presented. The implications of boron addition on the microstructural evolution and affordability of subsequent processing will be discussed.

2:50 PM
Effects of Carbon on the Creep Behavior of Burn-Resistant â-Phase-Based Ti-35V-15Cr-xC Alloys: *Fusheng Sun*[1]; E. J. Lavernia[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

There is a great deal of interest in developing burn-resistant titanium alloys for aerospace applications. The objective of this research is to study the effects of carbon on the creep resistance in burn-resistant beta-phase-based titanium alloys. The creep behavior of the Ti-35V-15Cr and Ti-35V-15Cr-0.2C alloys at 500-580°C within a stress range of 200-300MPa was studied. The deformation behavior was characterized using a JEM-2010 transmission electron microscope (TEM). The experimental results showed that the creep resistance of Ti-35V-15Cr-0.2C is considerably improved by incorporation of Ti2C particulates into the matrix. The creep deformation of the Ti-35V-15Cr-xC (x=0, 0.2%) alloys was dominated by a dislocation-controlled creep-process. The improved creep resistance of the Ti-35V-15Cr-0.2C is attributed to both solid solution strengthening of carbon and dispersion strengthening of Ti2C particles by inhibiting dislocation motion in the matrix.

3:15 PM
New Class of Heat-Resistant Titanium Alloys Produced by MEM Technology: *Yaroslav Yurievich Kompan*[1]; Valentin Nikolaevich Moiseev[2]; [1]E.O. Paton Electric Welding Institute, Dept. of Magnetic Hydrodynamics of Electroslag Processes, 11, Bozhenko Str., Kyiv 03680 Ukraine; [2]All Russian Institute of Aircraft Materials, 17, Radio Str., Moscow 107005 Russia

Presentation covers the first steps towards creation of new class of heat-resistant alloys and investigates the new class of alloys based on alpha- and beta-solid solutions with addition of intermetallic second phase particles. These alloys are characterized by the intermetallic dispersion strengthening and/or a "rigid frame" of eutectoid forming phase in a soft matrix. These alloys have been achieved by the method of magnetically controlled electroslag melting (MEM) which assures purified, refined heterogeneous (in terms of the eutectoid-forming phase) cast structure. The MEM technology allows to produce round ingots of 300 mm plus diameter and rectangular ingots of similar mass. Average strength of the alloys at room temperature and at UTS=1400-1600MPa and operating temperatures are estimated 700-800°C respectively.

3:40 PM Break

3:55 PM
Effect of Deformation Conditions on Grain Size and Microstructure Homogeneity of Beta-Rich Titanium Alloys: *G. A. Salishchev*[1]; R. M. Galeyev[1]; O. R. Valiakhmetov[1]; M. F.X. Gigliotti[2]; B. P. Bewlay[2]; C. U. Hardwicke[2]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin Str., Ufa 450001 Russia; [2]GE Research, Niskayuna, NY USA

The control of grain size, shape, and microstructural homogeneity is of great importance for secondary processing and to achieve optimal mechanical properties in a final forging. We will present and discuss the development of processing windows in both beta and alpha/beta fields for forming homogeneous microstructures with grain sizes down to sub-microcrystalline. Alloys studied include stable beta, metastable beta and high-beta alpha/beta alloys. The effects of deformation temperature, strain and strain rate, initial microstructures and alloy-content on final grain size and microstructural homogeneity will be reported. In one example of forming a homogeneous microstructure in a beta-rich alloy, isothermal multiple-step forging was used to produce billets with low ultrasonic noise. Heat treatment response, and room and elevated temperature mechanical properties of these billets will be discussed.

4:20 PM
Anisotropy of Mechanical Properties in High-Strength Titanium Beta-Alloys: *Orest M. Ivasishin*[1]; Pavlo E. Markovsky[1]; Sergiy A. Kotrechko[1]; Vadim I. Bondarchuk[1]; Stephen P. Fox[2]; [1]Institute for Metal Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [2]TIMET Henderson Laboratory, Henderson, NV USA

Thermomechanical processing of beta alloys substantially influences their microstructure and crystallographic texture, thus resulting

in anisotropy of mechanical properties. Usually, final high-strength conditions in beta alloys are formed by solution treatment followed by aging (STA), in which alloy is solution treated below beta-transus temperature. This means that high strength material exhibits anisotropy undesired for the many applications. It was shown in the present paper, taking TIMETAL-LCB beta alloy as an example, that application of special heat treatment consisting of solid solutioning by rapid continuous heating into single phase beta field and subsequent aging allows to reduce an influence of primary thermomechanical processing and attain more isotropic structural condition as compared to conventionally heat treated material. Tensile mechanical properties were determined in rolling and transverse directions. In the last case a unique testing technique was developed. Obtained results are discussed in terms of high-strength material resistance to brittle fracture. Since nucleation cracks open up in distinct crystallographic planes, their orientational distribution was determined what allowed to predict influence of texture on anisotropy of mechanical properties.

4:45 PM
Comparative Study of the Mechanical Properties of High-Strength Beta-Titanium Alloys: *Orest M. Ivasishin*[1]; Yuriy V. Matviychuk[1]; Pavlo E. Markovsky[1]; S. L. Semiatin[2]; [1]Institute for Metal Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright-Patterson AFB, OH 45433-7817 USA

The mechanical properties of three commercial beta-titanium alloys (TIMETAL-LCB, Ti-15-3, and VT22) were compared in the STA-condition following thermomechanical processing comprising beta-solid solutioning, cold deformation (CD), continuous rapid heating, and final aging. The peak temperature during the rapid heating step was chosen to obtain either a polygonized or recrystallized condition and thereby to control the distribution of residual deformation defects, which, in turn, influenced the dispersion and distribution of alpha-phase precipitates in the final microstructure. It was established that a good balance of high strength (in excess of 1600 MPa) and reasonable ductility could be obtained if a fine-grained microstructure with a beta-grain size of ~10 mm was formed by recrystallization. The development of such a fine grain size enabled a reduction in the aging temperature and thus increased the strength while maintaining ductility within the desired limit. In this respect, the properties could be varied to a great extent by varying the rate of heating to the aging temperature. On the other hand, aging of the polygonized condition also led to a strength level of approximately 1600 MPa but with generally lower ductility than in the fine-grained recrystallized condition. Another distinctive feature of the polygonized condition was that the sensitivity of properties to the rate of heating to the aging temperature was not very strong.

5:10 PM
Rapid Beta Solution Treated Timetal® LCB Alloy Mechanical Properties and Texture: *Allan J. Hutt*[1]; Orest M. Ivasishin[2]; [1]Perryman Company, 213 Vandale Dr., Houston, PA 15342 USA; [2]National Academy of Sciences, Inst. for Metal Physics, 36, Vernadsky str., Kiev 252142 Ukraine

Timetal® LCB Alloy has demonstrated a good balance of strength, ductility, and fatigue properties for automotive spring applications in the alpha-beta solution treated and aged condition. Mechanical properties and texture were evaluated in the rapid beta solution treated and aged condition. Conventional beta treatments yield material with low ductility. However, if the beta grain size can be kept below 10 microns by using controlled heating rates, beta solution treatment can improve strength and ductility from alpha-beta solution treated material.

Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM — Room: 3009
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Robert O. Ritchie, University of California, Matls. Sci. & Engrg., Berkley, CA 94720-1760 USA; Paul Calvert, University of Massachusetts, Dept. of Textile Scis., N. Dartmouth, MA 02747 USA

2:00 PM Invited
Biomimetic Bonelike Composites and Novel Bioactive Glass Coatings: *Antoni P. Tomsia*[1]; [1]Lawrence Berkeley Laboratory, Matls. Scis. Div., One Cyclotron Rd., MS 62R0203, Berkeley, CA 94720 USA

This presentation focuses on the design and synthesis of artificial bone-like composite materials and novel bioactive glass coatings for metallic implants using natural bone as a guide. Hydrogel polymers that possess anionic groups suitably positioned for nucleating biominerals, and therefore mimic the natural function of the collagen-phosphoprotein matrix in bone, were designed to direct template-driven biomineralization in a microscopically controlled fashion. Different mineralization techniques were developed to enable the formation of hydrogel-calcium phosphate composites with either porous microstructures suitable for potential tissue penetration or excellent polymer-mineral interfacial affinity that is desirable for bone mimics with enhanced biocompatibility and interfacial mechanical properties. Novel osteophilic glass coatings with thermal expansion properties matching with those of their metallic bone implant substrates such as Ti or Co/Cr, and therefore preventing interfacial cracking during the enameling process, were also developed.

2:30 PM Invited
Effect of Bone Density on the Damping Coefficients of Dental Implants: An In Vitro Method: Lindsey R. VanSchoiack[1]; Jean C. Wu[3]; *James C. Earthman*[1]; [1]University of California, Biomed. Engrg., 916 Engrg. Tower, Irvine, CA 92697 USA; [3]Newport Coast Oral Facial Institute, 360 San Miguel Dr., Ste. 204, Newport Beach, CA 92660-7828 USA

For normal healthy teeth the percussive energy generated by mastication is attenuated by the periodontal ligament at the healthy bone-natural tooth interface, this is not the case when dental implants are present. Previous studies have looked at the mechanical damping in dental implants, however the studies have never examined the effect of bone density at the time of implant placement in conjunction with quantitative measures of mechanical energy dissipation. Utilizing a series of artificial foam bone models which vary in density and structure, we have tested the mechanical energy dissipation of several different dental implant makes and models as a function of simulated bone density. Our hypothesis is that the Periometer, a percussion probe system designed to measure local damping capacity, can assess the quality of the underlying support structure. We also hypothesize that the Periometer will be able to differentiate between implant model geometries within the same support structures.

3:00 PM Invited
Titanium Alloys for Biological Applications: *H. J. Rack*[1]; [1]Clemson University, Sch. of Matls. Sci. & Engrg., 213 Olin Hall, Clemson, SC 29634-0971 USA

Titanium alloys, because of their excellent mechanical, physical and biological performance are finding ever-increasing application in biomedical devices. This presentation will review and illustrate the history of titanium alloy use for medical devices, their current status, future opportunities and obstacles for expanded application. Illustrations will be given for commercial purity titanium, alpha-beta and metastable beta alloys, intermetallic compounds and metal matrix composites.

3:30 PM Break

3:45 PM
Surface Treatments of Ti Dental Implant: *Carlos Nelson Elias*[1]; Rodrigo Prioli[2]; Guilhermo Solorzano[2]; Ricardo Bathomarco[2]; [1]Military Institute of Engineering, Pr Gen. Tiburcio 80, Rio de Janeiro Brazil; [2]Pontifícia Universidade Católica, Caixa Postal 38071, Rio de Janeiro, RJ Brazil

The surface of commercial unalloyed titanium used in dental implants was analyzed by atomic force microscopy. The morphology, roughness and surface area of the samples submitted to anodizing, shot-peening, acid etching and a combination of them, were compared. The results show that surface treatments strongly influence the dental implant physical and chemical properties. An analysis of the length dependence of the implant surface roughness shows that, for scan sizes larger than 50mm, the average surface roughness is independent of the scanning length and that all the surface treatments lead to surfaces with similar roughness in the range of 435 nm ± 49 nm. It is shown that the implant surface energy is sensitive to the titanium surface area. As the area increases there is a decrease in the surface contact angle.

4:05 PM
Surface Characteristic of Metallic Biomaterials and Their Effect on Interaction with Osteoblast Cells: L. Bren[1]; *J. Drelich*[1]; L. English[1]; J. Fogarty[1]; N. Istephanous[2]; R. Policoro[1]; A. Zsidi[1]; [1]Michigan Tech, Matls. Sci. & Engrg., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Medtronic USA, Inc., Matls. & Bioscies. Ctr., 710 Medtronic Pkwy., Minneapolis, MN 55432 USA

The application of bone screws was examined with the goal of learning which surface properties of 316L stainless steel and Ti-6Al-4V metal alloy elicit the desired osteoblast cell response. Mice osteoblast cells were cultured on seven different surface treatments and material combinations and the resulting cell proliferation, differentiation and morphologies were studied. The research results indicate on the positive effects of nano-scaled roughness, random surface topography, and reduced surface potential on improved cell attachment and differentiation. It was also found that the alkaline phosphatase activity increased with increasing surface tension and increasing electron-acceptor surface tension parameter of the implant material. These results suggest that the formation of surface hydroxyl groups with acidic character of metallic biomaterial gives rise to enhanced differentiation of osteoblast cells.

4:25 PM
Comparison of the Fatigue and Tensile Behavior and Biocompatibility of Ti-17Al-33Nb(at.%) and Ti-22Al-28Nb(at.%) with Ti-6Al-4V(wt.%): *C. J. Cowen*[1]; M. Niinomi[2]; T. Akahori[2]; L. M. Flick[3]; K. A. Rider[3]; C. J. Boehlert[1]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., Engrg. Bldg., E. Lansing, MI 48824-1226 USA; [2]Toyohashi University of Technology, Production Sys. Engrg., Toyahashi 441-8580 Japan; [3]Alfred University, Alfred, NY 14802 USA

In this work the fatigue (R=0.1, 10Hz, max. stress between 60-95% of the UTS) and tensile properties of Ti-17Al-33Nb(at.%) and Ti-22Al-28Nb(at.%) were compared to those for Ti-6Al-4V(wt.%), which is commonly used for biomedical implant applications. The S-N behavior illustrated that both the as-processed and heat-treated Ti-Al-Nb alloys exhibited fatigue lives equal to or greater than those for Ti-6Al-4V. The RT tensile strength and elastic modulus of the Ti-Al-Nb alloys were also comparable. Biocompatibility experiments, measuring the resorption of mouse calvarial tissue in response to the Ti-Al-Nb particles, indicated that no significant reaction occurs between the particles and living cells, resulting in a midline sagittal suture area comparable to that of untreated mice. These experiments demonstrated that there is no difference in the biological response to either Ti-17Al-33Nb or Ti-22Al-28Nb. The results of this study suggest Ti-Al-Nb alloys have potential for biomedical implant applications. This work was partially supported by the National Science Foundation (DMR 0134789).

4:45 PM
Design of an Optimum Acetabular Cup Prosthesis: *Kamran Tabeshfar*[1]; [1]Bournemouth University, Design Grp., 12 Christchurch Rd., Bournemouth BH13NA UK

With the current trend of performing Total Hip Replacements (THR) on younger patients, incurring an increasing number of revisions, prolonging the life of a THR is of paramount importance and one of the main goals of research in the field. Younger patients not only require increased longevity from their prosthesis but also increased performance so as to be able to undertake more strenuous everyday activities, such as sports. Historically the main objective of THR was to relieve pain and increase quality of life in the elderly; generally, these were not intended for young active patients. Previous research has proposed that the acetabular cup design has far more impact on long-term survival of the THR than the femoral component. Optimising the acetabular cup prosthesis produces a highly complex problem where many of the individual design factors have massive impact on the system. A main aim is to develop a material or combination of materials to optimise the stress distribution in the system without sacrificing the service life of the THR. Of the 800,000 hip replacements carried out annually, many of the current acetabular cups have some form of polyethylene bearing surface, but polyethylene wear debris is seen as a major contributing factor to bone resorbtion and hence prosthesis loosening. Changes in stress values, even caused by initial primary fixation during the operation, can result in stresses being transferred in an unrealistic manner. The effect is that the bone grows to differing thickness and strengths (remodelling). For optimisation of the acetabular cup, the properties of the natural hip must be retained by minimising both remodelling and bone resorbtion. This paper describes research and development related to a novel composite acetabular cup prostheses with a ceramic on ceramic bearing surface that should last longer and perform better, thus reducing the necessity for costly and debilitating revisions later on in life. In addition, the use of the proposed novel anatomically orientated mechanical testing methods gave fast, cheap results and could also allow further research into fatigue failure of alumina bearing couples in THR.

5:05 PM
Fabrication of Novel TiZr Alloy Foams for Biomedical Application: *Cui'e Wen*[1]; Peter Damian Hodgson[1]; Yasuo Yamada[2]; [1]Deakin University, Sch. of Engrg. & Tech., Pigdons Rd., Geelong, VIC 3217 Australia; [2]National Institute of Advanced Industrial Science and Technology, Matls. Rsch. Inst. for Sustainable Dvlp., 2266-98 Anagahora, Simoshidami, Moriyama-ku, Nagoya, Aichi 463-8560 Japan

Bone injuries and failures often require the inception of implant biomaterials. Research in this area is receiving increased attention globally. In particular, porous metals are attractive due to its unique physical, mechanical, and new bone tissue ingrowth properties. In the present study, TiZr alloy powders were prepared by a mechanical alloying process. Novel TiZr alloy foams with porosities of about 80% were fabricated by a powder metallurgical process using the MA powders. The TiZr alloy foams display an interconnected porous structure resembling bone and the pore size ranges from 200 μm to 500 μm. The TiZr alloy foams exhibit an appropriate strength to withstand physiologic loading and a low elastic modulus very close to bone.

5:25 PM
A Novel Combinatorial Approach to the Development of Beta Titanium Alloys for Orthopaedic Implants: *Soumya Nag*[1]; Rajarshi Banerjee[1]; John Stechschulte[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Cornell University, Matls. Sci. & Engrg., Ithaca, NY USA

Orthopeadic alloys for implant applications typically require a combination of different properties such as excellent biocompatibility with no adverse tissue reactions, excellent corrosion resistance in the body fluid where it will be used, high mechanical strength and fatigue resistance, low modulus, and good wear resistance. Since the beta phase in Ti alloys exhibits a significantly lower modulus than the alpha phase, and the beta alloys also satisfy most of the other requirements for an ideal orthopaedic alloy, there is a thrust towards the development lower modulus beta-Ti alloys which retain a single beta phase microstructure on rapidly cooling from high temperatures. While a number of biocompatible beta-Ti alloys have been reported in recent literature, there is still a tremendous scope for improvement in terms of alloy design via optimization of alloy composition and thermo-mechanical treatments. A novel combinatorial approach has been developed for rapid assessment of the microstructure and properties of such alloys and consequently aid in the development of new orthopaedic alloys as well as in the maturation of existing ones. This approach is based on the use of directed laser deposition to rapidly process compositionally graded alloys, administer appropriate heat-treatments to these alloys, characterize and quantify their microstructures, assess their mechanical properties, and finally build a database relating the composition-microstructure-property. This database has been used to train and test fuzzy-logic models to predict property-microtructure-composition relationships in these alloy systems. These results will be discussed in this presentation.

Bulk Metallic Glasses: Shear Banding and Deformation

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Tuesday PM Room: 3006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; Mo Li, Georgia Institute of Technology, Matls. Sci. & Engrg., Atlanta, GA 30332 USA

2:00 PM
Annealing of Shear Bands — A Nanoindentation Study of Plasticity in a Cold-Rolled Al-Based Metallic Glass: Wenhui Jiang[2]; *Michael Atzmon*[1]; [1]University of Michigan, Dept. of NERS & MSE, Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Dept. of NERS, Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA

Few studies have addressed the effect of existing shear bands on the deformation behavior of metallic glass. Serrated flow has recently been observed in nanoindentation studies of metallic glasses, and linked to shear-band formation. Using instrumented nanoindentation, we have investigated the plastic flow behavior of a cold-rolled, amorphous, $Al_{90}Fe_5Gd_5$ ribbon at various loading rates and compared it with that of undeformed, as-spun ribbon. Cold rolling to a total thickness reduction of 45.5% was performed in a large number of small steps so as to prevent sample heating. While the as-spun ribbon exhibited serrated flow and pileups around the indents, the rolled amorphous ribbon demonstrated a smooth load-displacement curve and no pileup. Furthermore, annealing of the cold-rolled ribbon below the crystallization temperature restored the serrated flow and pileups. The role of shear-band initiation in the deformation behavior will be discussed. Support provided by the National Science Foundation under Grant DMR-0314214.

2:20 PM
Experimental Observations of Shear Banding in Bulk Metallic Glasses: *J. J. Lewandowski*[1]; N. A. Stelmashenko[2]; A. L. Greer[2]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106-7204 USA; [2]University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK

Structural applications of bulk metallic glasses (BMGs) are limited by the inhomogeneous nature of their plastic deformation, leading to interest in the mechanisms of shear banding. The temperature rise associated with shear-band operation has been investigated for BMGs based on Cu, Hf or Zr, and for in-situ composites of beta phase in Zr-based BMG, by coating them with thin films of low-melting-point metals. Temperature increases have been detected in all cases by morphological changes taken to arise from melting of the coating. The increases are at least 200 K, and within the band are much higher. As indicated by melting of the coating, here is heating not only associated with final rupture, but also around subsidiary bands, extending 1 to 10 micrometres on either side of the band. No similar observations were noted on identical experiments conducted on an oxide glass or a high strength aluminum alloy. The local heating estimated from these observations on the BMGs will be compared with published observations made by different techniques and with calculations based on models of the flow mechanism.

2:40 PM
In Situ Visualization of Shear-Band Evolution During Tensile Testing of a Bulk Metallic Glass: *Mark L. Morrison*[1]; Bing Yang[1]; Peter K. Liaw[1]; C. T. Liu[2]; Raymond A. Buchanan[1]; Cecil A. Carmichael[2]; Ramon V. Leon[3]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA; [3]University of Tennessee, Dept. of Stats., Ops & Mgmt. Scis., 337 Stokely Mgmt. Ctr., Knoxville, TN 37996-0532 USA

For the first time, the evolution of multiple shear bands has been visualized in a bulk metallic glass (BMG) with infrared (IR) thermography. Surprisingly, up to 58 shear bands were observed to initiate, propagate, and arrest during a single tensile test of a $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10.0}Ti_{5.0}$ (atomic percent) BMG alloy, commonly known as Vitreloy 105. By utilizing this unique visualization technique, the length, width, location, sequence, temperature evolution, and velocity of individual shear bands were quantified. Furthermore, correlations among the variables also were investigated. Detailed statistical analyses of these parameters were conducted in order to gain insight into the process of the inhomogeneous deformation in BMG materials. The authors are grateful to the National Science Foundations, Integrative Graduate Education and Research Training (IGERT) program under grant number DGE-9989548; the Combined Research and Curriculum Development (CRCD) Training program under ZZC-9527527 and EEC-0203415 with Dr. L. Clesceri, Dr. W. Jennings, Dr. L. Goldberg, and Ms. M. Poats as the contract monitors; and the Division of Materials Science and Engineering, Department of Energy under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory (ORNL) operated by UT-Battelle, LLC.

3:00 PM
Modeling Shear Band Propagation in Bulk Metallic Glasses: *Brian J. Edwards*[1]; Kathleen Feigl[2]; Peter K. Liaw[3]; Mark Morrison[3]; Bing Yang[3]; Ray A. Buchanan[3]; [1]University of Tennessee, Cheml. Engrg., Knoxville, TN 37996 USA; [2]Michigan Tech. University, Math. Scis., Houghton, MI 49931 USA; [3]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA

Recent observations have indicated that shear bands originate and propagate in bulk metallic glasses (BMGs) under tensile loading once plastic deformation has begun. These shear bands propagate and dissipate on the order of milliseconds, as witnessed with high-speed and high-sensitivity infrared thermography. In this presentation, we present results of a study aimed at understanding the onset, propagation, and eventual dissipation of these shear bands under tensile loading in BMGs. Initial results are discussed, based on the application of a non-equilibrium thermodynamics approach to this problem, which results in a system of equations that couples the applied stress distribution within the sample with temperature and an additional vector field associated with the free volume in BMGs. This set of equations describes the shear-band formation, propagation, and dissipation within the BMGs, and gives hints concerning the origination of shear bands, their speeds of propagation, their width and length, their direction of propagation, and the magnitude of any permanent plastic deformation that occurs across them.

3:20 PM
Mesoscopic Theory and Modeling of Shear Localization in Metallic Glass: *Mo Li*[1]; Guang-Ping Zheng[2]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA; [2]Hong Kong University, Dept. of Mechl. Engrg., Hong Kong China

A mesoscopic theory for shear localization in metallic glass is proposed. Based on experimental observations and atomistic modeling results, we establish the relation between local free volume density and applied stress. This phenomenological theory predicts shear localization, shear instability and shear zone propagation. A phase-field modeling is applied based on this theory.

3:40 PM Break

4:00 PM
Shear Localization in Metallic Glass: An Atomistic Study: *Mo Li*[1]; QiKai Li[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA

Shear localization in binary metallic glass ZrNi, CuZr and PdSi are investigated using extensive molecular dynamics simulations. It is found that shear localization is a result of local instability of deformation triggered by local concentration of stress. Contrary to long-held view that shear localization is adiabatic in its origin, our work suggest that local heating and fluid-like behavior are simply a consequence of the shear instability, not the cause. Excess free volume is found in the shear zone. The speed of the shear zone propagation is estimated. Effects of various processing factors on shear zone formation are also investigated.

4:20 PM
Shear Localization and Percolation of Quasicrystalline Structure in a Simulated Model Metallic Glass: *Yunfeng Shi*[1]; Michael L. Falk[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Molecular dynamics simulations of uniaxial tension in a two-dimensional model of a metallic glass exhibit varying degrees of shear localization depending upon the process by which each of the materials was produced. The samples that were quenched most gradually show the largest degree of localization. In addition higher strain rates lead to

increased localization in the most rapidly quenched samples, while the more gradually quenched samples show the reverse strain rate dependence. This transition in localization and strain rate dependence coincides with a structural transition in the material. Gradually quenched samples have a higher percentage of atoms in quasi-crystal-like local environments. The transition in the mechanical properties coincides with the percolation of this backbone of quasi-crystal-like material. Shear localization occurs in regions where material is altered from this more stable structure to a fully amorphous structure under the effect of plastic deformation.

4:40 PM
Micromechanics and Macroscopic Mechanical Behavior of In Situ Formed Metallic Glass Matrix Composites: *Ryan T. Ott*[1]; Frederic Sansoz[2]; Jean-Francois Molinari[3]; Jon Almer[4]; Todd C. Hufnagel[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 MD Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]University of Vermont, Dept. of Mechl. Engrg., Burlington, VT 05405 USA; [3]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA; [4]Argonne National Laboratory, Advd. Photon Source, Argonne, IL 60439 USA

We have examined the mechanical behavior of in situ formed composites consisting of crystalline Ta particles in a Zr-based amorphous matrix. We evaluate yield criteria for the composite based on yield stresses in tension v. compression, as well as shear band angles. We also examine the micromechanics of deformation of the composite alloys using in situ high-energy x-ray scattering. Yielding of the Ta-rich particles creates a misfit strain in the surrounding glass matrix; this creates a stress concentration that leads to initiation of shear bands. As a result, the glass yields locally around the Ta-rich particles prior to global yielding of the amorphous matrix. Combining the results from the uniaxial compression tests, the in situ strain measurements, and finite element models of deformation, we discuss the relationship between the micromechanics and the macroscopic mechanical behavior of the composite alloys.

5:00 PM
Evolution of Multiple Shear Band Patterns Beneath an Indentation in Bulk Metallic Glasses Using a Bonded Interface Technique: *Ghatu Subhash*[1]; Hongwen Zhang[1]; Laszlo J. Kecskes[2]; Robert J. Dowding[2]; [1]Michigan Technological University, ME-EM Dept., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Army Research Laboratory, AMSRD-ARL-WM-MB, Aberdeen Proving Ground, Aberdeen, MD 21005 USA

Although shear band formation has been known to be the dominant deformation mechanism in bulk metallic glasses (BMGs), many indentation studies have revealed only a few shear bands that cannot account for the large ductility observed in BMGs. Therefore a bonded interface technique has been developed to observe the shear bands beneath the indentation. Vickers indentations were performed along this interface. At small indentation loads (<50g) numerous semicircular shear bands surrounding the indentation were observed. The spacing between these shear bands decreases with increasing distance from the indentation. At moderate loads (100-300g), largely spaced secondary shear bands emanate from each of the deformed surfaces of the indentation. At higher loads (>300g), a third set of shear bands radiate from the tip of the indentation and cut across the first two sets. The evolution of shear band patterns and the effects of indenter orientation with respect to the interface will be presented.

Cast Shop Technology: Melt Treatment: Degassing and Filtration

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: John Courtenay, MQP Ltd, Casthouse Tech., Solihull, W. Midlands B93 9EW UK

2:00 PM
An Analysis of Impeller Performance Based on an Air-Water Model: *J. J.J. Chen*[1]; M. Nilmani[2]; [1]University of Auckland, Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand; [2]NCS Associates (Australia) Pty Ltd., 101/180, Flinders Ln., Melbourne Australia

While there are many designs of degassing impellers available in the market, it had been reported that the majority of these impellers gave similar degassing efficiency under supplier recommended operating conditions based on tests conducted in full scale water model experiments. This paper shows that the degassing performance of these impellers can be described by a mass transfer equation which considers the power input based on a consideration of the Power number versus Reynolds number relationship in the absence of gas injection and under fully turbulent conditions, and the actual power input in the gassed condition. For one of the impellers used, the Power number versus Reynolds relationship has been measured. For the other impellers, the Power number at large Reynolds number was assumed. The oxygen desorption data for all four commercial impellers were found to be well-represented by one single equation.

2:25 PM
Ultrasonic Degassing of Molten Aluminum Under Reduced Pressure: *Hanbing Xu*[1]; Xiaogang Jian[1]; Thomas T. Meek[1]; Qingyou Han[2]; [1]University of Tennessee, Matls. Scis. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Matls. Procg. Grp., Oak Ridge, TN 37831-6083 USA

Ultrasonic degassing, an environmentally clean and cheap technique, is an efficient way of degassing in a static volume melt. However, there is a steady-state hydrogen concentration below which the effect of ultrasonic vibration will be absent. The limit of ultrasonic degassing will occur within a few minutes of ultrasonic vibration, regardless of the initial hydrogen concentration in the melt. Vacuum degassing, a practical technique used in Europe, also has been tested a beneficial and clean method in producing high quality products. Combination of these two techniques will help to lower the limit in ultrasonic degassing and promote the efficiency in vacuum degassing. An experimental device which combines the vacuum degassing and ultrasonic degassing has been built in Oak Ridge National Laboratory recently. Parametric studies have been carried out to investigate the efficacy of the ultrasonic degassing of molten Aluminum alloy under reduced pressure. This article reports the experimental results and discusses the mechanism of degassing in molten Aluminum under the influence of vacuum and ultrasonic vibrations.

2:50 PM
Characterization of Droplets Produced by Bubbles Bursting: *Autumn Fjeld*[1]; James W. Evans[1]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg., MS 1760, Berkely, CA 94720 USA

A limitation on the metal throughput of a gas fluxing unit is posed by splashing and spraying of metal droplets as the gas throughput is increased to keep pace. In an investigation at Berkeley funded by DOE (OIT DE-FC07-01ID14192) and Alcoa, droplets ejected as a result of bubble rupture at the free surface of a molten metal are examined via high speed digital photography and image tracking software. Experiments are carried out in a custom built, glass walled vessel designed for observation of splashing and spraying at the surface of a low melting temperature alloy. A controlled bubble release system permits variation in bubble size, release of single or multiple bubbles, or a continuous stream of bubbles. The effects of melt depth in the vessel, oxide formation at the melt surface, and the presence of a second liquid on droplet behaviour are also examined. Droplet numbers, velocities, trajectories and size distributions are determined for the aforementioned conditions with image analysis software.

3:15 PM
Upstream Fluid Flow Particle Removal: *Laurens Katgerman*[1]; Jan Zuidema[2]; [1]Delft University, NIMR, Rotterdamseweg 137, Delft 2628AL The Netherlands; [2]Corus RD&T, P.O.Box 10000, IJmuiden 1970 CA The Netherlands

The occurrence of oxides films and inclusions can give major problems during aluminium casting and processing. The level of oxides and inclusions in standard DC casting practices is controlled with the use of in-line filter boxes and degassing units. However, during the transfer from these metal treatment systems to the casting station pick up of inclusions and oxides can occur. This can be either caused by the varying efficiency of the metal treatment system and/or uncontrolled fluctuations in the metal flow. The aim of this paper is to assess quantitatively some of the upstream flow modifiers and their effect on melt cleanliness. The first method is placing baffles in the launder system. The location of these baffles is an important parameter in modifying the fluid flow behaviour. Both the controlled filling of the mould as well as entrapment of inclusions can be achieved. The second method is modifying the flow pattern in such a way that separation of

fluid from the particles occurs. This is done by employing a cyclone. Results of numerical simulations of fluid flow with discrete particles of different mass and size distribution are given as well as experimental results obtained from a water model.

3:40 PM Break

3:50 PM
XC Filter - A New Hybrid Filter for Improved Filtration Efficiency: *Stephen Instone*[1]; Mark Badowski[1]; Wolfgang Schneider[1]; [1]Hydro Aluminium Deutschland, CC Casting, Alloys & Recycling, Georg-von-Boeselager-Str. 21, Bonn, NRW 53117 Germany

The quality of the aluminium rolling ingot is increasingly important to fulfil requirements of the various aluminium rolling and subsequent downstream finishing process chains. The presence of non-metallic inclusions is particularly important for products such as aluminium foil and lithographic sheet. Inclusions in the size range 10-40μm can pass through current in-line filters and lead to limitations in the processing capabilities of the ingot. A new design for a filter unit, named the XC filter, for the treatment of liquid aluminium has been tested and found to give superior filtration efficiency particularly in the important size range mentioned above. The XC Filter, combines elements of ceramic foam filtration (CFF) and deep bed filtration (DBF) to overcome limitations of the established technologies. The performance of the XC Filter, measured during industrial casting trials using LiMCA, is presented. Comparison is made to performance data of CFF filters obtained under similar conditions.

4:15 PM
AlF_3 as an Aluminium Filter Medium: *Harald Görner*[1]; Martin Syvertsen[2]; Eivind J. Øvrelid[2]; Thorvald Abel Engh[1]; [1]NTNU, Matls. Tech., Alfred Getz Vei 2, Trondheim N-7491 Norway; [2]SINTEF Materials and Chemistry, Metall., Alfred Getz Vei 2, Trondheim N-7465 Norway

An AlF_3 "active" filter is studied for removal of dissolved impurities (alkali, Mg, Sr, H) in addition to suspended particles in Al melts. Industrially, AlF_3 is injected as a refining powder. However, then the yield for removal of Na is low. A bed filter containing AlF_3 as a filter media should improve the kinetics by increasing both contact time and area. A lab setup has been built consisting of a AlF_3 filter and an Al_2O_3 dummy. This makes it possible to quantify the fraction of sodium removal due to the "active" filter media. Pure Al melts with Na additions were filtered. Disc samples were taken to determine the Na concentration at the filter inlet and the two outlets. Removal is from 55% to 73% for the conventional filter and from 80% to 91% for the active filter. The data are employed to derive a kinetic model for such filters.

4:40 PM
An Ultrasonic Sensor for the Continuous, On-Line Monitoring of the Cleanliness of Liquid Aluminum: M. Kurban[2]; P. H. Mountford[2]; *Ian D. Sommerville*[1]; N. D.G. Mountford[2]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., Toronto, Ontario M5S 3E4 Canada; [2]Metal Vision Manufacturing, Toronto, Ontario Canada

Over the past twenty years or so, an ultrasonic sensor has been developed which is capable of providing continuous, on-line monitoring of the inclusion content of liquid aluminum, where these inclusions can be either non-metallic or intermetallic. By inserting air cooled guide rods into the liquid metal, ultrasound can be passed into the liquid and the return signals processed to provide three pieces of information: a measurement of the average particle size of inclusions counted, a histogram display of the size distribution of the largest particles counted and an attenuation resulting from the scattering of the ultrasound by the particles suspended in the melt. This scattering is caused by all the particles present, but mainly by the very large number of particles which are too small to count because they are too small to cause discrete reflections. This technique has been used to detect particles in the size range 15-100 μm, and in principle is capable of detecting sizes up to 400 μm, which is regarded as the lower threshold for visual detection by the naked eye. Thus, it is capable of monitoring the acceptability of metal for a wide range of applications from the very high quality required in continuously cast metal to the less demanding requirements typical of foundry and die casting operations. The technique is virtually non-obtrusive, and is capable of monitoring significant proportions of the total metal volume. In the case of metal flowing in a launder, the proportion monitored is a linear function of the flowrate, and could amount to as much as 20 or even 25% of the total metal volume.

5:05 PM
Cleanliness of Aluminum and Steel: A Comparison of Assessment Methods: T. A. Utigard[1]; *Ian D. Sommerville*[1]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., Toronto, Ontario M5S 3E4 Canada

The quality and fitness for service of metals is determined in large part by casting them at the appropriate temperature, with the correct chemical composition and sufficiently clean that the inclusion content does not seriously compromise their performance in the intended application. In comparison with the methods available for determination of temperature and chemical composition, those available until recently for the assessment of cleanliness have been very unsophisticated and unsatisfactory. Many different methods have been devised for assessment of the cleanliness of both metals, most of them applicable to the solid metal, which is "after the fact" and allows only very limited opportunity for any required corrective measures to be taken. The fact that so many methods exist is clear evidence that none has been generally accepted as providing an accurate measure of the incidence of inclusions. Because there are so many methods, they can not be discussed in detail, but rather they are briefly reviewed in an attempt to highlight their strengths and weaknesses. Some emphasis is placed on those techniques which can be applied on-line to liquid metals, and which therefore provide a greater opportunity for corrective measures to be taken when an unsatisfactory level of cleanliness is detected.

5:30 PM
Filtration Efficiency and Melt Cleanness Evaluation Using LAIS Sampling at Valesul Aluminio S.A.: *Alexandre Vianna da Silva*[1]; Alberto Maia[1]; Luiz C.B. Martins[2]; Ramon Duque[2]; [1]Valesul Aluminio S.A., Estrada Aterrado do Leme, 1225, Santa Cruz, Rio de Janeiro, RJ 23579-900 Brazil; [2]SELEE Corporation, 700 Shepherd St., Hendersonville, NC 28792 USA

Valesul Aluminio castshop is a premier supplier of extrusion billets to the Brazilian and Overseas market. Billets are cast using an in-line MINT degasser and SELEE filtration system. A designed experiment was conducted to determine the influence on melt quality and filtration efficiency of chlorine content (3% and 0), and ceramic foam filter type (17-inch 20 ppi and 20-inch 30 ppi). Melt cleanliness was measured using a Liquid Aluminum Inclusion Sampler (LAIS), a total of 12 drops were tested. Oxide-type inclusions included spinels and oxides, grain refiner was present in all three sampling locations. Final melt quality was 0.010 mm^2/kg total less grain refiner, being similar for 3% and 0 chlorine. Melt cleanness improvement across both filter types were statistically significant, with filtration efficiencies of 51.9 % for the 17-inch 20 ppi filter and 77.5% for the 20-inch 30 ppi filter.

Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials - II

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday PM Room: 2010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Arun M. Gokhale, Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30318 USA

2:00 PM
An Investigation of Mechanical Behavior and Damage Evolution of Ti-6Al-4V-Al3Ti Metal Intermetallic Laminate (MIL) Composites: *Tiezheng Li*[1]; Marc A. Meyers[1]; Kenneth S. Vecchio[1]; Engene A. Olevsky[2]; [1]University of California, Mechl. & Aeros. Engrg., 9500 Gilman Dr., UCSD-0411, La Jolla, CA 92093-0411 USA; [2]San Diego State University, Mechl. Engrg., 5500 Campanile Dr., San Diego, CA 92182-1323 USA

The mechanical performance of Ti-6-4-Al3Ti metal-intermetallic laminate (MIL) composites synthesized by a reactive foil sintering technique was evaluated. Crack morphology of untested Ti-6-4-Al3Ti MIL composites has been characterized by optical microscopy. Mechanical tests were performed on Ti-Al3Ti metal-intermetallic laminate (MIL) composites and pure Al3Ti, and the principal mechanisms of damage initiation and accumulation were identified experimentally. The elastic properties and anisotropy of the laminates were calculated and successfully compared with Resonant Ultrasonic Spectroscopy

(RUS) measurements. The effect of residual stress on the fracture toughness of the Ti-6-4-Al3Ti MIL composites was analyzed.

2:25 PM
Stereological Unfolding to Determine the Three-Dimensional Bivariate Size and Shape Distribution of TiB Whiskers in Ti-6Al-4V-2.9B: *Scott Lieberman*[1]; David Mebane[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA

Metal matrix composite (MMC) consisting of TiB whiskers distributed in a Ti-6Al-4V alloy matrix was produced using blended elemental powder metallurgy and compaction of Ti-6Al-4V-2.9B. In such composites an important aspect of microstructure representation is the size and shape distribution of the whiskers. The desired three-dimensional bivariate size and shape distribution of TiB whiskers has been estimated from measurements performed on a two-dimensional metallographic section by using a recently developed stereological unfolding procedure. The distribution confirmed that the orientation of TiB whiskers is random in three dimensions, and that whisker growth is anisotropic with an increase in aspect ratio with increased length.

2:50 PM
Characterization of Microstructural Evolution in AlMgSi(Cu) Alloys Using Calorimetry Methods: *Shahrzad Esmaeili*[1]; Warren J. Poole[2]; David J. Lloyd[3]; [1]University of Waterloo, Dept. of Mechl. Engrg., 200 Univ. Ave. W., Waterloo, Ontario N2L 3G1 Canada; [2]University of British Columbia, Dept. of Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [3]Alcan International Limited, Kingston R&D Ctr., PO Box 8400, Kingston, Ontario K7L 5L9 Canada

Until recently, the evaluation of microstructural evolution during age hardening in the automotive AlMgSi(Cu) alloys was considered a difficult task due to a combination of factors including the type and low concentration of solutes, the complicated precipitation sequence and small size of precipitates. Two analytical methods based on isothermal calorimetry and differential scanning calorimetry have been recently developed which provide practical tools for the characterization of microstructural evolution in the aging regimes where precipitation hardening occurs in these alloys. The present work summarizes the basic procedures, as well as the combined analytical and modeling approach to determine the relative volume fraction and size of precipitates in commercially relevant aging treatments.

3:15 PM
Acoustic Properties of Ni3Si Intermetallic Materials: Shih-Jeh Wu[1]; *Shian-Ching Jason Jang*[2]; Chen-Ming Kuo[1]; Dong-Yih Lin[2]; Chen-Ching Ting[1]; [1]I-Shou University, Dept. of Mechl. Engrg., 1, Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan; [2]I-Shou University, Dept. of Matls. Sci. & Engrg., 1, Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Intermetallic compounds are defined as a mixture in specific proportion of two metallic elements that form a periodic crystalline structure different from those of the original elements. Ni3Si intermetallic is a popular structural material in aerospace engines and acidulous environment due to its good mechanical integrity and acid resistance at elevated temperatures. The mechanical properties were investigated by ultrasonic method non-invasively from different forming processes at different temperatures. The Young's and shear moduli can be calculated easily from their relationship with longitudinal and shear acoustic speeds. Experimental data also showed the ultrasonic attenuation is quite different from that of ordinary metals or alloys. The much stronger attenuation may be related to the special L12 structure of Ni3Si and indicate the microstructure change at different compounds.

3:40 PM Break

3:50 PM
Study of Long-Term Durability of Aluminum Lithium Alloys: *Hajer Mahmoud Awatta*[1]; Ali Merati[2]; Marko Yanishevsky[2]; Vivier Lefebvre[3]; [1]Carleton University, Dept. of Mechl. & Aeros. Engrg., 1125 Col. By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]National Research Council Canada, Inst. for Aeros. Rsch., 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada; [3]Department of National Defence, NRC/IAR, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

Weight reduction of aerospace vehicles is best achieved by decreasing the density of aircraft structural materials. However, the development of Al-Li alloys has faced several technical problems, which include high anisotropy of mechanical strength, low stress corrosion threshold and low ductility. Long-term performance, in particular fatigue and corrosion behaviours, are also concerns that need to be further studied. Al-Li alloys have been used in the new fleets of the search and rescue helicopters to replace most of the conventional aircraft alloys such as 7075-T6 and 2024-T3 due to their low density and high elastic modulus. The present work investigates Al-Li alloy durability issues to provide proactive support to the Canadian Forces.

4:15 PM
Rheological Study of the Foundry 356 Al-Alloy Prepared by a New Innovated SSM Process: *Omid Lashkari*[1]; Reza Ghomashchi[1]; [1]University of Quebec, Applied Scis., DSA, UQAC, 555, Blvd. de l'Univ., Chicoutimi, Quebec G7H 2B1 Canada

SEED (Swirled Enthalpy Equilibrium Device) is established itself as a new technology amongst the SSM processes, which uses swirling as agitator parameter of the molten metal and dendrite breaking operant within mushy zone state. Swirling causes primary particle evolution during solidification. Swirling changes the dendritic microstructure and creates different primary phase size and distribution. In the current research, variation of microstructure due to swirling intensity has been studied by an image analysis system and a simple parallel plate test machine, i.e. rheological test machine. Microstructure and viscosity of the new SSM 356 Al-alloy has been proportioned by the rheological tests.

4:40 PM
Quantitative Linking of Ceramic Matrix Composite Microstructure with Impedance Spectra: *Rosario A. Gerhardt*[1]; David S. Mebane[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

The orientation and length-radius distributions of silicon carbide whiskers embedded in alumina are shown to correlate with the composite's resistivity. The resistivity was measured using impedance spectroscopy. Stereological unfolding techniques are applied to SEM images in order to measure the size-orientation distributions. A mathematically defined, stochastic microstructural simulation provides a link between the measured microstructure and percolating volume fraction. Percolating volume fraction, as measured through the simulation, is shown to be proportional to high-frequency features in the impedance spectra for Al2O3-SiCw composites of 10 and 20% SiCw by volume. These results suggest a general method for linking composite microstructure to AC electrical response of many different composite systems.

5:05 PM
Characterization of Heat-Treatable Aluminum Matrix Composite from Recycled Aluminum Cans Alloys Containing Boron as Reinforcers: *Ely X. Colon*[1]; O. Marcelo Suarez[2]; [1]University of Puerto Rico, Mechl. Engrg. Dept., PO Box 9045, Mayagüez, PR 00681-9045 USA; [2]University of Puerto Rico, Gen. Engrg. Dept., PO Box 9044, Mayagüez, PR 00681-9044 USA

The present project proposes the fabrication of a novel series of cast aluminum matrix composites (AMC), using aluminum-boron master alloys and aluminum-magnesium from recycled aluminum cans. This new AMC series do not present several deleterious effects found in currently available cast AMC containing ceramic reinforcements such as SiC, graphite, and Al2O3 particulates. The resulting material has low cost of production and low reactivity between reinforcing particles and matrix. Mg is incorporated as an alloying element to improve ageing characteristics of the composite that boosts hardness in these precipitation-hardened Al-Mg-B composites. The Mg-containing alloy is of the AA 5XXX series provided by clean used beverage cans. The present investigation represents a baseline of research for further investigations that are being conducted on Al-Cu-B, Al-B-Cu-Mg and Al-B-Mg composites. Microhardness measurements, Scanning Electron Microscope (SEM) and X-Ray Diffraction are being used to test and characterize the resulting composites.

Computational Aspects of Mechanical Properties of Materials: Meso-Scale and Continuum Modeling

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Tuesday PM Room: 3012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael J. Mills, Ohio State University, Matls. Sci. & Engrg., Columbus, OH 43210 USA; Nik Chawla, Arizona State University, Dept. of Cheml. & Matls. Engrg., Tempe, AZ 85287 USA

TUESDAY PM

2:00 PM
A Data-Mining Approach for the Design of Optimized Polycrystalline Materials: *Veeraraghavan Sundararaghavan*[2]; Nicholas J. Zabaras[1]; [1]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 188 Frank H. T. Rhodes Hall, Ithaca, NY 14853-3801 USA; [2]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 169 Frank H. T. Rhodes Hall, Ithaca, NY 14853-3801 USA

A data-driven reduced-order optimization procedure is presented for the design of deformation process sequences for controlling texture and texture-dependent properties. The inverse problem of identifying processing stages leading to a desired texture is initially solved using an unsupervised data-mining methodology based on the x-means algorithm. The hierarchical classifier matches the lower-order ODF features in the form of pole density functions of important orientation fibers and associates the desired texture to a class of pre-existing textures within a database. Texture classes in the database are affiliated with processing information, hence, enabling identification of multiple process paths that lead to a desired texture. The process parameters are fine-tuned using a gradient optimization algorithm driven by continuum sensitivity analysis of texture evolution. An adaptive reduced-order model based on proper orthogonal decomposition is employed wherein the texture modes corresponding to the intermediate stages of the design process are adaptively selected from the database. Further, the database continuously improves during the optimization problem through addition of new, unknown data sets, which would be useful during future optimization runs.

2:20 PM
Predicion of Crystallographic Texture Evolution and Anisotropic Stress-Strain Curves in α-Titanium Using a Taylor-Type Crystal Plasticity Model: *Xianping Wu*[1]; Surya R. Kalidindi[1]; Roger D. Doherty[1]; [1]Drexel University, Dept. of Matls. Sci. & Engrg., 3141 Chestnut St., Philadelphia, PA 19104 USA

A Taylor-type polycrystalline model has been developed to simulate the evolution of crystallographic texture during large deformation processing of α-Titanium at room temperature. In addition to slip, deformation twinning has been incorporated as an additional plastic deformation mode in this model. Each activated family of twins in a grain is addressed as a quasi-independent grain that will undergo further deformation and rotation independently, but its volume fraction and orientation are updated at the end of each time step. Slip inside twinning has been allowed in this computation. New hardening functions for both slip and twinning are provided in this model. Good prediction of the overall stress-strain response and texture evolution in a number of different deformation modes were obtained using this newly developed crystal plasticity model.

2:40 PM
3D Characterisation and Modelling of an Aluminium Matrix Composite Using X-Ray Microtomography: *Ian G. Watson*[1]; Peter D. Lee[1]; Richard J. Dashwood[1]; [1]Imperial College London, Dept. of Matls., Prince Consort Rd., S. Kensington, London SW7 2BP UK

The high strength and stiffness of particulate-reinforced aluminium matrix composites is well documented. The size, morphology, orientation and distribution of the reinforcement particles are critical parameters in achieving optimum stiffness, strength and fatigue resistance. Understanding the interaction of the reinforcement particles with the matrix alloy is a dual-scale problem. On the micro-scale, individual reinforcement particles coagulate during slow cooling to form clusters. The local strengthening effect of these clusters depends on their packing density and morphology. On the macro-scale, the three dimensional (3D) distribution of the clusters dictates the overall efficiency of load transfer. Traditionally, two-dimensional (2D) visualisation techniques such as optical and scanning electron microscopy have been used to characterise composite microstructures. However, the extrapolation of 3D morphologies from 2D images is only valid if the material is not textured and the features are equiaxed and of uniform size. In this investigation x-ray microtomography (XRMT) was used to characterise the complex three dimensional morphology and distribution of TiB2 clusters in an aluminium alloy matrix. A finite element model was used to estimate the local stiffening and strengthening effects of an individual cluster. This representative volume element (RVE) was combined with the 3D cluster distribution obtained via XRMT in a macroscale model to determine the elastic-plastic response of the MMC as a whole.

3:00 PM
Simulation of Mechanical Behaviors of Die-Cast Mg Alloy Based on Three-Dimensional Porosity and Finite Element Method: *Soon Gi Lee*[1]; Arun Sreeranganathan[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30332-0245 USA

Porosity is one of the most important microstructural attributes of die-cast Mg-alloys. Several features of porosity such as the amount, geometry, and locations have dominant influence on the mechanical behavior of die-cast Mg alloys. Therefore, it is of interest to incorporate quantitative description of actual three-dimensional porosity in micro-mechanical analysis of the cast alloys. In this contribution, a montage-based serial sectioning technique has been applied for constructing actual three-dimensional porosity, and the finite element (FE)-based simulations have been performed on the 3D microstructure of a high-pressure die-cast Mg-alloy containing "real" pores to reveal the relationships between the distributions of local stresses and strains and size, orientation, and spatial arrangement of the porosity.

3:20 PM
Rapid Evaluation of Mechanics of Defects in Casting: *Yi Cheung Lok*[1]; Adam Clayton Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Cambridge, MA 02139 USA

The presence of porosity has always been a major concern in castings. While the detrimental effect of porosity on the mechanical properties of casting is well established, their presence in varying amount throughout the casting can often be tolerated. Numerical simulations on the stress state of a component can provide an insight into the optimal tolerance level and configuration of defects. The simulation is done using the Boundary Element Method (BEM) because it only requires surface meshing and thus allows for geometrical changes without complete mesh regeneration. This enables a rapid solution regeneration for various interior defect configurations because the inverted matrix corresponding to the outer part surface is reusable for subsequent simulations. Result of the simulation might help foundry engineer design castings with lower a rejection rate.

3:40 PM Break

3:50 PM
Variability of Fatigue Life in Two-Phase Alloys: *Kwai S. Chan*[1]; Yi-Der Lee[1]; Michael P. Enright[1]; [1]Southwest Research Institute, Matls. Engrg. Dept., 6220 Culebra Rd., San Antonio, TX 78238 USA

Experimental evidence indicates that a large variability in fatigue life can exist in two-phase alloys at or near the fatigue limit. This variability is generally accompanied by a transition of fatigue crack initiation site from surface grains to internal grains. To better understand these experimental observations, theoretical models have been developed to compute fatigue life variations of two-phase alloys resulting from three possible scenarios: (1) hard and soft phases in the two-phase microstructure, (2) planar slip versus cell-forming grains, and (3) crack initiation versus crack growth. The theoretical results are utilized to elucidate the effects of microstructure, slip morphology, and grain location on fatigue life variability in two-phase alloys, using Ti-6Al-4V as an illustration. Work supported by AFOSR MEANS Program through Contract No. F49620-01-1-0547, Dr. Craig S. Hartley, Program Manager.

4:10 PM
Microstructure-Based Modeling of Crack Growth in Particle Reinforced Metal Matrix Composites: *Adarsh Ayyar*[1]; Jason Williams[2]; Nik Chawla[2]; [1]Arizona State University, Dept. of Mechl. & Aeros. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287 USA; [2]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287 USA

Crack growth in SiC particle reinforced Al matrix composites is significantly influenced by the size, distribution, and morphology of the SiC particles. Reinforcement particle clustering also influences the mechanical behavior of metal matrix composites. Hence, to accurately model the crack growth in such a system it is important that the complex microstructure of the particles be taken into account and not simplified by circles or ellipses. In this paper, the effects of particle morphology and distribution (homogenous and clustered) on crack growth have been studied using the finite element method. The degree of particle clustering in aluminum/silicon-carbide composites was quantified by the coefficient of variance in the mean near-neighbor particle spacing, and cluster size distributions using an image analysis technique. The clustering analysis correlated well with the observed microstructures. Two dimensional linear elastic fracture mechanics principles were used to propagate the crack and obtain the local stress intensity values, and to obtain an understanding of the local stress state. Predictions from this analysis correlated well with experimental observations of crack growth in these systems.

4:30 PM
The Effect of Colony Orientation on Deformation Behavior and Slip Transmission During Hot Working of Ti-6Al-4V Single-Colony Samples: *A. A. Salem*[1]; S. L. Semiatin[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The deformation behavior of individual alpha/beta colonies of Ti-6Al-4V has been established under hot working conditions. Constant strain-rate uniaxial compression tests were conducted on samples cut from single-colony crystals that were grown using a float zone technique. Each sample was oriented for slip along a different prismatic slip system in the alpha-phase. The mechanical behavior exhibited a strong dependence on colony orientation. The apparent anisotropy in the critical resolved shear stress was explained based on the burgers orientation relationship between the alpha (hcp) and beta (bcc) phases of the colonies and hence the orientations of alpha slip directions relative to those in the beta phase.

4:50 PM
Prediction of Creep Behavior of Ti-6Al-4V on the Basis of Time Dependent Models Involving Microstructural Parameters and Mechanical Properties: *Dhriti Bhattacharyya*[1]; Sujoy Kar[1]; John J. Schirra[2]; Michael Savage[2]; Walter W. Milligan[3]; Hamish L. Fraser[1]; Michael J. Mills[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Pratt & Whitney, Matls. & Processes Dvlp., Structural Alloys; [3]Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI USA

The creep behavior of titanium alloys is an important factor in the life expectancy of crucial service parts in aircraft engines. Room temp creep exists, and should be incorporated in designs. In this work, an attempt has been made to predict the creep behavior and mechanisms of the Ti alloy Ti-6Al-4V at room temperature. For this purpose, extensive mechanical tests, including tensile and creep tests, have been done on a set of alpha-beta-forged samples with different heat treatment parameters in order to obtain a large range of microstructures. The tensile curves have been fitted with a time-dependent viscoplastic model to obtain parameters like the strain hardening exponent n and the strain rate sensitivity m. These results, combined with measured microstructural parameters such as grain size and volume fraction of primary alpha, have been used to feed an artificial neural network model. This model is subsequently used to predict the creep behavior of Ti-6Al-4V with arbitrary heat treatment schedules and microstructural parameters. These results will also be discussed in light of existing knowledge of room temperature creep mechanisms in the two-phase titanium alloys.

5:10 PM
Strength Predictions in Sintered "Tough Coated Hard Particle" Systems: *Ivi Smid*[1]; Rick E. Toth[2]; [1]Pennsylvania State University, Engrg. Sci. & Mech., 147 Rsch. W., Univ. Park, PA 16802-6809 USA; [2]Allomet Corporation, 509 Hahntown-Wendel Rd., N. Huntingdon, PA 15642 USA

Tough coated hard particles (TCHP) are a new microstructure designed to offer high performance levels in cutting tools, wear components and forming dies. Control of the sintered microstructure is critical to tailor and promote optimal property combinations. If preserved into the final densified microstructure, the coated hard particles will open new, unprecedented performance combinations. The TCHP concept will be introduced, and the theoretical basis for new performance combinations via various powder and coating and matrix phase combinations will be shown. Results of liquid phase sintering experiments will be presented with model TCHP powders, showing the needed balance between liquid content, thermodynamic reactions during heating, time and temperature with respect to densification, grain size and grain shape changes, and preservation of the desired coated grain microstructure. A calibration of strength predictions based on filler-particle/matrix adhesion and particle shape, and results on performance will be presented.

5:30 PM
Modeling of Grinding Dynamics Using Second Order Dynamic System: *Olga Karabelchtchikova*[1]; Simon M. Hsiang[2]; [1]Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609-2280 USA; [2]Texas Tech University, Industl. Engrg., Box 43061, Lubbock, TX 79409-3061 USA

A second order dynamic system is proposed to model residual stresses (RS) distributions under various heat treatment procedures and grinding dynamics. The study was motivated to (1) predict the magnitude of the RS and tensile peak location, and (2) establish superposition relationship in the RS distribution due to number of grinding passes. Grinding dynamics was represented as a lumped system composed of spring-mass-damper of the grinding machine and damper-stiffness of the workpiece. A nested factorial experiment of 18 conditions with 3 levels of tempering factor, 2 levels of grinding conditions and 3 multipass grinding levels was used for the model development and validation. The proposed model depicts changes of the complex experimental conditions and demonstrates a good estimation of the subsurface RS distribution. With only one parameter involved, the prediction elucidates grinding dynamics and supports a theory of the heat treatment and grinding effects on the material characteristics.

Computational Thermodynamics and Phase Transformations: Atomistic and Ab Initio Methods

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday PM — Room: 3005
February 15, 2005 — Location: Moscone West Convention Center

Session Chair: Mark T. Lusk, Colorado School of Mines, Mechl. Engrg. Prog., Golden, CO 80401 USA

2:00 PM Invited
Elucidating Kinetic Pathways for Martensitic Phase Transformations in Crystals: *Emily Ann Carter*[1]; Kyle J. Caspersen[1]; [1]Princeton University, Mechl. & Aeros. Engrg., The Engrg. Quad., Princeton, NJ 08544 USA

A method for characterizing the minimum energy path for solid-solid phase transitions is presented. The technique involves a generalization of the so-called Nudged Elastic Band method for finding the minimum energy path for atomic and molecular reaction paths. Instead of only minimizing the forces on the atoms to find critical points, we also minimize the Cauchy stress on the unit cell of the crystal, in order to find saddle points for the conversion of one phase of material to another. We can also calculate the minimum energy path in the presence of external pressure, so that pressure-induced phase transformations may be explored at the atomistic level. The method can be used with any energy representation of a material, however, we focus here on applications using first principles methods, specifically density functional theory. Applications include, but are not limited to, metallic phase transformations.

2:30 PM
A First Principles/Kinetic Monte Carlo Study of Oxygen Diffusion in YSZ-Based Oxides: *Ramanathan Krishnamurthy*[1]; Young-Gui Yoon[1]; Konstantin Kudin[1]; Roberto Car[1]; David J. Srolovitz[1]; [1]Princeton University, Princeton Inst. for the Sci. & Tech. of Matls. & Dept. of Mechl. & Aeros. Engrg., 70 Prospect Ave., Princeton, NJ 08542 USA

Understanding oxygen diffusion in yttria stabilized zirconia based oxides is of paramount importance for their effective use in

applioations. We present a multi-scale model to study oxygen diffusion in YSZ. We employ density functional theory methods to calculate activation energies for oxygen migration in different local cation environments. These results serve as input to a kinetic Monte Carlo study of oxygen diffusivity as a function of temperature and yttria content. The simulations, in agreement with experiment, show that the oxygen diffusivity attains a maximum value around 10 mol% yttria. The oxygen vacancy concentration, diffusion activation energy and correlation effects all vary differently with yttria content, producing the behavior described above when their collective effects are considered. A simple analysis of dopant-induced correlations supports this explanation. This methodology is extended to study oxygen diffusion in similar oxides. The results are discussed with regard to their implications for several applications.

2:50 PM
Kinetic Monte Carlo Study of a Mechanism for Coalescence of Precipitates: *Zugang Mao*[1]; Georges Martin[2]; *David N. Seidman*[1]; [1]Northwestern University, Matls. Sci. & Engrg., Cook Hall, 2220 Campus Dr., Evanston, IL 60208-3108 USA; [2]French Atomic Energy Commission, Paris France

The decomposition of a Ni-Al-Cr alloy at 873 K is studied by kinetic Monte Carlo (KMC) simulation. In agreement with our three-dimensional atom-probe microscope experiments we find that a significant fraction (30%) of the gamma-prime (L12) precipitates coarsen via coalescence and that the fraction coalesced is a function of aging time. The atomic scale mechanism for our observations is studied by KMC simulation. A key quantity is the monovacancy-solute binding energy, which is shown to control the presence or absence of coalescence. It is demonstrated that the mechanism involves the formation of clusters of Al and Cr solute atoms (dimers, trimers, quadramers, and pentamers) all of which are more mobile than Al or Cr monomers. The diffusive behavior of these higher order clusters is studied and it is shown that they undergo both 2D and 3D motion, and that the 2D diffusivity is greater than the 3D diffusivity.

3:10 PM
Self-Diffusion Mechanisms in Two-Dimensional Metals: Gennady Mikhailovich Poletaev[1]; *Mikhail Dmitrievich Starostenkov*[1]; Julia Vladimirovna Patzeva[1]; [1]Altai State Technical University, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia

The paper is concerned with the research of self-diffusion mechanisms in two-dimensional crystals of Ni, Al, Cu metals by the method of molecular dynamics. The packing of two-dimensional metals in a computer model was corresponded to the plane (111) of FCC lattice. Morse pair potentials and Finnis-Sinclair multipartial potentials were used in the researches. Different self-diffusion mechanisms were studied. Activation energies of the mechanisms, the contribution of every mechanism into diffusion process in the dependence on temperature were calculated. It was found, that vacancy mechanism (the vacancy was formed by Shottki method) and the mechanism of the formation and annihilation of Frenckel pairs made major contribution into self-diffusion in two-dimensional metals. Frenckel pair forms in the crystal in the result of the crossing of thermal crawdion displacements. The other diffusion mechanisms influence on self-diffusion in two-dimensional metals not so gritty.

3:30 PM
Kinetics of Precipitation in Al-Zr-Sc Alloys: From Atomic to Mesoscopic Models: *Emmanuel Clouet*[1]; Maylise Nastar[1]; Christophe Sigli[2]; [1]Service de Recherches de Metallurgie Physique, CEA/Saclay France; [2]Pechiney, Centre de Recherches de Voreppe France

Zr and Sc precipitate in aluminum alloys to form the compounds Al3Zr, Al3Sc, and Al3ZrxSc1-x which for low super-saturations of the solid solution have the L12 structure. The aim of the present study is to model at an atomic scale this kinetics of precipitation and to build a mesoscopic model based on classical nucleation theory so as to extend the field of super-saturations and annealing times that can be simulated. In this purpose, we use some ab-initio calculations and experimental data to fit an Ising model describing thermodynamics of the Al-Zr-Sc system. Kinetics of precipitation are studied using a kinetic Monte Carlo algorithm based on an atom-vacancy exchange mechanism. These simulations show that in ternary Al-Zr-Sc system Zr addition mainly affects nucleation whereas Sc addition influences nucleation as well as growth and coarsening stage. This allows us to understand experimental results showing that a Zr addition to an Al-Sc alloy leads to finer precipitates and increases their density. Structure of precipitates obtained in these simulations, a Sc rich hearth surrounded by Zr rich shelves, agrees with the one observed with 3D atom-probe.[1] A comparison of these kinetics of precipitation for Al-Zr and Al-Sc binary systems with prediction of the classical nucleation theory shows that the nucleation stage can be well reproduced by mesoscopic models as long as the short range order tendency of the system is considered.[2] This result is then used so as to build a mesoscopic model allowing to study nucleation in the ternary Al-Zr-Sc system. [1]B. Forbord, W. Lefebvre, F. Danoix, H. Hallem, and K. Marthinsen. Three dimensional atom probe investigation on the formation of Al3(Sc,Zr) dispersoids in aluminium alloys. Scripta Mater., 51:333 337, 2004. [2]E. Clouet, M. Nastar, and C. Sigli. Nucleation of Al3Zr and Al3Sc in aluminum alloys: from kinetic Monte Carlo simulations to classical theory. Phys. Rev. B, 69:064109, 2004.

3:50 PM Break

4:00 PM Invited
Phonon Transport and Scattering at the Nanoscale: *Patrick Kenneth Schelling*[1]; Simon R. Phillpot[2]; Brian Becker[1]; [1]University of Central Florida, AMPAC, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA; [2]University of Florida, Dept. of Matls. Sci. & Engrg., 162 Rhines Hall, Gainesville, FL 32611 USA

Phonon wave-packet dynamics is a powerful technique for gathering detailed information about phonon dynamics, scattering, and thermal transport. In this talk, I will present recent result for the scattering of phonons at silicon grain-boundaries, silicon nanowires, and carbon nanotubes. I will describe recent conceptual improvements that have allowed us to compute experimentally-measurable quantities in a one-shot simulation, without the need for multiple simulations previously needed to sample the entire Brillouin zone. Finally, I will show how this work can lead to the development of mesoscale models of thermal transport.

4:30 PM Invited
Adjusting the Melting Point of a Model System via Gibbs-Duhem Integration: Application to a Model of Aluminum: *Brian B. Laird*[1]; Jess B. Sturgeon[2]; [1]University of Kansas, Dept. of Chmst., Lawrence, KS 66045 USA; [2]Lawrence Livermore National Laboratory, 7000 E. Ave., Livermore, CA 94550 USA

Model interaction potentials for real materials are generally optimized with respect to experimental properties that are easily evaluated as mechanical averages (e.g., elastic constants (at T=0K), static lattice energies and liquid structure). Agreement with experiment for the non-mechanical properties, such as the melting point, is not guaranteed and such values can deviate significantly from experiment. We present a method for re-parameterizing any model interaction potential of a real material to adjust its melting temperature. This is done without significantly affecting other mechanical properties of the system. This method is an application of Gibbs-Duhem integration [D. Kofke, Mol. Phys.78, 1331 (1993)]. As a test we apply the method to an embedded atom model of aluminum [J. Mei and J.W. Davenport, Phys. Rev. B 46, 21 (1992)] for which the melting temperature for the thermodynamic limit is 826.4 ± 1.3K - somewhat below the experimental value of 933K. After re-parameterization, the melting temperature of the modified potential is found to be 931.5K ± 1.5K.

5:00 PM
The Research of Evolution of Microstructure in Two-Dimensional Crystals Cu3Au and Ni3Al at Phase Transition Order-Disorder: *Mikhail Dmitrievich Starostenkov*[1]; Gennady Mikhailovich Poletaev[1]; Natalia B. Cholodova[1]; Irina A. Demina[2]; [1]Altai State Technical University, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia; [2]East Kazakhstan State University, Ust Kamenogorsk Kazakhstan

The mechanisms of the disordering process of two-dimensional crystals Cu3Au and Ni3Al were studied by the method of molecular dynamics. The order of crystals was corresponded to L12 superstructure. The two-dimensional crystal was presented by the packing of atoms corresponding to the plane (111). The crystal block was subjected to the impulsive heating to the definite temperature. The following diffusion mechanisms of the disordering of alloys were found: crawdion, ring, vacancy and mechanism of Frenckel pairs formation (vacancies and interstitial atoms). Every mechanism began to function from the definite temperature of impulsive heating in the sequence, as it was above mentioned. The temperature of the beginning of disordering process activation was lower for the alloy Cu3Au. Frenckel pairs contributed greatly in the evolution of microstructure at high temperatures. It was shown, that the diffusion coefficient is integral characteristic, depending on all the mechanisms of diffusion reconstruction of the material.

5:20 PM
The Research of Mechanical Stability of Two-Dimensional Metallic Composites: *Mikhail Dmitrievich Starostenkov*[1]; Gennady Mikhailovich Poletaev[1]; G. V. Popova[2]; [1]Altai State Technical Univer-

sity, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia; [2]East Kazakhstan State University, Ust Kamenogorsk Kazakhstan

The stability of interphase boundaries in two-dimensional thin films of composites, consisting of the phases Ni3Al-Al and Ni3Al-Ni is studied in the dependence on deformation of hydrostatic pressure and uniaxial tension. The structure of composition material is given by different variants of packing of Ni3Al intermetallid phases and pure Al or Ni metals. Physical and physics-mechanical properties of the composite are connected with the structure of interphase boundaries. Washing process of interphase boundaries begins with the growth of temperature at the expense of different types of diffusion mechanisms. The temperature of the beginning of the transformation stage of interphase boundaries is connected with the type of metallic layer, given between the phases of Ni3Al intermetallid. Washing process begins at higher temperatures (in comparison with Al layer) in the layer, consisting of pure Ni. The deformation changes the temperature of the beginning of washing process of interphase boundary.

Converter and Fire Refining Practices: Processing Fundamentals

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee
Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Tuesday PM Room: 2016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Tony Warner, Inco Ltd, Techl. Serv., Mississauga, Ontario L5K 1Z9 Canada; Cameron Harris, H.G. Engineering Ltd., Toronto, ON M6S 3G3 Canada

2:30 PM
Copper Converting Fluxing Practise During Instantaneous High Arsenic Containing Feed Mixture in Harjavalta: *Kim Olof Fagerlund*[1]; Pekka Pyykkö[2]; Pekka A. Taskinen[1]; [1]Outokumpu Research, Kuparitie 10, Box 60, Pori FIN-28101 Finland; [2]Harjavalta Copper, Teollisuuskatu 1, Harjavalta FIN-29200 Finland

Boliden Harjavalta Oy operates a copper smelter in Finland, which is based on flash smelting together with Peirce-Smith converting of copper matte. This paper reviews a case study when a high arsenic feed mixture was successfully eliminated in order to guarantee the anode quality. Based on theoretical and practical knowledge in slag chemistry at Outokumpu Research, the CaO-fluxing practice was introduced. Description of a thermodynamic simulation using MTDATA and implementation of the results into converting practice is given. Results showed that the impurity fluctuations in raw materials could be effectively controlled by efficient utilization of different slag chemistry.

3:00 PM
Controlling the Processing Parameters Affecting the Refractory Requirements for Peirce-Smith Converters and Anode Refining Vessels: *Anthony J. Rigby*[1]; [1]RHI Canada, 4355 Fairview St., Burlington, ON L7L 2A4 Canada

Refractory linings in converters or anode furnaces used to overoxidize the blister copper will suffer rapid deterioration, due to prolonged contact with copper oxide-rich slag. Nitrogen stirring with COP KIN porous plugs and use of SEMTECH OPC in converters and anode refining vessels can considerably reduce both refractory wear and processing times. In addition, careful control of the vessel atmosphere and the use of real-time optical process control minimize both slag volume and contact time with the vessel refractory.

3:30 PM
Metallurgical Considerations for Recycling Siliceous Bearing Materials in the Mitsubishi Continuous Copper Converter: *Fumito Tanaka*[1]; [1]Mitsubishi Materials Corp., Central Rsch. Inst., 1-297 Kitabukuro-cho, Omiya-ku, Saitama 330-8508 Japan

The Mitsubishi Process is the sole pyrometallurgical process for the continuous production of blister copper from copper concentrates. Continuous converting originates from the application of the proven technology of a lime-ferrite slag (CaO-FeOX-Cu2O) on the Converting Furnace (C-furnace). Recent improvements on the C-furnace operation have included increasing the processing of recycled materials and the investigation of alternative fluxing agents. However, some minor oxides adversely affect C-furnace operation, with respect to magnetite behavior of the slag, with the most significant impact having been observed with the silica content of the slag. This paper will quantify the impact of silica on the lime-based slag, and discusses the controls that have been identified to sustain continuous converting operation.

4:00 PM Break

4:15 PM
Minor Element Control by Vacuum Calcination and Recycling of Copper Smelter Dust: *Jin Qiu*[1]; Ralph Harris[1]; [1]McGill University, Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Experiments were performed to determine the extent of minor element elimination that occurred when mixtures of copper smelter dust and copper concentrate, were calcined under vacuum. Almost complete removal of the As, Sb, Bi and Pb occurred due to the conversion of the minor element compounds of these elements into their highly volatile sulphides by reaction with sulphur from the concentrate. Heat and mass balance calculations of the slag-blow in a copper converter found that ~30 tonnes of the vacuum calcine could be used to produce 100 tonnes of copper as white metal. The use of dirty concentrate as the sulphidizing agent in the vacuum calcination means that the minor element treatment capacity of the smelter complex could be increased by 60%. Also the productivity of the converters may be increased by up to 8% due to the supply of copper values and oxygen in the calcined mixture.

4:45 PM
Control of Magnetite Formation During Slag-Making in Copper P-S Converter: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

Converter slag chemistry control has played an important role in optimizing converter operations. A thermodynamic model of copper P-S converter has been developed to predict the behavior of magnetite in the converter slag. Predictions of bath temperature, slag and matte compositions, and magnetite content in the slag have been validated by industrial data. The effects of fluxing strategy, returns and skim charges, oxygen enrichment, and temperature on the magnetite formation in the slag have been predicted and discussed. Some improvements of the industrial operations have been presented.

5:15 PM
Physicochemical Study of Reduction of Nickel Concentrate Calcine With Products of Vapor Oxidation of Fuel Oil and Solid Carbon: *A. V. Tarasov*[1]; *V. M. Paretsky*[1]; V. A. Bryukvin[1]; [1]State Research Center of Russian Federation, 13, Acad. Korolyov St., 129515, Moscow Russia

To carry out a physicochemical substantiation of a transition to "soft" modes of reduction of calcine obtained by desulfurizing roasting of nickel concentrate produced as a result of copper-nickel converter matte separation in a nickel anode production circuit, an analysis has been conducted of the macrokinetic behavior of actual calcine samples in the presence of gaseous H2-CO mixtures and solid reductant additives. Within a temperature range of 900C to 1100C the main regularities typical of the calcine reduction process with individual gases (H2, CO) also take place when using a gaseous mixture. Under the conditions of low-temperature (900C to 1000C) nickel oxide reduction with products of fuel oil conversion in the presence of coke, the role of the latter is insignificant from the technological viewpoint. As a result of the present investigations, we have obtained analytical expressions of macrokinetic parameters of the reaction interaction within the [NiO] calcine-H2-CO-C-CO2-H2O-O2-N2 system.

Extractive Metallurgy: Copper

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Tuesday PM Room: 2018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Florian Kongoli, Flogen Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2CS Canada; Arthur Morris, Thermart, San Diego, CA 92128 USA

TUESDAY PM

2:30 PM
Copper Distribution Between Reverb Furnace Smelting Products: *Natasha Mitevska*[1]; [1]RTB BOR, Copper Institute, Dept. of Metall., Zeleni bulevar 35, BOR 19210 Serbia

The results of industrial investigations of copper distribution between matte and slag, and in slag melt along the slag depth and length of the reverberatory furnace No.1 in RTB BOR, Copper Smelter and Refinery (Serbia) in 2004 are presented in this paper. Copper distribution coefficient between copper matte and slag phase is calculated, too. On the base of results obtained, the computer program for simulation of copper distribution in both melt phases in furnace is developed.

2:55 PM
Thermal Conductivity of Copper Flash Smelting Flue Dust: *Elli Vilhelmiina Nurminen*[1]; Laura Stykki[1]; Kim O. Fagerlund[2]; Pekka A. Taskinen[2]; [1]Helsinki University of Technology, Matls. Procg. & Powder Metall., PO Box 6200, Espoo 02015 Finland; [2]Outokumpu Research, PO Box 60, Pori 28101 Finland

Aggregations and build-ups in metallurgical heat recovery boilers decrease the efficiency of the heat transfer and may cause process shut downs. Transient plane source method (Hot Disk) has been applied to measure the thermal conductivities of the Copper Flash Smelting flue dusts. Thermal conductivities of copper, iron and zinc sulphates, and process flue dusts were measured. The results indicate that all the components are effective thermal insulators, having a thermal conductivity of less than 1 W/mK. Decreasing dust layer porosity has an increasing effect on thermal conductivity. Insulating layers on the boiler walls decrease greatly the heat transfer efficiency of the boiler. The gathered information can be used in the CFD-modelling of the process, and in process control.

3:20 PM
Kinetics of Copper Slag Reduction with Natural Gas: *Gabriel Riveros*[1]; Andrzej Warczok[1]; Roberto Parada[2]; [1]Universidad de Chile, Ingenieria de Minas, Av.Tupper 2069, Santiago Casilla 2777 Chile; [2]Anglo American Chile, Minera Sur Andes, Fundición Chagres, Chagres Chile

Smelting of copper concentrate in a flash smelter produces high grade matte and a slag containing from 2 to 3% Cu and 10-12% Fe3O4. The slag is processed in Teniente slag cleaning furnace by reduction with injected bunker oil, followed by slag sedimentation. The use of natural gas as a reductant is of great interest due to its price, easier management and benefits related to the decrease of negative environmental impact. The results of crucible scale simulation of slag reduction with injected air/natural gas mix showed as a major factor affecting the rate of magnetite reduction the air/natural gas ratio. The dependence of reaction rate on temperature is weaker in comparison to magnetite reduction from slag with solid carbon and carbon monoxide. Activation energy 100 kJ/mol and first order reaction regarding to magnetite content in the slag point out diffusional control in the gas towards gas/slag interface. Reaction thermal instability indicates the temperature of bubble/slag interface as a major factor determining the kinetic of magnetite reduction.

3:45 PM
Copper Flash Smelting Mass Balance Calculations Using Genetic Algorithms: *Victor Manuel Sanchez-Corrales*[1]; Jose Adolfo Valera-Gonzalez[1]; Pedro Flores-Perez[2]; Manuel Perez-Tello[1]; [1]Universidad de Sonora, Ingeniería Química y Metalurgia, Blvd. Luis Encinas y Rosales, Colonia Centro, Hermosillo, Sonora 83000 Mexico; [2]Universidad de Sonora, Dept. de Matematicas, Blvd. Luis Encinas y Rosales, Colonia Centro, Hermosillo, Sonora 83000 Mexico

In this paper, mass balance calculations using genetic algorithms for copper smelting in an Outokumpu flash furnace are presented. Based on the elemental composition of the copper concentrates being fed to the reactor, the mineralogical composition of the concentrate mixture is adjusted by means of genetic algorithms. The macroscopic mass balance equations for the species entering and leaving the furnace are solved and the compositions and flow rates of matte, slag, and the off-gas stream are computed. Good agreement between theoretical predictions and plant data was obtained in terms of matte and slag flow rates, matte grade, and copper, iron, magnetite, and silica contents in the slag. Predictions of the present method showed to be superior to those of a conventional one in which the mineralogical composition of the feed is not adjusted. Future applications of the present formulation are discussed.

4:10 PM Break

4:25 PM
Separation of Copper and Cobalt from Nickel Sulphate Solution by Organophosphorus Mixied Extractant: Chen Song[1]; Luo Yuan Hui[1]; Wang Li Jun[1]; Zhang Li[1]; Wang Rui Zhong[1]; [1]General Research Institute for Non-Ferrous Metals, Minl. Resources, Metall. & Matls., No.2 XinJie Kou Wai Da Jie St., Beijing 100088 China

During separation process of copper and cobalt from nickel sulphate solution, the effects of constitutions and compositions of mixted extractant D2EHPA and PC88-A or Cyanex272, has been studied. In one step extraction cycle, the following results are obtained:[Ni]/[Co]>4000 and [Ni]/[Cu]>40000 in nickel sulphate solution, [Co]/[Ni]>1000 and [Co]/[Cu]>30000 in cobalt chlorinate solution, [Cu]/[Ni]>10000 and [Cu]/[Co]>200 in copper sulphate solution. It was elucidated that the above-mentioned synergistic mixtures can effectively separate cobalt and copper from nickel sulphate acid solution and achieve a greater synergistic effect.

4:50 PM
Electrolysis and Reduction of Copper Slag: *Andrzej Warczok*[1]; Gabriel Riveros[1]; Martin Artigas[1]; [1]Universidad de Chile, Ingenieria de Minas, Av.Tupper 2069, Santiago Casilla 2777 Chile

Fayalite slags from smelting of copper concentrate contain from 2 to 25% of copper and from 8 to 25% of magnetite depending on the smelting process. Pyrometallurgical slag cleaning based on its reduction and sedimentation is carried out in an electric furnace or various furnaces with injection of carboneous reductant. Since common acceptance of ionic structure of liquid slags the possibilities of utilization of electrochemical phenomena in copper recovery are the point of interest. Slag electrolysis combined with chemical reduction together with electrokinetics phenomena may accelerate copper removal during slag cleaning. Results of crucible scale tests of slag electrolysis with inert and graphite electrodes showed relatively high current efficiency of copper and significant acceleration of copper removal from the slag.

Friction Stir Welding and Processing III: Process/Applications

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday PM Room: Nob Hill C/D
February 15, 2005 Location: San Francisco Marriott

Session Chair: Julie A. Christodoulou, Office of Naval Research, Arlington, VA 22217-5660 USA

2:00 PM Keynote
Friction Stir Welding - A Brief Review and Perspective for the Future: *Tracy W. Nelson*[1]; [1]Brigham Young University, Mechl. Engrg., 435 CTB, Provo, UT 84663 USA

Friction stir welding (FSW), a tremendous development in mechanical working of metals, is a proven technology for joining aluminum and other lower temperature metals. Application in aluminum and copper are growing while FSW in steels and other higher temperature

materials are nearing implementation status due to the advent of new tool materials and process understanding. In addition, Friction Stir Processing (FSP) has been gaining interest over the past few years in a variety of commercial applications. With such promising benefits, the technology of FSW&P has consumed much of the welding R&D community in just over a decade. Despite the progress in process development and technology application, the drive to implementation has left behind the research necessary for understanding essential process fundamentals. Fundamental research, in parallel with technology development, must be undertaken in order to achieve broad application of FSW&P. A researcher's perspective regarding what research and development activities are needed to evolve the technology for future applications will be given.

2:30 PM Invited
Process Dynamics of Friction Stir Welding (FSW) of 2024 Al: *J. A. Baumann*[1]; R. J. Lederich[1]; W. C. Starnes[1]; [1]The Boeing Company, PO Box 516, MC: S245-1003, St. Louis, MO 63166-0516 USA

For many FSW applications, a relatively simple delineation of the appropriate weld operating window in terms of travel rate and spindle speeds ("feeds and speeds") can be sufficient for successfully making suitable welded components. In some envisioned applications, however, such as closing-out structural components by welding skins to substructure, that substructure must react or carry the loads generated by the FSW process. In this scenario, a fuller understanding needs to be developed between the relationships of tool features, travel rate, rotation speeds and required or resultant process loads and torques, in order to properly design the substructure to withstand these forces during the welding operation, or, conversely, to tailor the FSW process to a specific structural architecture. We have explored these relationships in the lap joining of 2024 Al sheets, with top-sheet thickness ranging from 3.2 mm (0.125") thick to 9.5 mm (0.375"), using both standard, one-piece (solid shoulder and pin) FSW tools and two-piece, Retractable Pin Tools (RPT). This work has enabled us to evaluate and separate the contributions to process loads and torques arising from the pin, turning within the aluminum, and that of the shoulder, engaged with the surface of the material.

2:50 PM
Features and Configurations of FSW Equipment: *Donald J. Holman*[1]; [1]Nova-Tech Engineering, 6808 220th St. SW, Ste. 200, Mountlake Terrace, WA 98043-2187 USA

The FSW process has researched for several years. Most of the research has been dedicated to optimizing welding parameters to facilitate improved properties compared to conventional welding techniques. Extensive publications and presentations have been made regarding the material science and application benefits of friction stir welding. As the process becomes better defined, the criteria for FSW equipment design is becoming evident, yet many people are un-familiar of what constitutes a FSW machine. This presentation will outline the major systems of a FSW machine, and how they are unique compared to conventional milling machines.

3:10 PM
Friction Stir Welding of Castings: *Richard Johnson*[1]; Philip L. Threadgill[2]; William J. Kyffin[1]; [1]TWI Technology Centre (Yorkshire) Ltd, PO Box 3314, Sheffield S13 9WZ UK; [2]TWI Ltd, Friction & Forge Processes Tech. Grp., Granta Park, Great Abington, Cambridge, Cambridgeshire CB1 6AL UK

There has been considerable interest in the application of friction stir welding processes to castings in many materials, as a method of joining to other materials (cast or wrought), for repair of local defects, and for re-processing, (where the microstructure can be converted to a forged microstructure without a significant change in shape). This paper will summarise recent progress made in friction stir welding/processing of a range of aluminium alloys, copper alloys and steels, and will give examples of where these techniques could be used commercially. The paper will include process information and microstructural assessments of the welded areas.

3:30 PM Break

3:50 PM Invited
Unraveling the Material Processing Conditions for Optimizing the FSW Process: *Judy A. Schneider*[1]; Arthur C. Nunes[2]; [1]Mississippi State University, Mechl. Engrg. Dept., PO Box ME, 210 Carpenter Engrg. Bldg., Mississippi State, MS 39762 USA; [2]NASA-Marshall Space Flight Center, Matls., Processes & Mfg. of Metallic Matls., ED33, Huntsville, AL 35812 USA

In friction stir welding (FSW), a rotating threaded pin tool is inserted into a weld seam and literally stirs the edges of the seam together. This environmentally friendly, solid-state technique has been successfully used in the joining of materials that are difficult to fusion weld. To determine optimal processing parameters for producing a defect free weld, a better understanding of the resulting metal deformation flow path and velocity is required. In this study the metal flow fields are marked by the use of thin (0.001 tungsten) wires embedded in the weld seam at various locations. X-ray radiographs record the position and segmentation of the wire and are used to elucidate the flow field. Microstructures observed in a FSW cross-section in an aluminum alloy are related to their respective strain-strain rate-temperature histories along their respective flow trajectories. Two kinds of trajectories, each subjecting the weld metal to a distinct thermomechanical process and imparting a distinct microstructure, can be differentiated within the weld structure.

4:10 PM
Determining Optimum Friction Stir Weld Process Variables to Minimize Abnormal Grain Growth in Al-2195: *Srikanth Labhala*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakotra School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA

The objective of this work was to investigate the inhibition of abnormal grain growth (AGG) in friction stir welded (FSW) Al-2195. The weld process parameters of weld speed, rotation speed, and pin tool geometry were varied. Additionally, cryogenic quenching with liquid nitrogen during FSW was applied in an attempt to inhibit AGG. Liquid nitrogen was directed closely behind the pin tool on the retreating side of the weld. The test matrix contains 48 six-inch welds and includes the use of three different pin tools. Post FSW AGG was initiated by heating and holding the samples at 950°F for five minutes. The results are analyzed using plots of the extrusion ratio of the weld nugget with respect to grain size. The relationship between the AGG and the extrusion ratio of welds is summarized. Optimum processes parameters and weld conditions to inhibit AGG are suggested.

4:30 PM
FSW of Aluminum Using a Tapered and Scrolled Shoulder Tool: *Kevin J. Colligan*[1]; [1]Concurrent Technologies Corporation, Harvest, AL USA

Since FSW was originally invented by The Welding Institute advancements in welding tool design have allowed the process to be operated at higher travel speeds, in thicker sections, and with more reliable quality. A new feature has been developed, referred to as the tapered shoulder, which allows FSW to be operated more easily and with more simple equipment. The tapered shoulder consists of a scrolled shoulder that is not generally flat but is tapered outward from the outside diameter to the pin. This scrolled cone can be embedded beneath the surface of the plate to any depth, with the width of the crown of the weld being variable depending on the depth of penetration. This patented tool design has implications for conventional welds, for welds in material with complex curvature and in pipe, and for self-reacting welds using tools that have a fixed geometry. This paper describes the welding tool design and its use in different welding applications.

4:50 PM
Surface Friction Welding – A New Process for Butt Welding of Thin Metal Sheets: *Sung-Joon Kim*[1]; Heung Nam Han[1]; Chang Gil Lee[1]; Sang-Sik Kim[2]; [1]Korea Institute of Machinery & Materials, 66 Sangnam, Changwon 641-010 Korea; [2]Gyeongsang National University, Jinju 660-701 Korea

A novel process for butt welding of metal sheets thinner than 2.0 mm was invented and patented by the authors. The process was named as surface friction welding (SFW) which utilizes friction heat and plastic deformation like friction stir welding (FSW). The SFW was successfully applied to butt welding of 6061Al sheets of 1.5 mm thick and Mg alloy sheet of 1.0 mm thick. This paper will introduce the principle of SFW, the difference between FSW and SFW, the effect of welding parameters, and the microstructure and mechanical properties of welded sheets.

5:10 PM
Comparison of Self-Reacting and Standard Fixed Pin Friction Stir Welding for Fabrication of Aluminum Box Beams: *Alex Paul Toskey*[1]; William J. Arbegast[1]; Anil K. Patnaik[1]; Casey A. Allen[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Work has been done to investigate friction stir welding as a cost effective alternative to extruded hollow aluminum box beams. The proposed design is to extrude two C-Channels and join them along the length by FSW. Both self-reacting and standard fixed pin tools have

been evaluated as methods of joining. The effects of different pin tool geometries will be presented as well as tooling setups for each type. Metallurgy and mechanical assessment of resulting welds will be compared along with process forces and thermal data. In the end, tooling limitations and issues with long self reacting pin tool use in closed sections resulted in the standard fixed pin tool being chosen for the final design fabrication approach.

5:30 PM
Experimental Measurements of Longitudinal Load Distributions on Friction Stir Weld Pin Tools: *Aaron L. Stahl*[1]; Carl D. Sorensen[1]; [1]Brigham Young University, Mechl. Engrg., 435 CTB, Provo, UT 84602 USA

An understanding of the forces acting on the pin of FSW tools is critical to appropriate design, especially in materials with limited toughness like PCBN. This paper describes a study to measure the longitudinal force distribution on a friction stir weld pin tool. Total longitudinal forces were recorded on a dynamometer while welding 6061 aluminum with pins that varied in length and diameter. A model was developed that characterizes pin force as a function of pin length and diameter. As the pin length approaches zero, the longitudinal force reaches an asymptote, which is apparently the longitudinal force due to the shoulder. The force due to the pin increases with pin length, but varies insignificantly with pin diameter. The force distribution on the pin appears to increase linearly with distance from the shoulder. Unexpected force variation was found at large pin lengths, a result which has yet to be explained.

TUESDAY PM

Frontiers in Solidification Science: Crystal-Melt Interfaces: Fundamental Properties and Related Behavior

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday PM Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mike I. Baskes, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Martin E. Glicksman, Rensselaer Polytechnic Institute, Troy, NY 12180 USA

2:00 PM Invited
Atomistic Simulations of Solid-Liquid Interface Mobility in FCC and BCC Metals: *J. J. Hoyt*[1]; M. D. Asta[2]; D. Y. Sun[3]; A. Karma[4]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA; [2]Northwestern University, Dept. of Matls Sci. & Engrg., Evanston, IL 60208 USA; [3]East China Normal University, Physics Dept., Shanghai China; [4]Northeastern University, Physics Dept., Boston, MA 02115 USA

The crystal-melt interface mobility plays an important role in controlling the morphology and growth kinetics of dendrites. The mobility is typically characterized by the kinetic coefficient, μ, defined as the constant of proportionality between the interface undercooling and the solidification velocity. Using molecular dynamics simulations and interatomic potentials of the embedded atom form, we have computed the kinetic coefficient in several FCC and BCC pure metals. With the exception of BCC metals growing along [100], the crystallization rates and their dependence on crystallographic growth direction are well described by a density functional theory based model due to Mikheev and Chernov. The discrepancy in the BCC case stems from an anomalously large solid-liquid interface width observed for the [100] orientation. In addition, we have computed the kinetic coefficient in a binary alloy, namely the B2 ordered NiAl system. For NiAl a low value of μ and a pronounced asymmetry between solidification and melting rates is observed.

2:35 PM Invited
Crystal-Melt Interfacial Free Energies in Metals: Role of Crystal Structure: *Mark D. Asta*[1]; Jeffrey J. Hoyt[2]; Deyan Sun[3]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., Evanston, IL 60208 USA; [2]Sandia National Laboratories, Albuquerque, NM USA; [3]East China Normal University, Dept. of Physics, Shanghai China

The magnitudes and crystalline anisotropies of crystal-melt interfacial free energy have been calculated for a variety of metal and alloy systems, based upon molecular-dynamics simulations employing embedded-atom potentials. Results for fcc Ag, Al, Au, Cu, Fe, Ni and Pb are compared with recent calculations for bcc Fe, Mo, V and hcp Mg. Calculated interfacial free energies are found to be well described by Turnbull's empirical scaling relation, with Turnbull coefficients that are roughly 30 percent lower for the bcc metals than for fcc and hcp. A trend towards lower anisotropies for bcc relative to fcc metals is observed. Results for the ordered bcc-based B2 NiAl compound show Turnbull coefficients and anisotropies consistent with those for the elemental bcc metals. Overall these results suggest that the underlying crystal lattice structure plays a primary role in governing crystal-melt interfacial free energies in metals.

3:10 PM Invited
Calculation of Crystal-Melt Interfacial Free Energies by Atomistic Simulation: Dependence Upon Crystal Structure and Interatomic Forces: *Brian B. Laird*[1]; Ruslan L. Davidchack[2]; [1]University of Kansas, Chmst., 1251 Wescoe Hall Dr., Lawrence, KS 66045 USA; [2]University of Leicester, Math., Univ. Rd., Leicester LE1 7RH UK

We review our recent work on the direct calculation, via MD computer simulation, of the interfacial free energy, γ, of the crystal-melt interface for a number of model systems. The value of γ as a function of crystal orientation is determined using a thermodynamic integration technique employing moving cleaving walls [Phys. Rev. Lett., 85, 4751 (2000)]. The calculation is sufficiently accurate to at least partially resolve the small anisotropy in γ, which is important input for continuum simulation of dendritic growth. We report values of γ for the hard-sphere and Lennard-Jones systems, as well as recent results on the series of repulsive inverse-power potentials. For the inverse sixth and inverse-eighth power systems, we determine γ for both fcc and bcc crystal structures.For these systems, the bcc-melt γ is lower that that of fcc by about 30%, in qualitative agreement with recent simulations on iron by Asta, Hoyt and Karma.

3:45 PM Break

3:55 PM Invited
Modeling Interaction of Dendritic Interfaces with Biological Cells: Anthony Chang[1]; *Jonathan A. Dantzig*[1]; [1]University of Illinois, Mechl. & Industl. Engrg., MC-244, 1206 W. Green St., Urbana, IL 61801 USA

Cryopreservation involves solidification of a water-based solution and incorporation of biological cells. A successful protocol ensures that the cells are not damaged during freezing by any of several possible factors, including excessive solute concentration, intracellular ice formation and mechanical damage. The solidifying interface is almost always dendritic. In this work, we present models for directional solidification (DS) of aqueous solutions containing cells. We use a level set method to model the solidification, and include direct calculation of the interaction forces between the cells and the moving interface. We examine the role of particle size, and location with repsect to the evolving interface structure, on capture and pushing ahead of the interface.

4:30 PM
In-Situ Synchrotron Microtomography Study of Morphology Evolution and Microporosity Formation in Solidifying AlSiCu-Alloys: Dominique Bernard[2]; Marco Di Michiel[3]; *Øyvind Nielsen*[1]; Luc Salvo[4]; [1]SINTEF, Matls. & Chmst., PB 124 Blindern, Oslo 0314 Norway; [2]ICMCB-CNRS, 33608 Pessac, Cedex France; [3]ESRF, Polygone Scientifique Louis Néel, 6 rue Jules Horowitz, Grenoble 38000 France; [4]GPM2-INPG-CNRS, Saint Martin d'Heres 38402 France

Direct observation of the microstructure evolution in solidifying metal alloys is still a great challenge in solidification science, which is manifested by the extensive literature on quenching experiments and transparent analogues. Although recent progress has been made in time resolved, high resolution x-ray imaging of thin metal samples during solidification (2D), the extension to microtomography (3D) has not yet been made, due to limitations in e.g., the x-ray beam intensity and characteristics, and the data acquisition and transfer speed. In the present work, recent results are shown from a study aiming at time resolved, high resolution 3D x-ray-imaging using microtomography at the European Synchrotron Radiation Facility (ESRF). Cylindrical samples (D=1.2 mm, H=1.2 mm) of Al-9 wt.% Si-3.5 wt.% Cu alloys were mounted in the microtomography setup at ESRF Beamline ID15A, melted completely by the use of a small furnace, and then cooled at a constant rate of 0.1°C/s. During solidification, about 40 tomograms were acquired for each sample. The spatial resolution is 2.8 μm and the data acquisition time for each tomogram (400 images) is 13 s. Due to

the segregation of copper into the liquid phase, solid/liquid absorption contrast is achieved and the 3D evolution of the solid/liquid interface was observed and quantified. Moreover, the nucleation and growth of gas and shrinkage porosity were observed.

4:50 PM

Containerless Solidification of Undercooled Eutectic Melts: *Mingjun Li*[1]; Yasutomo Arai[1]; Jianding Yu[1]; Takehiko Ishikawa[1]; Shinichi Yoda[1]; Kosuke Nagashio[2]; Kazuhiko Kuribayashi[2]; [1]Japan Aerospace Exploration Agency, Inst. of Space & Astronautical Sci., Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan; [2]Japan Aerospace Exploration Agency, Inst. of Space & Astronautical Sci., Sagamihara Campus, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan

We employed an electromagnetic levitator to investigate Ni-Sn, Ni-Si, Co-Sn, Co-Sb, and Co-Ge eutectics at various undercoolings. Microstructural observation revealed that individual eutectic colonies distribute not only at sample surface, but also throughout the volume of an entire sample. Similar microstructural characteristics were found in binary Al2O3-ZrO2, Fe2O3-La2O3, Yb2O3-Al2O3, Al2O3-MgO, Yb2O3-SiO2, Sm2O3-SiO2, and Y2O3-SiO2 oxide eutectics solidified on an aero-acoustic levitator. The latest microstructure on Al2O3-ZrO2-Y2O3 eutectics consisted of independent eutectic colonies, indicating that massive or copious nucleation occurs in free solidification of these poly-domain systems, either binary metallic or binary and ternary oxide eutectics. The eutectic growth model is modified to incorporate kinetic undercooling for competing solid solution and compound; collision-limited growth generates a highly mobile interface compared with that of diffusion-limited growth and thus the solid solution overgrows the compound to yield anomalous eutectic. This model can interpret most published data on the formation of anomalous eutectics.

5:10 PM

Microstructure Evolution on Shock Melt and Refreeze in Bismuth: *Bryan W. Reed*[1]; Jeff Colvin[1]; Alan Jankowski[1]; Mukul Kumar[1]; Dennis Paisley[2]; Damian Swift[2]; Tom Tierney[2]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., PO Box 808, L-370, Livermore, CA 94551 USA; [2]Los Alamos National Laboratory, Plasma Physics, P-24, MS E526, PO Box 1663, Los Alamos, NM 87545 USA

Because solid bismuth is less dense than the liquid, pressurized liquid bismuth should begin to freeze immediately upon release of the pressure, yielding extremely fast initial undercooling rates (~10^{10} K/s upon initial release of a shock wave, according to a single-phase model). Using the technique of tamped ablation (Colvin et al., Phys. Plasmas 10/7, 2940, 2003), we used long-pulse lasers to shock-melt preheated Bi in order to induce a rapid melt/refreeze cycle. The recovered material has a mix of microstructures including fine dendrites indicating rapid resolidification. We interpret the shocked microstructures in light of theoretical predictions, real-time shock diagnostics (resistivity and free-surface velocity), and controlled variations of the pre-shock microstructure. The results carry implications for the study of solid-liquid phase changes on very short time scales. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

Frontiers in Solidification Science: Poster Session

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee

Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday, 5:30-7:30pm Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Shan Liu, Ames Laboratory, Ames, IA 50011 USA; Ralph Napolitano, Iowa State University, Ames, IA 50011 USA; James Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Convective Effects on Thermosolutal Free Dendritic Growth: *Juan C. Ramirez*[1]; Christoph Beckermann[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-8, PO Box 1663, MS G755, Los Alamos, NM 87545 USA; [2]University of Iowa, Dept. of Mechl. & Industl. Engrg., 2412 Seamans Ctr., Iowa City, IA 52242-1527 USA

Two-dimensional phase field simulations are performed to examine the effects of melt convection on the dendrite tip operating state of dilute binary alloys. The model employed reproduces the usual sharp interface conditions in the limit of a thin, diffuse interface region. Detailed knowledge of thermal, solutal and velocity fields around the dendrite tip from simulations provide great insight into the understanding of convective effects on dendritic growth. Depending on the relative importance of thermal and solutal effects, three regimes are considered: purely thermal, purely solutal and thermosolutal solidification. The simulation results are compared to theoretical models.

Mechanisms of Primary Silicon Growth in Near-Eutectic Al-Si Alloys: *Ralph E. Napolitano*[1]; Choonho Jung[1]; [1]Iowa State University, Matls. Sci. & Engrg., Ames Lab., Matls. & Engrg. Physics, Ames, IA 50011 USA

Morphological evolution and selection of angular primary silicon is investigated in near-eutectic Al-Si alloys. Angular silicon "dendritic" arrays are grown directionally in a Bridgman furnace at velocities in the regime of 10-3 m/sec. Serial milling and x-ray techniques are combined with backscattered electron diffraction analysis to examine the early-stage selection dynamics and primary array evolution through various tip-splitting and branching mechanisms. The detailed structure of the twinned bicrystal dendritic cores and the role of twinning in the mechanisms of branching and spacing adjustment are discussed. Compositions from 10 wt% Si to 14 wt% Si are investigated, and the transition from primary Si growth to coupled eutectic growth is examined.

Convectional Effect on Morphology and Segregation of Directionally Solidified Ductile Irons: *Wen Shiung Chang*[1]; Tien Shou Lei[1]; [1]National Taiwan University of Science and Technology, Dept. of Mechl. Engrg., #43, Sect. 4, Keelung Rd., Taipei 106 Taiwan

Convection effect plays an important role in the final microstructures and chemical segregations during the solidification of castings. In this paper the effect of fluid convection on morphology and segregation of directionally solidified ductile irons will be presented. A vertical cylindrical mold cavity set-up which consists by two furan molds sandwiched with a water chilled copper mold in the middle of the furans was used for studying that provides three distinct solidifying zones: an upward, a bidirectional and a downward. In the downward portion, it was observed that a number of larger nodular graphite near the chilled mold which solidified first, but some smaller nodular graphite existed at the bottom which was last solidified. These phenomena are different from that of casting which solidified in upward direction. The mechanism of elements redistribution such as molybdenum and chromium, analyzed by EPMA, in two founded alloy-rich carbides will also be discussed.

The Effect of Seed Dendrite Arm Spacing on Single Crystal Ni-Base Superalloys: *Elyssa Renee Cutler*[1]; Gerhard E. Fuchs[1]; [1]PO Box 116400, Gainesville, FL 32611 USA

Recent cellular automaton-finite difference (CA-FD) models have predicted a range of stable dendrite arm spacings for single crystal binary castings according to thermal history. An attempt to verify this model by changing seed crystal dendrite arm spacing within a single casting was made with samples of a single crystal Ni-base superalloy, CMSX-4. Three widely varying seed dendrite arm spacings were chosen both inside and out of predicted stable ranges. Both bars and blade shapes were cast using standard industry parameters. Each casting was sectioned to determine the time required to reach steady state and to characterize the dendrite morphology and the origin of any casting defects.

Examining the Benefits of Liquid Metal Cooling (LMC) for Directional Solidification of Large Ni-Base Superalloy Castings: *Andrew J. Elliott*[1]; Tresa M. Pollock[1]; Michael F.X. Gigliotti[2]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]General Electric, Global Rsch., Niskayuna, NY 12309 USA

A series of experiments investigating liquid-tin assisted directional solidification (DS) of large Ni-base superalloy castings has been conducted. Compared to the conventional Bridgman (radiation cooling) process, liquid metal cooling (LMC) resulted in considerably enhanced cooling rates, refined microstructure, and reduced occurrence of casting defects, including elimination of freckle-type defects. Additionally, withdrawal rates at least two to three times those capable with the conventional process were achieved while maintaining an aligned crystal structure free from casting defects. Preliminary mechanical testing of specimens made using both the conventional and LMC processes was conducted to examine any possible mechanical benefit of the LMC

process. The effect of various LMC process parameters were also investigated through experiments and thermal modeling. The experimental casting results, microstructural analysis, mechanical comparison, and thermal modeling are examined to provide a broader understanding of how the LMC process is beneficial for producing large DS castings.

Multi-Scale Modeling of Particle-Solidification Front Dynamics: *Justin Wayne Garvin*[1]; Yi Yang[1]; Holavanahalli S. Udaykumar[1]; [1]University of Iowa, Dept. of Mechl. Engrg., 3131 Seamans Ctr., Iowa City, IA 52242 USA

The interaction between a micron-sized particle and a solidification front is inherently a multi-scale problem. For particles of microns in size or greater, the solution of the Navier-Stokes equations in the particle-front system including resolution of the nano-scale gap between surfaces would be impossible due to the fine mesh requirement needed. A lubrication model is therefore developed for the interaction between any two immersed solid surfaces moving relative to one another at arbitrary velocities. The model includes the disjoining pressure effects due to intermolecular forces as well as liquid-solid phase change at one or both of the interfaces. The solution to the lubrication model is coupled to the solution outside the gap that is solved using the Navier-Stokes equations. The interfaces are tracked using a level-set method. Results are obtained for the interaction between a particle and a solidification front and are compared to previous theoretical results for the interaction.

Impurity-Induced Instabilities: Layachi Hadji[1]; [1]University of Alabama, Dept. of Math., 345 Gordon Palmer Hall, Tuscaloosa, AL 35487-0350 USA

We examine the interaction of a minute foreign impurity with a directionally solidified interface. We show that the interaction induces the onset of morphological instability provided that the distance between the impurity and the interface falls below a critical value. This instability occurs at pulling speeds that are below the threshold for the onset of the Mullins-Sekerka instability. The expression for the critical distance reveals that this instability is manifested only for certain combination of the physical and processing parameters, and its occurence is attributed to the reversal of the thermal gradient in the melt between the impurity and the solid-liquid interface. Another instability may arise when the impurity is in near-contact with the interface. The disjoining pressure affects the interaction. We derive an expression for the film thickness at which rupture occurs. The influence of these instabilities on the capture or rejection of the impurity is discussed.

The Effects of Internal Convection on Dendritic Evolution in Stainless Steel Alloys: *Alaina B. Hanlon*[1]; Robert W. Hyers[1]; Douglas M. Matson[2]; [1]University of Massachusetts, Dept. of Mechl. & Industl. Engrg., Engrg. Lab Bldg., 160 Governors Dr., Amherst, MA 01003 USA; [2]Tufts University, Dept. of Mechl. Engrg., Rm. 025 Anderson Hall, 200 College Ave., Medford, MA 02155 USA

In certain compositions, Fe-Cr-Ni stainless steels solidify from an undercooled melt by a 2-step process in which the metastable ferrite phase transforms to a stable austenite phase. Recent experiments have shown that flow within the molten sample strongly influences the lifetime of the metastable phase. The current research will provide insight to why flow affects the metastable phase, and lead to the use of convection to control microstructural evolution. If the convective velocities are great enough to cause the dendrites to bend then low angle boundaries form at the points of collision. These result in high energy sites that could serve as nuclei for the stable phase. Numerical models are employed to evaluate interactions between fluid and microstructures during rapid solidification. Simulations yield a range of convective flow velocities that causes mechanical damage to the dendrites and can be compared to experimental results.

Phase-Field Model for Alloys with Arbitrary Phase Diagrams: Seong Gyoon Kim[1]; *Won Tae Kim*[2]; [1]Kunsan National University, Dept. Matls. Sci. & Engrg., 68 Miryong Dong, Kunsan 573-701 Korea; [2]Cheongju University, Applied Sci. Div., 36 Naedok Dong, Cheongju 360-764 Korea

Remarkable progress in phase-field simulations of alloy solidification has been made by using the thin-interface analysis and introducing an anti-trapping current into the standard phase-field model for alloys. This make us to overcome the stringent restriction on the interface width and enhance both the computational efficiency and accuracy. In this study, we extend the phase-field model with the anti-trapping current to alloy systems with arbitrary phase diagrams and find the mapping conditions of the phase-field model onto the classical moving boundary problem. A few simulations for testing the self-consistency will be shown and compared with the previous model of localized solute partitioning in diffuse interface.

Anisotropic Free Energies of Crystal-Melt Interfaces: *James R. Morris*[1]; Ruslan L. Davidchack[2]; Mikhail I. Mendelev[3]; David J. Srolovitz[4]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]University of Leicester, Dept. of Math., Leicester LE1 7RH UK; [3]Iowa State University, Ames Lab., Ames, IA 50011 USA; [4]Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton, NJ 08544 USA

Recently, there have been significant advances in approaches to calculating the free energy of crystal-melt interfaces from simulations. Current approaches can not only predict accurate values, but can resolve the anisotropies in the values on the order of 1-2%. We demonstrate these approaches, in particular the fluctuation approach applicable to rough interfaces, and compare the results. For the Lennard-Jones system, the results are in very close agreement, whereas the hard-sphere results give smaller values than in previous calculations. For more realistic models of Al, we show that different potentials have very different values, which are correlated with the liquid structure. We also include recent results for purely repulsive potentials, of the form $1/r^n$. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR-22725 with UT-Battelle.

Microstructure Evaluation and Prediction During the Investment Casting of Ti-6Al-4V Plates: *Laurentiu Nastac*[1]; Kevin Klug[2]; Mehmet N. Gungor[2]; Troy Tack[2]; [1]Concurrent Technologies Corporation, 425 Sixth Ave., Regional Enterprise Tower, 28th Fl., Pittsburgh, PA 15219 USA; [2]Concurrent Technologies Corporation, Johnstown, PA 15904 USA

This paper describes the modeling and the experimental efforts for the evaluation and prediction of the microstructure and its relationship with the mechanical properties in investment casting of Ti-6Al-4V (Ti-6-4) alloy. This work supports the M777 Lightweight Howitzer (LWH) program, where investment casting was selected over machining and welding titanium plate to reduce part count and associated manufacturing expense for several LWH components. Key predictive capabilities such as shrinkage, alpha-case thickness, and microstructure were simulated. The purpose of the work was to better understand the investment casting process to eliminate/minimize defects in LWH components and provide useful insights to meet aggressive schedule requirements by minimizing experimental production trials. The modeling approach was based on the numerical solution of fluid flow and heat transfer equations and mesoscopic modeling of the microstructure. The effects of casting parameters on the plate microstructures were studied. The amount of alpha and beta phases as a function of cooling rates was experimentally measured. A relationship between the microstructure and mechanical properties was established. The influence of composition variance on both the microstructure and mechanical properties was investigated. Evaluation results showed that the HIP and heat treated Ti-6-4 plates have acceptable mechanical properties and microstructure with minimum grain coarsening and porosity. Acknowledgment. This work was conducted by the National Center for Excellence in Metalworking Technology (NCEMT), operated by Concurrent Technologies Corporation under contract No. N00014-00-C-0544 to the U.S. Navy as part of the U.S. Navy Manufacturing Technology Program. Approved for public; distribution is unlimited.

Effects of Solidification-Induced Segregation on Alloy 22 Phase Stability: *Yi-Ming Pan*[1]; Darrell S. Dunn[1]; Gustavo A. Cragnolino[1]; [1]CNWRA, Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

Simulations have been conducted to determine the phase stability of Alloy 22 affected by elemental segregation in the solidified weld microstructure. Compositional analyses of gas-tungsten arc welded Alloy 22 using energy-dispersive x-ray spectroscopy revealed that the interdendritic regions are enriched in Mo and depleted in Ni relative to the dendrite cores. Both equilibrium and Scheil solidification simulations were performed with the Thermo-Calc Version N software and the Ni-DATA Version 5 database. The solidus, liquidus, and P-solvus temperatures were calculated for the bulk alloy composition, as well as for the measured compositions for the dendrite cores and the interdendritic regions. The equilibrium simulations predict complete solidification of liquid to γ-phase whereas the Scheil simulations predicts the formation of P-phase (the predominant topologically close-packed phase of Alloy 22) near the end of solidification. In addition, the Scheil simulation predicts a high solvus temperature for the P-phase in the interdendritic regions due to element partitioning in the

solidified weld microstructure. The implications of these results for the solution annealing treatment of Alloy 22 weldments are discussed. Acknowledgment: This paper is an independent product of the CNWRA and does not necessarily reflect the views or regulatory position of the NRC.

Phase Field Modeling of Crystal Nucleation in 3D: *Tamas Pusztai*[1]; Laszlo Granasy[1]; [1]Research Institute for Solid State Physics and Optics, POB 49, Budapest H-1121 Hungary

Homogeneous and heterogeneous nucleation is studied in the framework of the phase field theory. The properties of critical fluctuations is determined by solving the Euler-Lagrange equations. The nucleation rate is evaluated from simulations performed with Langevin-noise. The magnitude of the nucleation prefactor is estimated from simulations and compared with theoretical predictions. Sharp walls defined via the "no-flux" boundary condition are introduced into the simulations. Heterogeneous nucleation on foreign particles, rough walls, and solidification in porous media are addressed.

Multiscale Modeling of Solidification Processes: *Yan Shu*[1]; Xin Ai[1]; Ben Q. Li[1]; [1]Washington State University, Mechl. Engrg. Dept., Pullman, WA 99163 USA

Solidification processing involves multiple length and time scales. This paper discusses a multiscale computational scheme for the modeling of the microstructure development during solidification processing. The molecular simulation, phase field modeling and continuum-based fluid flow and heat transfer phenomena are integrated to cover eventually from the molecular scale all the way to the continuum domain. The modeling strategies that involve both mutual and one-way coupling of molecular, micro and macroscale phenomena are discussed. Computational results are given for some solidification systems.

A Robust and Efficient Equiaxed Grain Growth Model and Coupling To a Macro Heat Transfer Algorithm: *Andrei Starobin*[1]; David Torres[2]; Marius Stan[3]; [1]Metal Casting Simulation Consultant, 211 Maynard Dr., Santa Fe, NM 87501 USA; [2]Los Alamos National Laboratory , T-3, Los Alamos, NM 87545; [3]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545

A robust and efficient equiaxed growth model is developed. It is a variant of that suggested by Beckermann et al (1993) modified to a 2-ODE form to allow for an exactly conservative in enthalpy meso-macro coupling. A further modification is a full, fast, numerical treatment of the solute diffusion in the shrinking extradendritic region based on the work of Torres et. al. (2004). This numerical approach allows uniform treatment of growth: from just after nucleation through coalescence. A fully iterative meso-macro coupling poses significant challenges for a macroscopic enthalpy algorithm in the recalescence region where the cooling curves are highly oscillatory. These numerical difficulties are addressed and a coupling with finite-volume, enthalpy based, Jacobian matrix-free heat transfer algorithm of Knoll et al (1999) is demonstrated. Comparison is made with a faster meso-macro passive 1-way coupling.

The Influence of Fluid Flow on the Microstructure of Directionally Solidified AlSi-Base Alloys: *S. Steinbach*[1]; L. Ratke[1]; [1]Institute of Space Simulation, German Aeros. Ctr. DLR, D-51147 Cologne Germany

A quantitative understanding of the effect of fluid flow on the microstructure of cast alloys is still lacking. We therefore solidified AlSi-base alloys directionally under well defined thermal and magnetically controlled, convective boundary conditions. This was achieved with a new furnace facility that utilizes the extreme properties of transparent nanostructured silica aerogels as a crucible material, leading to flat isotherms and allowing the direct optical observation of the solidification process. Three pairs of Helmholtz coils around the sample can induce a homogeneous rotating magnetic field. The field strength can be related directly to the flow field inside the liquid and the mush using numerical modelling. The investigations show that the microstructural features like the primary dendrite, the secondary dendrite arm spacing, the eutectic spacing and the fraction solid change in a unique manner with solidification speed and rotating magnetic field strength. The experimental results are compared to literature data and accepted steady-state growth models. Simple models are derived for the effect of convection on microstructure parameters.

Analysis of the Solidification of Particle-Laden Melts: *Dawei Sun*[1]; Suresh V. Garimella[1]; Aniruddha Mukhopadhyay[2]; [1]Purdue University, Sch. of Mechl. Engrg., W. Lafayette, IN 47907 USA; [2]Fluent Inc., Lebanon, NH 03766 USA

A numerical model, based on the enthalpy-porosity method and the volume of fluid (VOF) method, has been developed for the analysis of coupled fluid flow and heat transfer processes during the casting of particle-laden fluids. The primary focus of this work is the prediction of solidification shrinkage. The influence of the suspended particles in the non-Newtonian fluid on the phase change process is taken into account. The proposed model is first validated against experimental measurements, and then used to investigate pipe formation in the casting of energetic materials. It has been found that solidification shrinkage plays an important role in the casting process, and the extent to which the non-Newtonian nature of the fluid needs to be considered depends on the concentration of the suspended particles.

Crystal Growth in Polymeric Materials: *Jing Teng*[1]; Shan Liu[2]; Rohit Trivedi[1]; [1]Iowa State University, Dept. Matls. Sci. Engrg., 225 Wilhelm Hall, Ames, IA 50010 USA; [2]Iowa State University, Ames Lab.-USDOE, Matls. & Engrg. Physics, 235 Wilhelm Hall, Ames, IA 50010 USA

In comparison with metallic alloys, polymer crystals have a much less symmetrical crystal structure and very complicated growth morphologies. Instead of crystallizing by the atomic jump across the growth interface in the small molecular and metallic alloys, polymer crystallization proceeds with the secondary nucleation and conformational change. Spherulites are the most common growth form in polymers and have been widely used for study of kinetics in undercooled growth process. In this paper, we propose to use the directional growth technique to determine the growth kinetics since the interface temperature and growth velocity can be precisely measured concomitantly. The data were compared with the nucleation theory and a growth regime change at ~16K undercooling was observed for PEG. A crossover of the growth kinetics curves for the undercooled and directional growth was found, which is believed to be due to the difference in heat transfer between the two growth configurations.

Undercooling of Liquid Iron Droplets Partly Submerged in Slags and in Contact with Oxides: *Martin Emiliano Valdez*[1]; Seetharaman Sridhar[1]; Alan W. Cramb[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Wean Hall 2325, Pittsburgh, PA 15213 USA

This paper describes a novel experimental technique to measure undercooling of metals and alloy droplets when the only interface available is a steel/slag interface. The methodology involves observing the shape change of a small droplet submerged in a slag without contacting the crucible walls through a Confocal Scanning Laser Microscope (CSLM). The results presented are for pure Fe droplets in a silicate based slag. The experiments indicate that fully submerged particles can be significantly undercooled (up to 260 K). The extent of undercooling increases after each successive re-melting of the sample, which suggests that impurity content has a strong effect on the undercooling.

Characterization of Carbide Growth in Directionally Solidified High Chromium White Cast Iron: *Dong Shyen Yang*[1]; Tien Shou Lei[1]; [1]National Taiwan University of Science and Technology, Dept. of Mechl. Engrg., 43 Keelung Rd., Sect. 4, Taipei 106 Taiwan

The characterization of carbide growth in directionally solidified castings of ASTM A532-87 Class - V high chromium white cast iron is investigated. A vertical cylindrical casting set-up which consists of two layers of Furan sand mold and sandwiched with a chilled copper mold was used for directional solidification. This solidification set-up provides three zones with different solidification direction: an upward zone above the chilled mold, a bidirectional solidification zone within the chilled copper mold, and a downward zone below the chilled mold. Due to the effect of gravity and fluid convection the solidification characteristics in these three zones are different. The morphology of primary alloy carbide M7C3 shows differences in these three zones, specifically, long rods of M7C3 carbide up to 3 mm were grown in the chilled mold. Other than SEM used for morphologies, the EBSD results will be used to study the preferred crystallographic orientation of the M7C3.

Numerical Simulation of Thermal Behavior During 3-D Laser Deposition: *Baolong Zheng*[1]; Yaojun Lin[1]; Yizhang Zhou[1]; John E. Smugeresky[2]; Enrique J. Lavernia[1]; [1]University of California, Chemical. Engrg. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA; [2]Sandia National Laboratories, Dept. 8724, Livermore, CA 94551-0969 USA

As a result of the layer additive nature associated with the laser engineered net shaping (LENS™) process, the parts to be built would experience thermal cycles during LENS™ deposition. The laser deposition of LENS™ process can be viewed as a sequence of discrete events, and accordingly, the thermal history could be modeled numerically by using the finite difference methods (FDM). The objectives of this study are to: (1) understand the complete thermal behavior during LENS™ processing, and (2) correlate the processing parameters (e.g.,

substrate temperature, laser power, substrate traverse speed) with the microstructure and properties of parts. The numerical results from a series of designed experiments of different geometries will be compared with the experimental observations from samples processed with closed loop feedback control. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

Interactions of Dendrites with Embedded Particles: *Yi Yang*[1]; Justin Wayne Garvin[1]; Holavanahalli S. Udaykumar[1]; [1]University of Iowa, Dept. of Mechl. Engrg., 3131 Seamans Ctr., Iowa City, IA 52242 USA

Numerical simulations of dendrite-particle interactions, which are important in metal-matrix composite production, are performed with a sharp interface method. A pure under-cooled melt is used to grow the dendrite. When the dendrite approaches the particle, appropriate conditions are applied at the particle-solid interface before and after contact. The behaviour of dendrites as they approach and grow around the particle is closely examined. For a particle-melt thermal conductivity ratio kp/kl < 1.0, the solidification front does not get close enough to the particle to activate the particle pushing mechanism. Instead, the solidification front chooses to go around the particle, and eventually the particle is engulfed by sidebranches. This result contradicts theoretical treatments of planar front interactions with a particle. Dendrite-particle interactions in alloys are also investigated.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Semiconductors

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee

Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Tuesday PM Room: 3020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Roger Narayan, Georgia Institute of Technology, Biomatls. & Bioengrg., Atlanta, GA 30332-0245 USA; Douglas B. Chrisey, Naval Research Laboratory, Washington, DC 203375-5345 USA

2:00 PM
Dilute Magnetic Semiconductors: *John T. Prater*[1]; [1]Army Research Office, PO Box 12211, Rsch. Triangle Park, NC 27709-2211 USA

The recent revelation that the magnetic spin component associated with electronic charge could be used to provide enhanced functionality in electronic devices has led to a flurry of research on dilute magnetic semiconductors. In particular, studies on the magnetic doping of various wide band-gap semiconductors have been conducted across the country in search of systems that will display room temperature ferromagnetism. An overview of the progress made to date by the various groups around the country will be presented. This overview will also include some recent findings by Professor Narayan's research group at North Carolina State University, where I have had the honor of establishing a most rewarding research collaboration. Recent breakthroughs for cobalt doped systems suggest that a viable materials solution may be near at hand. The challenge will soon turn from material discovery to materials integration. Here the concepts of domain matching epitaxy (DME), which were first forwarded by Professor Narayan in the late 1990's, will afford expanded options for device fabrication. The DME concept will be presented as it applies to spintronic device integration and the deposition of magnetic semiconductors onto such mainline commercial substrates as silicon.

2:30 PM Invited
Non-Random Distribution of Atomic Species in Mixed III-V Layers: *Subhash Mahajan*[1]; [1]Arizona State University, Cheml. & Matls. Engrg., PO Box 876006, Tempe, AZ 85287-6006 USA

We will demonstrate that atomic species in mixed III-V layers, differing in their covalent tetrahedral radii, are not distributed at random on their respective sub-lattices. Two types of deviations from randomness are observed: phase separation and atomic ordering. Both of these features evolve at the surface or near surface regions during growth, and reduce the strain energy of the system. Our recent work on mixed group III nitride layers shows that InGaN layers indeed undergo two-dimensional phase separation, and exhibit weak tendency for 1:1 ordering. Al0.5Ga0.5N layers also show 1:1 ordering that is sensitive to composition. The above microstructural features affect the electronic and optical properties of layers, and should enhance the degradation resistance of light emitting devices. The author is grateful to NSF, AFOSR, ONR and DOE for research support.

3:00 PM Invited
An Atomistic Kinetic Framework for Strain Accomodation in Lattice Mismatched Semiconductor Epitaxy: *Anupam Madhukar*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Physics, Los Angeles, CA USA

The vast majority of descriptions of strain accommodation in lattice mismatched semiconductor overlayer-substrate systems rely upon comparison of the ground state energies of a (partially or wholly) strain relaxed atomic configuration with that of an assumed non-relaxed reference state, for a fixed number of particles. Tremendous strides have been made in reconciling and understanding experimental findings using such analyses, applied appropriately. Yet, growth of a film by its nature is the evolution of a system with changing number of particles. It is thus reasonable to consider an atomistic view in which the initiation, growth, and multiplication of strain relieving defects are dynamic events, tied to the changing kinetics and thermodynamics of the interacting open system. In this talk I shall present such a framework, motivated by molecular beam epitaxy of III-V compound semiconductors. It is necessarily incomplete, given the complex nature of a dynamical open system, but it does provide deeper insights into the nature of strain accommodation processes. It also lends itself to examination via multi-length and multi-time scale simulations and visualization carried out on increasingly more accessible parallel computing platforms, thus holding the promise of revealing potential pathways by which the system moves towards the experimentally observed state.

3:30 PM Invited
Core Structure of Screw Dislocation in GaN Studied by Transmission Electron Microscopy: *Z. Liliental Weber*[1]; [1]Lawrence Berkeley National Laboratory, Berkeley, CA USA

Thin film heteroepitaxy of polar materials such as GaN grown by MOCVD, MBE or HVPE Molecular Beam Epitaxy (MBE) grown on SiC or Al2O3 is frequently hampered by the formation of structural defects mostly dislocations, nanotubes and pinholes. In this presentation it will be shown that the screw dislocations present in HVPE samples are decorated by pinholes arranged on top of each other ("bamboo" structure) but these defects are not found in MBE samples grown on the top of HVPE samples. These might suggest either different core structure of a screw dislocation in HVPE and MBE grown material or suggest a different purity of these two materials. MBE samples grown under Ga-rich and Ga-lean material have been studied. A direct reconstruction of the phase and amplitude of the scattered electron wave from a focal series of high-resolution transmission electron microscopy (TEM) images were applied to learn about a core structure of screw dislocation. It will be shown that cores in MBE samples were filled and stoichiometric but in HVPE materials have an excess of Ga.

4:00 PM Break

4:15 PM Invited
Development of Separated Pulsed Laser Ablation for Oxide Light Emitting and Semiconducting Thin Films: *Kenji Ebihara*[1]; Manuel Alonso[1]; San-Moo Park[1]; Tomoaki Ikegami[1]; [1]Kumamoto University, Electl. & Computer Engrg., Kurokami 2-39-1, Kumamoto 860-8555 Japan

New type pulsed laser ablation process(separated pulsed laser deposition: SPLD) has been proposed for oxide thin film preparation using ozone and NO. The SPLD consists of the ablation chamber and the deposition chamber with the orifice. The ablation chamber is a stainless steel globe with 300 mm diameter. The deposition chamber is made with a quartz tube of 100 mm diameter and a metallic conic wall with variable orifice diameters. For the ablation process we use a KrF laser with =248 nm and 25 ns pulse duration. The differential vacuum and gas pressure in the two chambers allows us to have some control over the doping gas pressure, the plasma plume and the ion implantation by applying a variable voltage between the conic wall and the substrate. Typically this SPLD technique produces thin film ZnO at low temperature and leads to the creation of nano-hybrid thin film structures.

4:45 PM
Electrodeposition of Cd-Zn-Te Thin Films: *Krishnan Selva Raja*[1]; [1]University of Nevada, Metallurgl. & Matls. Engrg., 1661 N. Virginia St., MS 388, Reno, NV 89557 USA

Cadmium Zinc Telluride (CZT), a compound-type semiconductor, is a predominant radiation sensing material capable of detecting low to medium energy gamma radiation and hard X-rays. Conventionally, CZT (typical composition Cd0.9Zn0.1Te) is manufactured as a single crystal using Bridgman techniques and the detection efficiency of the CZT is improved by minimizing the bulk defects present in the crystal. Thin film and nano-wires of CZT material could exhibit 1or 2 D electron transport and therefore the detection efficiency can be improved substantially. In this study, thin films of CZT were electrochemically deposited in a single step process as well as two-steps process followed by annealing. Cyclic voltametry studies were carried out to understand the underlying electrochemistry of Cd-Zn-Te compound deposition. XRD and TEM studies are being carried out to characterize the thin films.

5:15 PM Invited
Structural, Optical, Magnetic and Electrical Properties of V Doped ZnO Thin Films: *Shivaraman Ramachandran*[1]; *Ashutosh Tiwari*[1]; *J. Narayan*[1]; [1]North Carolina State University, Matls. Sci., 2141 Burlington Labs, CB 7916, Raleigh, NC 27606 USA

Here we report a systematic study of structural, optical, electrical and magnetic properties of thin films of vanadium doped ZnO. Epitaxial thin films of ZnxV1-xO (x=0.01 to x=0.2) were prepared on sapphire c-plane single crystals using pulsed laser deposition technique. All the growths were done at temperatures ranging from 500-650°C. The emphasis in this work has been on determining the magnetic properties of these diluted magnetic semiconducting (DMS) systems and to correlate the properties, especially the magnetic properties with the microstructural aspects, such as occupation of the magnetic dopant in the lattice sites versus clusters and/or precipitates. It is now established that this system is devoid of any signatures of ferromagnetism either at room temperature or at low temperatures. The magnetic measurements were done using either a vibrating sample magnetometer or a superconducting quantum interference device (SQUID). The microstructural characterization was done using high-resolution transmission electron microscopy, coupled with electron energy loss spectroscopy (EELS) and STEM-Z (Scanning Transmission Electron Microscopy-Atomic Number) contrast experiments.

5:45 PM
Controlling Interface Effects in Si Solar Cells: *Bhushan Sopori*[1]; N. M. Ravindra[2]; Chuan Li[1]; Paresh Saxena[2]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA; [2]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

Fabrication of efficient Si solar cells requires high-quality wafers that have long minority carrier diffusion lengths and well-controlled interfaces/surfaces— interfaces that can offer very low carrier recombination. Control of interface properties is particularly important as the wafer thickness is reduced. Several approaches have been used for minimizing the carrier recombination velocity at the interfaces, which include minimizing interface state density and formation fields that can repel photogenerated minority carriers. Procedures for minimizing interface density are well established in the MOS technology. The minority carrier reflecting fields can be developed by formation of high-low junctions, dopant profiling, and by introduction of suitable surface charges, typically within a dielectric layer. In commercial Si solar cell fabrication, there can be conflicting demands between surface passivation and other cell requirements such as optical properties, processing schedules, and control of costs. This paper will discuss the effectiveness of these techniques and their compatibility with low-cost solar cell fabrication approaches.

General Abstract Session: Environmental Damage and Durability

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday PM — Room: 2011
February 15, 2005 — Location: Moscone West Convention Center

Session Chair: Alan K. Pelton, Nitinol Devices & Components, Fremont, CA 94539 USA

2:00 PM
Environmental Durability of Coated GRCop-84 Copper Alloys: *S. V. Raj*[1]; [1]NASA Glenn Research Center, Matls. Div., MS 106-5, 21000 Brookpark Rd., Cleveland, OH 44135 USA

An advanced Cu-8(at.%)Cr-4%Nb alloy developed at NASA's Glenn Research Center, and designated as GRCop-84, is currently being considered for use as liners in combustor chambers and nozzle ramps in NASA's future generations of reusable launch vehicles (RLVs). However, past experience has shown that unprotected copper alloys undergo an environmental attack called 'blanching' in rocket engines using liquid hydrogen as fuel and liquid oxygen as the oxidizer. Potential for sulfidation attack of the liners in hydrocarbon-fueled engines is also of concern. As a result, protective overlay coatings alloys are being developed for GRCop-84. The oxidation behavior of several new coating alloys has been evaluated. GRCop-84 specimens were coated with several copper and nickel-based coatings, where the coatings were deposited by either vacuum plasma spraying or cold spraying techniques. Coated and uncoated specimens were thermally cycled in a furnace at different temperatures in order to evaluate the performance of the coatings. Additional studies were conducted in a high pressure burner rig using a hydrocarbon fuel and subjected to a high heat flux hydrogen-oxygen combustion flame in NASA's Quick Access Rocket Exhaust (QARE) rig. The performance of these coatings are discussed.

2:30 PM
Hydrogen Effects on the Phase Stability of NiTi: *Katherine C. Chen*[1]; Amanda Runciman[1]; Alan R. Pelton[2]; Andreas Wick[2]; [1]California Polytechnic State University, Matls. Engrg. Dept., 1 Grand Ave., San Luis Obispo, CA 93407 USA; [2]Nitinol Devices & Components, 47533 Westinghouse Dr., Fremont, CA 94539 USA

Shape memory and superelastic NiTi are finding its way into several commercial products (e.g., endovascular stents, cell phone antennae wire, orthodontic arch wire), as well as potential new applications (such as morphing wings). The A_f (austenite transformation finish) temperature is usually the key property to control, and is very sensitive to processing conditions. During cleaning, etching, or heat treatments, hydrogen can be inadvertently introduced into the NiTi and affect the phase stability and properties. Samples of various amounts of hydrogen in NiTi have been prepared and studied by x-ray diffraction (XRD). With only roughly 80 wppm of H in austentic NiTi, new XRD peaks appear. Structural effects of hydrogen in martensitic NiTi are also investigated. Hydrogen-induced lattice strains and the appearance of new hydride phases are discussed and correlated against previous diffusion studies.

3:00 PM
A Study on the Electrochemical Properties of Si-Cu-Carbon Composite for an Anode Material of Li-Ion Batteries: *Yong-Mook Kang*[1]; Min-Sik Park[1]; Min-Sang Song[1]; Hyun-Seok Kim[1]; Kyu-Sung Han[1]; *Jai-Young Lee*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., 373-1, Guseong-Dong, Yuseong-gu, Daejeon S. Korea

In the previous reports, Si-Carbon composite has been suggested to improve the cycle life of Si. Even if Si-Carbon composite showed good cycle life in Si utilization of 32% (This means that 32% of the theoretical capacity was used.), its cycle life was precipitously deteriorated in Si utilization of 40%. On the other hand, Si-Cu-Carbon composite exhibited good cycle life (After 50 cycles, more than 98% of the initial capacity was maintained.) even in Si utilization of 55%. Then, the capacity of Si-Carbon composite was 679 mAh/g, while that of Si-Cu-Carbon composite came to 930 mAh/g. As shown in this result, the

cycle life of Si-Cu-Carbon composite was drastically improved compared with Si, and its Si utilization was much higher than that of Si-Carbon composite.

3:30 PM
Effect of Strain Rate and Environment on the Mechanical Behavior of the Ni-19Si-3Nb-0.15B-0.1C Intermetallic Alloy at High Temperature: *Shian-Ching Jason Jang*[1]; [1]I-Shou University, Dept. of Matls. Sci. & Engrg., 1, Sec. 1, Shiuecheng Rd., Dashu Shiang, Kaohsiung County 840 Taiwan

The effect of strain rate and environment on the mechanical behavior at different temperature for the Ni-19Si-3Nb-0.15B-0.1C was investigated by atmosphere-controlled tensile test in various conditions (different strain rate at different temperature). The results revealed that the Ni-19Si-3Nb-0.15B-0.1C alloy exhibits ductile mechanical behavior (UTS ~ 1250 MPa, ?Õ ~ 14%) at temperature below 873K in vacuum (2 x 10¡V4 torr), in air, and in water vapor (850 ppm water vapor) atmosphere. This indicates that the microalloying of boron and carbon does overcome the environmental embrittlement of water vapor at the testing temperature below 873K for the Ni-19Si-3Nb base alloy. However, the ductility of the Ni¡V19Si-3Nb-0.15B-0.1C alloy tested in air and vacuum drops significantly when the temperature increases to 973 K. In addition, the strain rate effect on the tensile strength of Ni¡V19Si-3Nb-0.15B-0.1C alloy was revealed insensitive over a wide temperature from room temperature to 1073 K. The detail of mechanical behavior related to the strain rate and temperature will be discussed in this paper.

4:00 PM Break

4:20 PM
Temperature Dependence of Delayed Hydride Cracking Velocity in Ti-6Al-4V Alloy: *Young Suk Kim*[1]; Seung Jun Choi[1]; Kyung Soo Im[1]; [1]Korea Atomic Energy Research Institute, Zirconium Team, PO Box 105, Yuseong, Daejeon 305-353 Korea

The Ti-6Al-4V sheet with the -Ti and the phase was subjected to electrolytic charging of 1000 ppm H and to constant load tests at temperatures varying from 10°C to 200°C. The delayed hydride cracking velocity (DHCV) was determined using compact tension specimens with the pre-fatigued crack growing along the rolling direction of the sheet. The DHCV of the Ti-6Al-4V alloy decreased with the test temperature increasing from 20 to 100°C and also with the test temperature decreasing below the zero temperature. This fact is quite in contrast with the DHCV of Zr-2.5Nb alloys that increases with the increasing temperature from RT to 300°C and decreases at temperatures in excess of 300°C. Based on the Kim's DHC model where the driving force for the DHC is a supersaturated hydrogen concentration or C over the terminal solid solubility of hydrogen for dissolution arising from a hysteresis of the terminal solid solubility of hydrogen, we discussed the different temperature dependency of the DHCV for the Ti-6Al-4V alloys and the Zr-2.5Nb both of which have the microstructure of the - and -phases.

4:50 PM
Performance Optimisation of Gas Turbine Power Output and Thermal Efficiency With F-Technology System: *Rama Rao Adapa*[1]; D. N. Reddy[2]; K. V. Sharma[3]; [1]VNR VJIET, Mechl. Engrg., Bachupally, Kukatpally, Hyderabad, Andra Pradesh 500 072 India; [2]Osmania University, Mechl. Engrg., Coll. of Engrg., Hyderabad, Andra Pradesh 500 007 India; [3]JNTU, Ctr. for Energy Studies, Coll. of Engrg., Hyderabad, Andra Pradesh 500 072 India

The performance optimization of Gas Turbine (GT) with F-Technology System is presented that yields higher thermal efficiency and higher power output than the existing G.T.Power Plants. F-Technology means to increase the Fire Point in the combustion chambers in G.T. which in turn increases the Turbine Inlet temperature thereby the Heat Energy (HE) converted into Kinetic Energy (KE) on the G.T. Nozzles and K.E. is converted into Mechanical Energy (ME) on the G.T.Rotor Blades which results in rotating the Generator Rotor thereby producing the higher power output and thermal efficiency. To withstand the higher temperature in the hot zones in the G.T. Sections Hastalloy material in combustion chambers, FSX 414 for G.T. Nozzles, IN 706 for G.T. hot zone parts, ASTM A 336 for shrouds, and Ni Cr MO forged steel for all the G.T. Rotor discs are preferred. Performance, calculations being done to get optimized results with relevant sources.

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Tuesday PM Room: 3008
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: S. Joseph Poon, University of Virginia, Dept. of Physics, Charlottesville, VA 22904-4714 USA; Jean-Marie Dubois, Ecole des Mines de Nancy, Lab. de Sci. et Génie des Matériaux et de Métall., Nancy 54042 France

2:00 PM Invited
Electronic Structure of Complex Hume-Rothery Phases in Transition-Metal Aluminides: *Guy Trambly de Laissardière*[1]; Duc Nguyen-Manh[2]; Didier Mayou[3]; [1]Université de Cergy-Pontoise, Lab. Physique Théorique et Modélisation, St. Martin, 2 av. A Chauvin, Cergy-Pontoise 95302 France; [2]UKAEA Fusion, Culham Sci. Ctr., Abingdon OX1 430B UK; [3]CNRS, Lab. D'Etudes des Propriétés Electroniques des Solides, BP 166, Grenoble, 38042 France

The discovery of quasicrystals phases and approximants in Al-Mn system has revived the interest of complex aluminides containing transition-metal atoms. On the one hand, it is now accepted that the Hume-Rothery stabilization plays a crucial role. On the other hand, TM atoms have also a very important effect on their stability and their physical properties. But, until recently, there has been no model taking into account these two aspects together. In this paper, we review a model that unifies the classical Hume-Rothery stabilization for sp electron phases with the virtual bound state model for transition-metal atoms embedded in the aluminum matrix. This new theory for "spd electron phases" is applied successfully to Al(Si)-transition-metal alloys and it gives a coherent picture of their stability and physical properties. It is compared to first-principles calculations of the electronic structure and experimental results.

2:30 PM Invited
Hume-Rothery Rules for Stable Quasicrystals: *An Pang Tsai*[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsch. for Advd. Matls., Katahira 2-1-1, Sendai 980-8577 Japan

The stability of the icosahedral quasicrystals has been studied in terms of Hume-Rothery rules. We have confirmed that most of stable quasicrystals cann be understood within the frame work of the Hume-Rothery rules. Even more interestingly, it is found that stable quasicrystals are strict electron compounds, which only form at sharp valence electron concentration. All of the quasicrystals commonly have the corresponding valence electron concentration, but the dependence of the stability on atomic size factor is different among the groups. Qualitatively, the most relevant criterion for the formation of stable quasicrystals is that the alloy should have a definite valenceelectron concentration. On top of this, a high stability of the quasicrystal is observed when the atomic size facto is favored.

3:00 PM Break

3:20 PM Invited
Hume-Rothery Rule as a Formation Condition of New Icosahedral Quasicrystals: *Tsutomu Ishimasa*[1]; [1]Hokkaido University, Div. of Applied Physics, Kita 13-jou, Nishi 8-chome, Kita-ku, Sapporo, Hokkaido 060-8628 Japan

In the past four years, several new quasicrystals have been discovered in Zn-, Cu-, Cd-, and Ag-In- based alloys. They belong to new family of quasicrystals having a unique type of atomic cluster as a local structural unit, which is different from those in the Al-based Mackay-type and the Zn-based Bergman-type quasicrystals. Many quasicrystals belonging to this family are thermodynamically stable and exhibit remarkable high degree of structural perfection, for example in the cases of Zn-M-Sc where M denotes Mg, Fe, Co, Ni, Pd and Ag. The purpose of this presentation is to review the structural properties, in particular structural relationship to the corresponding crystalline approximants, and the formation conditions of this unique type of quasicrystal. The role of Hume-Rothery rule as a formation condition is an interesting and important subject.

3:50 PM Invited
On the Formation of Quasicrystals in Zr-Based Metallic Glasses: *Uwe Koster*[1]; [1]University of Dortmund, Dept. Biochem. & Chem. Eng., Dortmund D - 44221 Germany

In a large number of metallic glasses quasicrystals form first upon devitrification. This indicates a strong influence of the short-range order in the melt, quenched-in into the metallic glass. Observations on the influence of the quenching temperature on quasicrystal formation in melt-spun Zr-Cu-Ni-Al glasses as well as on the influence of different routes of glass formation on the phase selection in Zr-Pd confirm such an assumption. Formation of quasicrystals is explained by various arguments: Quasicrystals are often considered as Hume-Rothery phases, atomic size or bond strength dependent structures, or hybrid structures built e.g. in Zr-Cu-Ni-Al glasses from Al_2Cu- and $MoSi_2$-type building blocks. In the discussion on quasicrystal formation in Zr-based metallic glasses melt temperature, quenching rate, oxygen or hydrogen contamination, alloying with small amounts of additional elements or the exchange of the late transition metals are assumed as ruling parameter for phase selection as well as their nucleation and growth rates.

4:20 PM Invited
Phase Equilibria and Thermodynamics of Ca-Based Metallic Glasses: Michael C. Gao[2]; *Gary J. Shiflet*[1]; S. Joseph Poon[3]; Marek Mihalkovic[4]; Mike Widom[4]; [1]University of Virginia, PO Box 400745, 116 Engr.'s Way, Charlottesville, VA 22904-4745 USA; [2]Northeastern University, Dept. of Physics, Boston, MA 02115 USA; [3]University of Virginia, Dept. of Physics, Charlottesville, VA 22904-4745 USA; [4]Carnegie Mellon University, Dept. of Physics, Pittsburgh, PA 15213 USA

The CALPHAD method has become a powerful tool in thermodynamic modeling of multi-component systems. However, when there is little literature information available, critical experiments are needed to validate the CALPHAD modeling. This is the case for the Al-Ca-Cu ternary phase diagram (especially on the Ca-rich side). Research in this system originates from the recent discovery, at the University of Virginia, that an unusually broad glass formation range (GFR) has been identified near the Ca-rich side that includes several bulk glass chemistries. This is in contrast to the narrow GFR reported earlier with much poorer glass forming ability on the Al-rich side. The GFR was determined using melt-spinning and die-casting techniques, and its structural and thermal stability was studied using XRD and DSC. The solid-state phase equilibria were investigated using XRD, SEM, EBSD and TEM, while DTA was used to characterize the solidus and liquidus temperatures. In order to assist this study, first-principles calculatio ns were performed on this system and have proven to be important in prediction of phase diagrams (e.g., solubility range) and minimizing experimental uncertainty, including phase chemistry and structure. In this talk, details on integration of these calculations with experiments and modeling will be emphasized. The application of such self-consistent accurate thermodynamic descriptions to explain the observed glass formation will be discussed.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Electromigration, and Electrical "Aging" of Lead-Free Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Tuesday PM — Room: 3014
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Laura J. Turbini, University of Toronto, CMAP, Toronto, ON M5S 3E4 Canada; Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Engrg., Hsinchu 300 Taiwan

2:00 PM Invited
The Effect of Temperature on Conductive Anodic Filament Formation: *Antonio Caputo*[1]; Laura J. Turbini[1]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Toronto, ON M5S 3E4 Canada

Conductive Anodic Filament (CAF) is a failure mode in printed wiring boards (PWBs) which occurs under high humidity and high voltage gradient conditions. The filament, a copper salt, grows from anode to cathode along the epoxy-glass interface. The rate limiting step is delamination of the epoxy-glass interface. This can occur due to mechanical stress or thermal stress associated with CTE mismatch between the polymer and reinforcement in the board. The move from lead-based (201-205°C) to lead-free soldering conditions (240-245°C) will accelerate epoxy-glass delamination and therefore enhance CAF growth. Bent and Turbini have shown that PWBs exposed to lead-free processing conditions have a greater incidence of CAF failure than those exposed to conventional processing temperatures. This paper presents data on new "halogen free" laminate materials being developed for lead-free soldering temperatures. It will evaluate their CAF susceptibility at both lead and lead-free soldering conditions.

2:30 PM
3-D Current Density Simulation in Flip Chip Solder Joint Under Electrical Current Stressing: *T. L. Shao*[1]; Shih-Wei Liang[2]; Chih Chen[1]; [1]National Chiao Tung University, Matl. Sci. & Engrg., 1001 Ta-Hseuh Rd., Hsin-Chu 30050 Taiwan; [2]National Chiao Tung University, Dept. of Applied Math., 1001 Ta-hseuh Rd., Hsin-Chu 30050 Taiwan

3-D current density distribution of the flip chip solder joint under electrical current stressing was simulated by finite element method. The current density distribution inside the flip chip joint was examined under 0.567 ampere current stressing. Four kinds of under bump metallurgies (UBM) structures, including the 0.7 micron Cu/0.3 micron Cr-Cu/0.1 micron Ti thin film UBM, 5 micron Cu UBM, 5 micron Ni UBM, and 3 micron Ni/5 micron Cu UBM, were investigated for the flip chip solder joints. The maximum current density, which ranges from 41,740 to 187,492 A/cm2, always occurs at the entrance of Al trace into the flip chip solder joint. The maximum current crowding ratio of the flip chip solder bump varies from 19.62 to 88.12. The flip chip solder bump of thin film UBM has the largest value of current density and current crowding ratio.

2:50 PM
In Situ Observation of Flip-Chip Solder Joints Under Current Stressing: *Chia Ming Tsai*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan

The effect of electron flow on the motion of Pb phase in flip chip solder joints was studied at room temperature. The solder joints had a diameter of 100 microns. The UBM (under-bump metallization) on the chip side had a Cu metallurgy, and the surface-finish on the substrate side had the Au/Ni metallurgy. In order to have in-situ observa-

tion, the samples were cross-sectioned through the centers of the solder joints before current stressing. The current density through the solder was about 3.1×10^4 A/cm^2. The dominant diffusing species was found to be Sn, as Pb had moved in the opposite direction of electron flow. The locations of the Pb grains were traced, and thus the motions of these grains were monitored. The nonuniform motion behavior of the Pb grains may correspond to the distribution of electron flow, which depends on the geometry of solder bump. The electronmigration flux and the effective charge were calculated.

3:10 PM
Effect of Current Crowding on the Failure Mechanism of Flip Chip Solder Joints Under Current Stressing: *Yen-Liang Lin*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan

The effect of current crowding on the flip chip solder joints during current stressing was studied. The solder was 63Sn37Pb eutectic solder, and the solder joints had a nominal diameter of 125 microns. In these joints, the UBM on the chip had a Cu/Ni/Al multiplayer structure, and that on the board side had a very thick Cu metallurgy. During current stressing, the flip chip packages were kept in an oven set at 150°C. The failure always occurred in joints that had electrons flowing from chip to substrate. Evidence of local melting was also observed. Some of the flip chip packages were placed at room temperature in order to perform in situ observation. In these in situ experiments, voids occurred in joints that had electrons flowing from chip to substrate. These voids grew wider and deeper with current stressing, and evetually could reach a depth of 36 microns.

3:30 PM Break

3:40 PM
The Effect of Applied Current on Sn-Pb Flip Chip Solder Bump: *Yeh-Hsiu Liu*[1]; Kwang-Lung Lin[1]; [1]National Cheng Kung University, Matls. Sci. & Engrg., No. 1, Ta-Hsueh Rd., Tainan 701 Taiwan

The electromigration-induced failure in flip chip solder bump of 5Sn-95Pb was studied after current stressing at 150°C with a current density of 5?~103 A/cm2 for up to 1711 h. The under-bump metallization (UBM) on the chip side was sputtered Al/Ni(V)/Cu thin films and on the board side was electroless Ni/Au surface finished. The diameter of the bumps was about 90 ?Êm. It was shown that the failure of the joints occurred at the chip side (cathode side). Simultaneously, the V layer in the UBM was damaged after current streesing up to 1711 h. Owing to no reaction occurring among V and Cu, Ni or Sn, there is no electromigration in the V layer so that its interface serves as an atomic flux divergence plane. The Cu-Ni-Sn intermetallic compound and void were found near the current crowding area in the chip side. The failure mechanism for 5Sn-95Pb flip chip solder bump will be discussed.

4:00 PM
Threshold Current Density of Electromigration in Pure Tin Films: *Hung-Chih Yu*[1]; Sue-Hong Liu[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Engrg., 1001 Ta Hsueh Rd., Hsin-Chu 30050 Taiwan

Electromigration in pure tin draws a lot of attention due to the implementation of Pb-free solders, in which tin weighs over 96% for most of the solders. Electromigration phenomenon is investigated under the current density of 7.5?~104?A1?~105 and 1.75?~105 A/cm2. The testing temperature were room temperature?A50?• and 75?•. Depletion and hillocks were found at the cathode side and anode side, respectively. The threshold current density at 50?• was measured to be about 4.4?~104 A/cm2 by extrapolating the plot of the applied current density versus drift velocity to zero drift velocity, and the threshold current density was approximately inversely proportional to the testing temperature. Other electromigration parameter, such as activation energy, and effective charge number, can be measured by this technique. They will be presented in details in the conference.

4:20 PM
Effect of Electromigration on Interdiffusion and Failure Mode in Composite Solder Joint: *Annie Tzu-yu Huang*[1]; Jae-Woong Nah[1]; [1]University of California, Matls. Sci. & Engrg., 6532 Boelter Hall, 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA

Combination of the high melting 97Pb-3Sn or 95Pb-5Sn solder on the chip side and the low melting eutectic 37Pb-63Sn solder on the organic substrate side has recently been investigated for applications in high-end devices such as server. Our studies showed that interdiffusion of the high-Pb and the high-Sn solder is minimal during thermal annealing and the composite joint remains stable at a temperature as high as 150°C. However, when the composite solder joint is under current stressing, electromigration occurs and the composite solder joint becomes unstable due to void formation and intermetallic compound transformation. Because the dominant diffusing species (Pb or Sn) differs at different temperatures under electromigration, current stressing temperature also plays a role on failure mechanism in the composite solder joint.

4:40 PM
Study of Coupling Effect by Using Sandwich Structures of Cu/Sn/Various Basis Metals: *Shen-Jie Wang*[1]; *Cheng-Yi Liu*[1]; [1]National Central University, Cheml. & Matls. Engrg., No.300, Jungda Rd., Jhongli, Taoyuan 320 Taiwan

The coupling effect between Cu-Sn and Ni-Sn interfacial soldering reactions have been reported intensively. The interfacial reaction on Ni side would strongly be affected by the interfacial reaction on the Cu side, which further influences the reliability of solder joints. Currently, a practical solder joint structure often contains other metal layers beside Cu and Ni. Therefore, it is very important to study coupling effects for any other two different basis metal bond pads. In this study, Cu/Sn/X metals sandwich structures were used. The X metals are Au, Ag, Pd and so on. The preliminary results did show that the interfacial Cu-Sn reaction was affected simultaneously by the opposite soldering interfacial reaction. In this talk, we will report the coupling effects on the kinetics and morphology of interfacial Cu-Sn reaction.

5:00 PM
Combined Effect of Electromigration and Applied Stress on Solder Joint Failure: *Fei Ren*[1]; Jong-ook Suh[1]; Jae-Woong Nah[1]; King-Ning Tu[1]; Bingshou Xiong[2]; Luhua Xu[2]; John Pang[2]; [1]University of California, Dept. of Matls. Sci. & Engrg., 405 Hilgard, Los Angeles, CA 90095-1595 USA; [2]Nanyang Technological University, Dept. of Mechl. & Production Engrg., 50 Nanyang Ave., Singapore 639798 Singapore

The combined effect of electromigration and applied stress on Pb-free solder joints has been investigated. For tensile and creep tests, samples of Pb-free solder balls with a diameter of 300 ¥ìm were reflowed between two Cu wire-electrodes to form one-dimensional line structures of Cu-solder-Cu. For shear tests, flip chip samples were used. The tensile tests at a strain rate of about 3 ¥ìm/min were conducted after the samples were undergone electromigration at 1~5¡¿103 A/cm2 and at 100~150¨¬C. We observed that the failure always occurs at the cathode interface. Same kind of failure also occurs in a daisy chain of flip chip solder jumps when tested by shear after electromigration. Creep and electromigration were conducted simultaneously using the line samples, with the applied stress at 1 to 15 MPa. The combined effect of electrical and mechanical forces on solder joint failure under these different conditions will be discussed.

5:20 PM
Grain Growth of Tin Under Electromigration Studied by Synchrotron X-Ray Microdiffraction: *Albert T. Wu*[1]; King-Ning Tu[1]; J. R. Lloyd[2]; N. Tamura[3]; [1]University of California, Matls. Sci. & Engrg., 1677 Boelter Hall, Los Angeles, CA 90095 USA; [2]IBM T. J. Watson Research Center, PO Box 218, Yorktown Hgts., NY 10598 USA; [3]Lawrence Berkeley National Laboratory, Advd. Light Source, Berkeley, CA 94720 USA

When a white Sn (â-Sn) thin film stripe is subjected to electromigration, it shows a voltage drop typically about 10%. The reason might be due to the dependence of resistivity on anisotropic crystal structure of â-Sn; the c-axis is about 35% higher than a- and b-axes. Synchrotron x-ray micro-diffraction (~1µm in beam diameter) has been used to achieve grain-by-grain analysis of the changes of grain orientation in the Sn stripe before and after electromigration. Grain growth, with a mechanism which seems different from normal grain growth, was observed under electromigration; the low-resistance grain grew at the expense of a neighboring high resistance grain. We propose a different mechanism of grain growth under electromigration.

Magnesium Technology 2005: Magnesium Alloy Development

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday PM Room: 2004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Alan A. Luo, General Motors Corp, R&D Ctr., Warren, MI 48090-9055 USA; Alok Singh, National Institute for Materials Science, Matls. Engrg. Lab., Tsukuba, Ibaraki 305-0047 Japan

2:00 PM
Magnesium Castings in Aeronautics Applications - Special Requirements: *Achim Wendt*[1]; Konrad Weiss[1]; Arye Ben-Dov[2]; Menachem Bamberger[3]; Boris Bronfin[4]; Nir Moscovitch[4]; [1]RWP GmbH, Am Muensterwald 11, Roetgen 52159 Germany; [2]Israel Aircraft Industries, Tel Aviv Israel; [3]Technion, Haifa Israel; [4]Magnesium Research Institute, Beer-Sheva Israel

Although weight reduction in aircrafts is a fundamental matter use of Magnesium alloys is not widespread in the interior of airplanes due to "traditional" problems with corrosion resistance. In spite of the creation of new Magnesium alloys with improved corrosion resistance, these alloys are mostly designed for automotive industry. Alloys for aerospace industry must combine high performance regarding mechanical properties and corrosion resistance. A review is given on the use of Magnesium in aircrafts in the past. The paper then presents requirements of modern aircraft industry to Magnesium castings regarding to mechanical properties as well as corrosion properties. Today's drawback is the lack of a sufficient number of available alloys fulfilling the needs of modern aerospace industry. The methodology used in an international research project (IDEA) to develop new magnesium alloys for special use in aircrafts is described as well as the experimental and virtual methods utilised in this development.

2:20 PM
Effects of Aluminum Addition on Properties of Magnesium-Lithium Alloys: *Hsin Chin Lin*[1]; Kun Ming Lin[2]; Hsin Man Lin[2]; Cheng Hsiu Yang[2]; Ming Tang Yeh[3]; [1]National Taiwan University, Dept. of Matls. Sci. & Engrg., No.1, Sec. 4, Roosevelt Rd., Taipei 106 Taiwan; [2]Feng Chia University, Dept. of Matls. Sci. & Engrg., No. 100 Wenhwa Rd., Seatwen, Taichung 40724 Taiwan; [3]Hsu-Yang Technologies Co. Ltd, R&D Ctr., 80 Erh-Chia Rd., Ying-Keh, Taipei-Hsian Taiwan

The products of Mg alloys are mainly manufactured by using the die-casting because of their poor formability. However, the high ratio of defect products in die-casting of Mg alloys will reduce their manufacturing efficiency. Therefore, the developments of new Mg alloys with high formability of rolling, pressing and forging are important issues to improve the manufacturing ability of thin plates of Mg alloys. The two-phase Mg-Li alloys are reported to have excellent formability. However, their mechanical property and corrosion resistance are not well understood. In this study, we aim to study the microstructure, crystal structure, mechanical behavior and corrosion resistance of these two-phase Mg-Li alloys. The effects of Al addition on the Mg-Li alloy are also investigated. Preliminary results reveal that Al addition will effectively improve the mechanical property and corrosion resistance of two-phase Mg-Li alloys.

2:40 PM
Effects of Al Addition on Microstructure and Mechanical Properties of Mg-Zn-Y Alloys: *H. K. Lim*[1]; J. Y. Lee[1]; W. T. Kim[2]; D. H. Kim[1]; [1]Yonsei University, Ctr. for Non-Crystalline Matls., Dept. of Metallurgl. Engrg., 134 Shinchon-dong, Seodaemun-gu, Seoul 120-749 S. Korea; [2]Cheongju University, Dept. of Applied Sci., 36, Naedok-dong, Sangdang-gu, Cheongju, Chongbuk 360-764 S. Korea

Recently, efforts on quasicrystal reinforced magnesium alloys have been focused in order to overcome the disadvantages of magnesium alloys. The quasicrystal reinforced Mg-9Zn-2Y (in wt. %) alloy has been reported to exhibit a combination of high strength and ductility, and good formability at high temperature.[1] In the present study, we investigated the variation of microstructures and mechanical properties when the Zn/Y ratio from 4.5 to 2 (Mg-9Zn-2Y, Mg-6Zn-2Y and Mg-4Zn-2Y) and Al is added in Mg-Zn-Y alloy (Mg-(9-x)Zn-4Al-2Y (x=0, 3, and 5)). The microstructural dependence upon composition variations of Mg-Zn-RE alloys is discussed in terms of structural identification and the corresponding mechanical properties. [1]D.H. Bae, S.H. Kim, D.H. Kim, W.T. Kim, Acta Mater. 50 (2002) 2343.

3:00 PM
The Behavior of CaO in Magnesium Alloys: Jin-Kyu Lee[1]; Won Ha[2]; *Shae K. Kim*[1]; Young-Jig Kim[2]; [1]Korea Institute of Industrial Technology, Advd. Matl. Ctr., 994-32 Dongchun-dong, Yeonsu-Gu, Incheon 406-130 Korea; [2]Sungkyunkwan University, Dept. of Advd. Matl. Engrg., 300 Chunchun-dong, Jangan-gu, Suwon, Gyounggi-do 440-746 Korea

Mg alloys present a number of interesting properties, such as high specific strength, good castability, low density, etc. Despite these properties, Mg alloys are used with many precautions of high chemical reactivity, limited high strength and creep resistance at elevated temperatures. Research has been directed to improve these properties by alloying and/or composites manufacturing. It is well known that Ca, though its high cost, is effective for oxidation resistance and particle reinforced Mg composites have desirable high mechanical properties. The aim of this study is to manufacture CaO particle reinforced Mg composites in terms of improving oxidation resistance and mechanical properties. The Mg composites were manufactured by RCM (Rotation Cylinder Method) and the effects of CaO on oxidation resistance, fluidity and mechanical properties of Mg composites were investigated.

3:20 PM
The USCAR Structural Cast Magnesium Development Project: *Naiyi Li*[1]; Richard Osborne[2]; Bruce Cox[3]; Donald Penrod[4]; [1]Ford Motor Company, Mfg. & Processes, 2101 Village Rd., MD3135, Dearborn, MI 48124 USA; [2]General Motors Corporation; [3]DaimlerChrysler Corporation; [4]Manufacturing Services & Development, Inc.

The Structural Cast Magnesium Development Project is a jointly sponsored effort by the US Department of Energy (DOE) and the US Council for Automotive Research (USCAR) to identify and resolve technical and manufacturing issues that limit the light weighting opportunities of applying large-scale structural cast magnesium automotive components. This project, starting at the end of year 2001, comprises General Motors, Ford, DaimlerChrysler and thirty-four other North America companies and organizations to set its overall objective to determine the technical feasibility and practicality of producing and implementing an one-piece front engine cradle casting. This paper provides an overview of the project scopes, magnesium technology development, scientific understanding and up-to-date accomplishments. The front engine cradle made of AE alloy is introduced in various aspects including magnesium alloys property database development, component design and analysis, corrosion protection and coating strategy.

3:40 PM Break

3:55 PM
The Role of Rare Earth Elements in Structure and Property Control of Magnesium Die Casting Alloys: *Per Bakke*[1]; Hakon Westengen[1]; [1]Hydro Aluminium, Mg Competence Ctr., PO Box 2560, Porsgrunn N-3908 Norway

The performance of magnesium die cast parts is governed by the microstructure and by the distribution of structural features which occur as a result of the chemical composition and processing history of the alloy. The elevated temperature properties, especially mechanical strength under creep conditions, are primarily determined by the grain structure, the elements in solid solution and the effectiveness of second phase particles in stabilizing the grain boundaries. The current emphases in alloy development focus on the utilization of elements with low solubility in the solid state, leading to the formation of stable precipitates during solidification. Such elements include the rare earths, as well as silicon, strontium and calcium. A detailed analysis of the various microstructural features and attributes is given for a new family of rare earth-containing alloys. The optimization of alloy composition is addressed in terms of blending advantageous microstructural characteristics with phase equilibria considerations.

4:15 PM
Assessment of Superheating in Mg-Al Alloys by Thermal Analysis: *Peng Cao*[1]; Qian Ma[2]; David H. StJohn[1]; [1]University of Queensland, CRC for Cast Metals Mfg., Sch. of Engrg., St. Lucia, Queensland 4072 Australia; [2]Brunel University, Brunel Ctr. for Advd. Solidification Tech., Uxbridge, Middlesex, London UB8 3PH UK

The superheating grain refinement of Mg-9%Al alloy has been assessed by thermal analysis. The results obtained from the thermal analysis showed that after superheating nucleation temperature in-

creased while nucleation undercooling decreased. The grain size of the Mg-9%Al alloys decreased significantly after superheating. Microscopic examination showed that the particles present in the samples contained Al and Fe, no matter whether these samples were superheated or not. It is suggested that superheating causes grain refinement by adjusting the shape, size or distribution of the nucleant particles, rather than changing the composition of the nucleant particles.

4:35 PM
The Influence of Alloying Elements Upon Properties of Elektron 21 Alloy: *Paul Lyon*[1]; *Timothy E. Wilks*[1]; [1]Magnesium Elektron, TSD, PO Box 23, Swinton, Manchester M27 8DD England

Elektron 21 (ASTM Designation EV31) is a Mg-RE-Zn-Zr alloy designed for Aerospace and Speciality Applications. The alloy is capable of operating up to approximately 200C (400F). To achieve successful applications, Elektron 21 has had to exhibit not only good mechanical properties but also good corrosion performance and ease of castability. When developing the alloy, the effect of the Rare Earth (RE) constituents and Zinc were optimised to achieve these requisite alloy characteristics. Changes in the Rare Earth and Zinc content affect age hardening response. Control of individual Rare Earth components also influences castability. The level of Rare Earth component and, more importantly Zinc content, must be limited and controlled to achieve good and consistent corrosion performance. Finally, the effects of heat treatment variables were assessed to ensure that they are as broad as possible to aid produceability.

4:55 PM
The Effects of Grain Refinement on the Castability of Magnesium Permanent Mould Castings: *Timothy Loughnane*[1]; Deliang Zhang[2]; Darius P.K. Singh[1]; Thomas Neitzert[2]; [1]ION Automotive Ltd, Light Metal Castings Div., Auckland New Zealand; [2]University of Waikato, Dept. of Matls. & Process Engrg., Hamilton New Zealand

A gravity filled permanent mould casting has been designed to simulate the geometry and feeding habits of a typical automotive wheel. The casting geometry reflects the areas of a wheel casting that are most prone to defect formation, that is, the hub and the transition from the spoke to the rim where hot spots are often experienced. Different magnesium alloys were trialled along with various melt additions to facilitate the refinement of the microstructure during solidification. The effect of grain refinement on the castability of the alloys was investigated by measuring the resulting grain sizes, porosity levels, porosity types, as well as levels of hot tearing and surface slumping defects in the castings produced. It is expected that the conclusions drawn from this, and future investigations can be applied to the permanent mould casting of magnesium alloy wheels. The aim of this paper is to present and discuss the results of the investigation to date.

5:15 PM
Modern Melting Devices for Mg-Alloy for Die Casting Industry (Hot and Cold Chamber): Christoph Scheer[1]; *Roger Rapp*[1]; [1]MELTEC Industrieofenbau GmbH, Guggenberg 15-17, Kirchham A-4656 Germany

Modern melting devices for Mg-Alloy for die casting industry (Hot and Cold Chamber) – consisting of preheating, automatic charging, melting and gas protection for the molten Mg. In particular about accurate and safe dosing of Mg-Alloy.

Materials Processing Fundamentals: Smelting & Refining II

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Tuesday PM Room: 3001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

2:30 PM
Heat Pipe Cooling of a Slag Tapblock: *Pietro Navarra*[1]; Hujun Zhao[1]; Frank Mucciardi[1]; Daniel Cheret[2]; Peter Verguts[2]; Karel Verscheure[3]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., Wong Bldg., Rm. 2160, Montreal, Quebec H3A 2B2 Canada; [2]Umicore Research, Dept. Pyro-Metall., Kastelstraat 7, Olen B-2250 Belgium; [3]K.U. Leuven, Afd. Chemische Materiaalkunde, Kasteelpark Arenberg 44, Heverlee B-3001 Belgium

The proximity of operators to copper tapholes in the metallurgical industry has caused a drive to seek an improved cooling mechanism vis-à-vis forced convection water cooling, which can fail catastrophically in the unlikely event of a failure. The purpose of the research presented is to develop an industrial-scale copper tapblock using novel heat pipe technology, which is fundamentally safer than conventionally cooled tapblocks. A tapblock cooling system incorporating two independent heat pipes was designed and assembled at Umicore Research in Belgium, each containing a total of 4 kg of water. Pilot tests achieved a heat load of 140 kW into a single heat pipe, with the potential for an 80% reduction in cooling water requirements compared to a forced convection system. Modeling indicates a maximum local heat extraction rate of 2.4 MW/m^2, with no indication of film boiling. The dry-out limitation was reached during testing and was subsequently circumvented.

2:55 PM
Modelling for Viscosities of Imperial Smelting and Lead Smelting Slags: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

A program 'SlagVis' has been developed to simulate the viscosities of Imperial Smelting slag and lead smelting slag, combined with a thermodynamic database and the mathematical model of slag viscosities based on a modified Urbain formalism. The modelling results have been validated by a number of measurements. The effects of slag compositions on the liquidus temperature and viscosities of Imperial Smelting slag have been simulated and discussed.

3:20 PM Cancelled
Investigation on the Synthesis of Titanate Barium in Molten NaOH-KOH by the A. C. Impedance Technique

3:45 PM
Preparation of Monodispersed NiO Precursor Particles by Induced Precipitation of Solid Oxalate Acid: *Huang Kai*[1]; Guo Xueyi[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

By adding the solid oxalate acid powder into the solution of nickel chloride or its ammonia coordinated solution, the uniform compound particles were prepared and the precipitation particles were transferred to be mono-dispersed NiO particles by calcination. In the study, the various experimental conditions were investigated and the characteristics of the corresponding obtained particles were analyzed, and the formation mechanism of the particles was elucidated. It was found that the solid oxalate acid provided the solid-solution interface for the precipitation and induced the nucleation and growth of the product particles.

4:10 PM Break

4:25 PM
Thermodynamic Modeling of Desilverising of Lead Bullion by Zinc During Lead Refining: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

A thermodynamic model and database has been developed to model the desilverising of lead by the reagent zinc during lead refining, whereby silver-zinc intermetallic compounds is formed, and floats out of the dissolved zinc/silver in the lead bath. The model predictions have been validated by the industrial data. The solubility of silver-zinc intermetallic compounds in lead decreases with falling temperature. A number of parameters of the Davey desilverising process are modeled, and applied to a wide range of silver contents in lead bullions in the industry.

4:50 PM
Optimization of the Pig Iron Desulfurization Inside a Torpedo Car by Physical Modeling Techniques: *Varadarajan Seshadri*[1]; Ildeu Alves Souza[2]; Carlos Antonio Silva[3]; Itavahn Alves Silva[3]; Versiane Albis Leão[3]; Diego Canez Fernandes[3]; [1]Universidade Federal de Minas Gerais, Metallurgl. Engrg., Rua Espirito Santo, 35, Belo Horizonte, Minas Gerais Brazil; [2]Belgo Mineira, Joâo Monlevade, Minas Gerais Brazil; [3]Universidade Federal de Ouro Preto, Escola de Minas/Engrg. Metalúrgica, Campus do Morro do Cruzeiro, Ouro Preto, Minas Gerais 35400-00 Brazil

A 1:6 torpedo car model was built in order to assess the refining conditions during pig iron desulfurization. The industrial conditions

are those used at Belgo-Mineira, João Monlevade, Brazil. At this plant the refining is made by injection of a CaO-CaF2-Mg powder. Similarity was accomplished by following criteria devel-oped from Froude and turbulent Reynolds groups. Mixing times were evaluated by pulse tracer addition and the kinetics of metal/slag interactions by assessing the iodine transfer from kerozene to water. The influence of parameters such as level of metal, gas flowrate, lance penetration, lance orientation was evaluated.

5:15 PM
Synthesis of Uniform Cobalt Oxalate Particles in the Micro-Fluid Tubular Reactor: *Huang Kai*[1]; Guo Xueyi[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Micro-fluid tubular reactors with the inner diameter of 2mm and 3mm were used to synthesis the cobalt oxalate particles from ammonia oxalate and cobalt chloride solutions. Influences of the characteristic size of the tubular reactor, residence time, and reaction temperature on the particle properties were studied. SEM, XRD, TG-DTA, and Size analyzer were used to characterize the particles. The results of this study is helpful for the preparation of uniform particle with high value products obtained from precipitation process.

Mechanical Behavior of Thin Films and Small Structures: Fatigue, Fracture, and Reliability of MEMS and Thin Structures I

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Tuesday PM Room: 2024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brad L. Boyce, Sandia National Laboratories, Dept. 1851, Albuquerque, NM 87185 USA; Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA

2:00 PM Invited
A Mechanistic Understanding of Fatigue in Polysilicon Structural Thin-Films: *Robert O. Ritchie*[1]; Daan Hein Alsem[1]; Eric A. Stach[2]; Christopher L. Muhlstein[3]; [1]University of California, Matls. Sci. & Engrg., One Cyclotron Rd., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., Berkeley, CA 94720 USA; [3]Pennsylvania State University, Matls. Sci. & Engrg., 310 Steidle Bldg., Univ. Park, PA 16802 USA

While bulk silicon is not susceptible to fatigue failure, silicon micron-scale structural thin films are. This has important implications for microelectromechanical systems (MEMS) applications, where parts are subjected to a large number of loading cycles with amplitudes below their (single-cycle) fracture stress, which may arise due to vibrations intentionally induced in the structure (i.e. a resonator) or those which arise from the service environment. While the system-reliability of MEMS has received increasing attention, the physical mechanisms responsible for these failure modes have yet to be conclusively determined. This is particularly true for fatigue, where the mechanisms have been subject to intense debate. Our past studies have shown that fatigue is a result of a "reaction-layer" process, whereby high stresses induce a thickening of the post-release oxide at stress concentrations - such as the root of a notched cantilever beam - which subsequently undergoes moisture-assisted cracking. More recent results obtained in high vacuum (~2.0×10^{-7} mbar) reveal absolutely no evidence of premature fatigue failures (i.e. no fatigue failure in vacuo and no oxide thickening). Moreover, fatigue tests conducted in ambient air on polysilicon samples from a more recent fabrication run confirm that the fatigue behavior and oxide-layer thicknesses observed in earlier experiments were not an artifact of that particular fabrication run. We believe that both of these results add further confirmation to the "reaction-layer" mechanism for fatigue of micron-scale polysilicon.

2:25 PM
Long-Term Reliability of Single-Crystal Silicon Thin Films: The Influence of Environment on the Fatigue Damage Accumulation Rate: O. N. Pierron[1]; *C. L. Muhlstein*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & the Matls. Rsch. Inst., 310 Steidle Bldg., Univ. Park, PA 16803 USA

The fatigue behavior of single-crystal silicon thin films tested in various environments (300.1 °C at 251, 401 or 501% relative humidity (R.H.), medium vacuum, and medium vacuum with pre-bake) and fully-reversed loading conditions at high frequency (~40 kHz) was investigated in this study. The fatigue characterization structures, consisting of a notched cantilever beam attached to a plate-shaped mass, were electrostatically actuated at resonance. The resonant frequency was periodically monitored, and its evolution during cycling could be used for interpretation of fatigue damage accumulation. Particularly, damage accumulation rate and therefore fatigue life were strongly affected by both relative humidity and stress amplitude. Fracture surface examination showed highly localized, distinct features in long-life fatigued specimens tested in both air and vacuum that were not found in short-life fatigued specimens. The reaction-layer fatigue mechanism, implying a process of sequential, mechanically-induced oxidation and environmentally-assisted cracking of the surface oxide layer, appears to be the most appropriate to fit this experimental evidence.

2:40 PM
An Electron Microscopy Study of Wear in Polysilicon Microelectromechanical Systems: *Daan Hein Alsem*[1]; Eric A. Stach[2]; Michael T. Dugger[3]; Robert O. Ritchie[1]; [1]University of California, Matls. Sci. & Engrg., One Cyclotron Rd., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., MS 72/150, Berkeley, CA 94720 USA; [3]Sandia National Laboratories, Matls. & Process Scis. Ctr., PO Box 5800, Albuquerque, NM 87185 USA

Wear is an important failure mechanism in microelectromechanical systems (MEMS) and is heavily affected by the tribological properties of the structural materials used. The most widely used material in MEMS is silicon, which is a brittle material. Because of the use of mostly ductile materials in macro-scale mechanical applications, a majority of bulk wear models focus on wear in these type of materials and can therefore not automatically be used for MEMS reliability. Surface characterization of silicon structural thin films has received a growing amount of attention and some cautious attempts to propose micron-scale wear models have been made. However, the exact physical mechanisms for micron-scale wear in silicon have yet to be conclusively determined. We have used polycrystalline silicon side-wall test specimens in combination with electron microscopy to study active mechanisms in sliding wear. In-situ experiments in the scanning electron microscope were performed, as well as transmission electron microscopy (TEM) of worn MEMS parts. After running the devices, worn parts were prepared for TEM using a dual-beam focused ion beam system. Analytical TEM was performed on wear debris and worn parts to investigate their morphology and microstructural changes. Debris particles were observed varying in size from below 100 nm to greater than 600nm and consist completely of silicon-dioxide. Furthermore, both ploughing wear tracks and surface cracks perpendicular to the wear direction were found, suggesting that fracture and fatigue of surface asperities could play an important role.

2:55 PM Invited
Fatigue Failure of Metal Thin Films in MEMS: *Oliver Kraft*[1]; [1]Forschungszentrum Karlsruhe, Inst. für Materialforschung II, Postfach 3640, Karlsruhe 76021 Germany

Continuous and patterned metal thin films with thicknesses well below 1 µm are widely used in micro-electro-mechanical systems (MEMS). Applications range from reflective coatings in micro-optics to current carrying metallizations in sensors or actuators. In these applications, thin film materials are often stressed in a cyclic manner with loading frequencies ranging from well below 1 Hz, e.g. related to usage cycles, up to GHz, e.g. in communication devices. In this paper, the effect of fatigue failure as a result of mechanical or thermomechanical cyclic loading conditions will be reviewed. Damage has been observed to consist of extrusions, voids, and cracks where details of the damage morphology depend on loading conditions, e.g. frequency and temperature, as well as film thickness and microstructure. Further, a general trend has been found that fatigue lifetime increases with decreasing film thickness. The insights gained from

these studies into lifetime prediction and mechanism of fatigue in thin film devices will be discussed.

3:20 PM
Nanoscale Wear Testing of LIGA Nickel Samples: *Neville R. Moody*[1]; John M. Jungk[2]; Marion S. Kennedy[3]; Soumari V. Prasad[4]; Thomas E. Buchheit[4]; David F. Bahr[3]; William W. Gerberich[2]; [1]Sandia National Laboratories, PO Box 969, MS9409, Livermore, CA 94551-0969 USA; [2]University of Minnesota, Minneapolis, MN 55455 USA; [3]Washington State University, Pullman, WA 99164 USA; [4]Sandia National Laboratories, Albuquerque, NM 87158 USA

Strength, friction, and wear are dominant factors in the performance and reliability of materials and devices fabricated using nickel based LIGA and silicon based MEMS technologies. However, the effects of frictional contacts and wear are not well-defined. We have therefore begun a program employing nanoscratch and nanoindentation on electrodeposited LIGA nickel samples. Nanoscratch techniques were used to generate wear patterns as a function of load and number of passes. Nanoindentation was then used to measure properties in each wear pattern. The results show a systematic increase in hardness with applied load with surprisingly little effect of repeat passes on sample deformation. In this presentation, we will use measured hardness values and Johnson's cavity model for plasticity to show how flow stress and the extent of plasticity evolve under sliding contacts. The authors gratefully acknowledge the support of Sandia National Laboratories under contract DE-AC04-94AL85000.

3:35 PM Break

3:50 PM
Fatigue of Electrodeposited Nickel Films: M. C. Zapata[1]; A. Minor[2]; *C. L. Muhlstein*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & Matls. Rsch. Inst., 310 Steidle Bldg., Univ. Park, PA 16803 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA USA

The fatigue resistance of the metallic thin films used in microelectronic circuits, microelectronic packages, and microelectromechanical systems (MEMS) can be a fundamental limitation to product performance. However, our fundamental understanding of how metallic thin films degrade under fatigue loading conditions is limited. In this work the stress-life (S/N) fatigue behavior of a 25mm-thick electrodeposited nickel film was evaluated by mechanical testing of subsized samples under tension-tension loading conditions. The fracture surfaces and dislocation substructure of the failed samples were then evaluated using electron microscopy. The ultrafine grained (i.e., grain sizes <100 nm) nickel exhibited extraordinarily high strength (s_{ult} >1.5 GPa) and its fatigue resistance was consistent with the trends for nickel films established in the literature. The implications of the electron microscopy observations in understanding the fatigue mechanisms will be discussed.

4:05 PM
Roles of Surface Oxide and Morphology in LIGA Ni Fatigue Behavior: Yong Yang[1]; *Jikou Zhou*[1]; S. Allameh[1]; W. O. Soboyejo[1]; [1]Princeton University, Dept. of MAE & PRISM, E-Quad, Olden St., Princeton, NJ 08540 USA

Fatigue life of LIGA Ni MEMS materials has been reported by several groups, but the failure mechanism has never been investigated for such single-layer film materials. In this work, we studied the effects of surface oxide scales on fatigue behavior. We found that surface oxide layer retards crack initiation by blocking the accessibility of water vapor to dislocation slip bands. Film surface morphology evaluation was also studied using AFM, and was found to play an important role in the film fatigue failure.

4:20 PM Invited
Mechanical Properties of Microscale Materials for MEMS Applications: *Yakaichi Higo*[1]; Kazuki Takashima[1]; [1]Tokyo Institute of Technology, P&I Lab., 4259 Nagatsuta, Midori-ku, R2-18, Yokohama 226-8503 Japan

For future application in micro/nano electro mechanical systems (MEMS/NEMS) devices, the elemental size for MEMS devices is thought to be 1/1000 to 1/10000 that of traditional structures. This down sizeing can causes many difficulties, called "Size Effects", which are described as follows, (1) Preparation Process, (2) Handling and Fixing, (3) Accuracy of Dimensions, (4) Accuracy of Measurement Method, (5) Surface Effect, (6) Materials Strengthening Methods and (7) Mechanical Properties. In order to understand these size effects and to overcome the difficulties associated with them, it is essential to develop mechanical testing methods for micro-sized specimens in order to obtain fundamental information for the design of durable and reliable MEMS devices, and to allow comparison of materials of different origin and the processing methods used to produce them. These methods can then be used to provide new strengthening methods to further advance the capabilities of MEMS devices.

4:45 PM
Application of General Weibull Statistics to MEMS: *I. Chasiotis*[1]; A. McCarty[1]; [1]University of Virginia, Mechl. & Aeros. Engrg., PO Box 400746, Charlottesville, VA 22904-4746 USA

The Weibull statistics have been used extensively to describe strength data for uniformly stressed MEMS specimens. In this study, we investigate the relevance of Weibull statistics to more general situations of MEMS-scale specimen failure. The applicability of the Weibull cumulative function to describe failure of (a) specimens with a single flaw distribution and variable geometry and (b) multiple flaw populations and a specific geometry is studied. In the first case, we examine the potential of Weibull statistics to provide a universal description for failure of specimens with non-uniform cross-sections. The uniaxial tension specimens tested and analyzed in this work included variable size perforations at the center of their gauge section, which allowed for twelve combinations of stress intensity factor and radii of curvature. The strength data for each of these geometries were used to calculate the material scale parameter and the Weibull modulus. The approach followed here took into account the non-uniform stress state resulting from the perforations in each specimen through a finite element model and the use of the general integral form for the Weibull function. The computed material scale parameter and the Weibull modulus allow for a general description of brittle failure for MEMS devices with different stress distributions, which can be further used in design and reliability of miniature components with complex geometries. In the second case, we investigated polysilicon MEMS-scale specimens that simultaneously possessed two different flaw populations, both of which were responsible for failure. The microtension specimens characterized in this investigation possessed one flaw population introduced by Reactive Ion Etching (RIE) during surface micromachining and a second population that was the effect of a surface treatment conducted during post-processing. The study also included specimens with the first of the two flaw populations only, which allowed the effects of the second source of failure to be isolated. The data show that the application of Weibull statistics with the assumption of a single flaw population is not appropriate. Instead, the failure behavior of the specimens with two flaw populations is better described by bi-Weibull statistics to incorporate the contribution of both flaw populations to the material strength. Such a statistical treatment provides insight into the origin of material failure and supports more reliable predictions for component failure.

5:00 PM Invited
The Use of Atomic Force Microscopy to Study Crack Tips in Glass: *S. M. Wiederhorn*[1]; J.-P. Guin[1]; [1]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., Gaithersburg, MD 20899-8500 USA

Crack tips in glass are difficult to resolve by most techniques. Transmission electron microscopy is not feasible because glass is amorphous and shows no contrast. Optical techniques are not useful because the surface separation is of the order of nanometers for considerable distances from the actual tip of the crack. In this study we used the Atomic Force Microscope to resolve crack tips in glass specimens subjected to high stresses below the static fatigue limit. Experiments were conducted by first propagating a crack in glass and then reducing the applied stress intensity factor to a value, K_{Ih}, that is less than or equal to the expected fatigue limit, and holding it for a period of time. The load was then increased to the original stress intensity factor and the time measured to restart the crack. After breaking the fracture specimen in two, the "upper" and "lower" fracture surfaces were mapped and compared using atomic force microscopy. Fracture surfaces matched to an accuracy of better than 1 nm normal to the fracture plane and 5 nm within the fracture plane. Displacements between the upper and lower fracture surfaces that developed after a critical holding time were independent of distance from the crack tip, but increased with holding time, approaching an upper limit of about 25 nm for very long times. Despite the surface displacement, cracks tips appeared to be sharp. Results are discussed in terms of a hydronium ion-alkali ion exchange along the crack surfaces and corrosion of the glass surface near the crack tip by hydroxyl ions.

5:25 PM
Fracture Strength of Silicon Carbide Microspecimens Versus Weibull Size Effect Predictions: *N. N. Nemeth*[1]; O. M. Jadaan[2]; M. A. Trapp[3]; W. N. Sharpe[4]; G. D. Quinn[5]; G. M. Beheim[6]; [1]NASA Glenn Research Center, Life Prediction Branch, Cleveland, OH 44135-3191 USA; [2]University of Wisconsin, Math. & Sci., Platteville, WI 53818 USA; [3]Carnegie Mellon University, Dept. of Mechl. Engrg., Pitts-

burgh, PA 15213 USA; [4]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA; [5]National Institute of Standards & Technology, Ceram. Div., Gaithersburg, MD 20899 USA; [6]NASA Glenn Research Center, Sensors & Elect. Tech. Branch, Cleveland, OH 44135 USA

Notched and un-notched micro-sized silicon carbide tensile specimens were fabricated in order to investigate the effect of stress concentration on the strength. Measured strengths in the polycrystalline material were typically below 1 GPa with a Weibull modulus of about 3. A size effect whereby the notched specimens were stronger than the un-notched specimens was observed. Fractographic examination showed the source of failure to be a combination of etching grooves along the specimen side-walls and large sized grains. Strength results were compared to predictions based on the Weibull distribution by using the NASA CARES/Life code. CARES/Life over-predicted the strength of the notched specimens versus the un-notched specimens (the size effect) but correctly predicted that some specimens would fail in the grip section. Correlation of predicted strength compared to experimental results improved when tighter dimensional control was achieved.

5:40 PM
A Numerical Analysis of Flexure Induced Cylindrical Cracks During Indentation of Thin Hard Films on Soft Substrates: Sampath K. Vanimisetti[1]; *R. Narasimhan*[1]; [1]Indian Institute of Science, Dept. of Mechl. Engrg., Bangalore 560012 India

Thin hard films deposited on soft substrates are employed in engineering applications where mechanical loads like those due to contact are experienced (for example, in cutting tools). It is important to understand the mechanics of fracture of these films in order to design better coating-substrate systems. Recent experimental work has shown that during spherical indentation, circumferential cracks may form outside the contact zone on the film surface or at the film-substrate interface directly underneath the indenter. The latter occur because the film experiences bending due to the contact load and behaves as a thin plate resting on a plastic foundation. The objective of this paper is to examine the behavior of these flexure induced cracks. To this end, finite element analyses of spherical indentation of a thin TiN film perfectly bonded to a steel substrate and containing circumferential (cylindrical) cracks as mentioned above are performed. The film is taken to be linear elastic while the substrate obeys an elastic-plastic constitutive model with linear strain hardening. The results show that when the crack length is small, predominantly mode I conditions prevail due to the flexure stresses near the interface. As the crack length increases, the mode mixity gradually changes from mode I to mode II, for cracks located away from the indenter axis. It is observed that the crack growth process is stable up to a crack length of about a third of the film thickness and thereafter becomes unstable. However, due to the presence of a strong compressive radial stress field near the contact zone, the mode I component of loading vanishes for crack length greater than 60% of the film thickness leading to crack closure and arrest. Finally, the role of the substrate yield strength on the above issues is systematically investigated.

Metallurgical Technology for Waste Minimization: Session I

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Toru Okabe, University of Tokyo, Institute of Industrial Science, Tokyo Japan; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA

Tuesday PM Room: 2012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Toru Okabe, University of Tokyo, Tokyo Japan; Ji-Whan Ahn, Korea Institute Geoscience & Mineral Resources Korea

2:30 PM Invited
Electrochemical Copper Removal from Semiconductor Industry Waste Streams: Ran Ding[1]; *James W. Evans*[1]; Fiona M. Doyle[1]; [1]University of California, Matls. Sci. & Engrg., Berkeley, CA 94720 USA

In the semiconductor industry copper is replacing tungsten and aluminum metallization for devices. That poses new challenges in waste-water treatment. Specifically, the manufacturing of current microprocessors generates waste from both the copper electroplating solution and the copper CMP/post-CMP cleaning processes. Traditionally, the concentrated depleted electrolyte is treated separately and there has been little work on the recovery of copper from the dilute waste solutions. Our project is aimed at recycling copper and water from all the waste solutions by a combination of ion-exchange and electrodeposition. Our first-stage work has been to examine the effects of typical electrolyte additives on copper deposition kinetics by the use of a rotating disc electrode. A few results from cells using porous carbon electrode for copper removal are also described. We have also conducted research on using the electrochemical quartz crystal microbalance (EQCM) to determine the adsorption of certain additives on the copper crystal surface. Finally, a fluidized bed electrode has been built and its performance in copper deposition is described. Research supported by US Environmental Protection Agency.

3:00 PM Invited
A Hydrometallurgical Process for Recovery of Vanadium and Molybdenum from Wasted Catalysts for Hydrotreating Desulfurization of Heavy Oil: *Toshiaki Akaboshi*[2]; Yoshiaki Umetsu[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsch. for Advd. Matls., 1-1 Katahira 2-chome, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [2]YSK Techno-system, Kamisu, Ibaraki Japan

The fundamental reactions have been investigated in order to develop a hydrometallurgical process for recovery of molybdenum and vanadium from wasted catalysts generated in the hydrotreating desulfurization of heavy oil. The principal steps of the process are a mild oxidation roasting of the waste catalysts at temperature around 500°C, leaching of the target metal components with dilute ammoniacal aqueous leachates, selective precipitation of vanadic acid, and ammonium molybdate precipitation. The selective recovery ofvanadium as vanadic acid is successfully performed using ammonium sulfate as a salting-out agent. Recovery of ammonium molybdate is based on large dependence of solubility of ammonium molybdate in ammoniacal solutions upon temperature and ammonium salt concentration. In this process, the solution after recovery of vanadium and molybdenum is recycled to the leaching stage of the roasted materials, and the ammonium salts in the process liquor is in the repeated use. Employment of ammoniacal system makes it possible to operate the roasting at much lower temperature than that for the currently operating process.

3:30 PM Break

3:45 PM
Assessment of Costs and Revenues for an Electronic Waste Materials Recovery Facility: *Hai-Yong Kang*[1]; Julie M. Schoenung[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., 3118 Bainer Hall, Davis, CA 95616 USA

At a Materials Recovery Facility (MRF) electronic waste (e-waste) can become marketable output products including resalable systems/components, and recyclable materials such as plastics and metals. The recovery process can be divided into four basic unit operations; sorting/testing, dismantle, size reduction and separation by materials. A spreadsheet model has been developed to estimate the costs and revenues in each unit operation. Input costs include machine costs, labor costs and recycling fees for CRTs, and output streams include revenues from resale systems/component, and recovery of scrap metals and plastics. By estimating the costs and revenues for each unit operation, the model calculates the total processing costs and revenues in operating an MRF that handles e-waste. Sensitivity analysis is used to validate the modeling results. Furthermore, critical cost and revenue drivers are identified, and the viability of utilizing an MRF to handle both current and future types and volumes of e-waste is evaluated.

4:05 PM
Static and Dynamic Synergistic Effects for Ni(II) Extraction and the Application to Waste Solution Containing Ni(II): *Junji Shibata*[1]; Yoshinori Motoda[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Cheml. Engrg., 3-3-35, Yamate, Suita, Osaka 564-8680 Japan

Waste solution from the Ni non-electric plating process contains 4.0kg/m3 Ni (II), 0.1kg/m3 Fe (III), 0.01kg/m3 Zn (II), 48kg/m3 SO42-, 98kg/m3 HPO32- and 31kg/m3 lactic acid as a typical composition. Solvent extraction, cementation, ion exchange resin and precipitation methods may be used for the treatment of this kind of solution. In this study, solvent extraction of Ni (II) is investigated by using two kinds of extractants in order to clarify the relationship between extraction equilibrium (static synergistic effect) and extraction rate (dynamic synergistic effect) for several extractant mixtures. The static synergistic effect does not take place significantly for the extractant mixtures of acidic extractants and neutral organphosphorous compounds, while a large static synergistic effect is confirmed when the extractant mixtures of acidic extractants and nitrogen-containing

compounds are used. Nickel ions can be extracted in the wide pH range of 3.0-8.0 without any pH adjustment by using the several extractant mixtures. At the same time, the extraction rate remarkably increases by using the extractant mixtures of acidic extractants and nitrogen-containing compounds. The mechanism for static and dynamic synergistic effects was investigated. It is possible to extract Ni (II) from artificial waste liquor using a mixture of D2EHPA and LIX860 without pH adjustment. Ni (II) can be stripped with 0.1 mol/dm3 sulfuric acid from the Ni (II)-loading mixtures of D2EHPA and LIX860. The recovery process of Ni (II) from the waste solution in the Ni non-electric plating was proposed from the results of this research.

4:25 PM
Analysis of Extraction Rate of Phosphorous Acid by Tri-n-Octylamine in Toluene: *Yoshinobu Kawano*[1]; Yasuo Hatate[2]; Koichiro Shiomori[1]; Takashi Sana[1]; Hideshi Tanaka[1]; [1]Miyazaki University, Applied Chmst., 1-1 Gakuen-KIbanadai, Miyazaki 889-2192 Japan; [2]Kagoshima University, Chem & Chem. Engrg., Korimoto, Kagoshima 890-0065 Japan

Phosphorous acid presented in the bath of electroless nickel-plating reduce the efficiency of nickel plating. Solvent extraction process is designed to remove the acid in bath. Extraction rate of phosphorous acid by Tri-n-octyl amine in toluene was investigated using a Lewiss type transfer cell having a stagnant interface. The dependencies of concentrations on the rates were analyzed considering the interfacial reaction steps and extraction equilibrium constants for the system. The results for the extraction rate in vibro mixer type extractor having small droplets formed in mixing of fluids were analyzed by the rate equation derived in Lewiss type transfer cell. The mass transfer rates constants was enlarged about 3x103 times of that in Lewiss type transfer cell. We propose the application of the extraction for phosphorous acid by tri-n-octylamine in vibro mixing extractor.

4:45 PM
Solvent Extraction of Precious Metals Using Thiodiglycolamide: *Hirokazu Narita*[1]; Mikiya Tanaka[1]; Kazuko Morisaku[1]; [1]National Institute of Advanced Industrial Science and Technology, Rsch. Inst. for Environml. Mmgt. Tech., 16-1 Onogawa, Tsukuba, Ibaraki 305-8569 Japan

Since precious metals are in extensive use for catalysts, electronic devices and so on, the recovery of precious metals from their wastes has recently received much attention. Especially, the separations of precious metals by solvent extraction are one of the most important studies on their recycling. In this study, we have investigated the extraction properties of some precious metals (Au(III), Pd(II), Pt(IV) and Rh(III)) and base metals (Fe(III), Cu(II) and Zn(II)) from hydrochloric acid solution using the N,N'-dimethyl-N,N'-dioctylthiodiglycolamide (MOTDA). The extraction percentages of Au(III) and Pd(II) using 0.05 M MOTDA are almost 100% over a wide HCl concentration range, while the other metals are hardly extracted. Additionally, the extraction of Pd(II) is extremely fast.

5:05 PM Break

5:20 PM
Iron Removal From Titanium Ore Using Selective Chlorination and Effective Utilization of Chloride Wastes: *Ryosuke Matsuoka*[1]; Toru H. Okabe[2]; [1]University of Tokyo, Dept. of Matls. Engrg., Grad. Sch. of Engrg., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]University of Tokyo, Internatl. Rsch. Ctr. for Sustainable Matls., Inst. of Industl. Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan

This study investigated iron removal from titanium ore using selective chlorination, and effective utilization of chloride wastes generated from titanium production process (Kroll process). The thermodynamic analyses of the chlorination reactions in the Ti-Fe-O-Cl system were carried out prior to the experimental work, and the conditions for chlorination experiments were optimized. The iron in the Ti ore was selectively chlorinated by reacting low-grade Ti ore and metal chloride at 1100 K under a nitrogen atmosphere, and low-iron Ti ore and iron chloride (FeClx) were obtained. Chlorine in the FeClx produced by selective chlorination was recovered as TiCl4 by reacting FeClx and metallic titanium at 1100 K under an argon atmosphere. This recycling process investigated in this study, which utilizes low-grade ore or chloride wastes, is useful because the Ti scrap and chloride wastes are expected to increase. This process has the potential for developing a new environmentally sound chloride metallurgy.

5:40 PM
Process Modelling of Rotary Kiln Treating EAF Dust: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

Electric arc furnace (EAF) dust from the steel industries is listed by EPA as a hazardous waste under the regulations of the Resource Conservation and Recovery Act. The disposal of the approximately 650,000 tons of EAF dust per year in the U.S. and Canada is an expensive and unresolved problem for the majority of steel companies. Waelz process has been considered as the best process treating EAF dust. A process model, combined thermodynamic modelling, chemical kinetics with heat transfer, has been developed to simulate the chemical reactions, mass and heat transfer and heat balance in the kiln. The injection of air into the slag and the temperature profile along the kiln have been modelled. The effect of (CaO+MgO)/SiO2 on the solidus temperature of slag has also been modelled and discussed. Some optimising results have been presented in this paper.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Microstructure and System Stability

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Tuesday PM Room: 3000
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: R. J. Arsenault, University of Maryland, Dept. of Matl. Sci. & Engrg., College Park, MD 20742 USA; Changxu Shi, National Natural Science Foundation of China, Beijing 100085

2:00 PM Invited
Negative Creep of Metallic Glasses as an Externally-Catalysed Dissipative Structure Within Ginzburg-Landau Kinetics: *J. S. Kirkaldy*[1]; [1]McMaster University, Brockhouse Inst. for Matls. Rsch., Hamilton, ON L8S-4M1 Canada

The process to which negative creep is applied concerns annealing temperatures of metallic glasses approaching ambient where normal densification is negligible, but negative dimensional change is dramatically catalysed as a response to a LeChatelier-like load perturbation and initially accelerated against the load by temperature and load increase. This obtains on the isotherm up to the load point where normal creep in the opposite direction must begin to counter the anomalous effects. The proposed dissipative structure, originally regarded by J.C.M. Li as analogous to uphill spinodal decomposition, is based upon strain as a global average order parameter satisfying the time-dependent Ginzburg Landau (TDGL) equation and more generally following the experimentally verified Ostwald Step Rule which subsumes autonomous selection for near-reversibility, minimum dissipation and equivalent high free energy configuration en route to equilibrium. This self-organizing phenomenon can be conceived as a continuous stroke, potential energy-increasing heat engine driven by the quenched-in thermal energy. It is remarkable in that a significant part of the stored free energy is strictly macroscopic, where the usual TDGL reactions such as order-disorder, clustering, Ostward Ripening and grain growth the unused free energy is microscopic or mesoscopic. The Ginzburg-Landau analogy which comes to mind is Type II superconductivity under irreversible flux creep where the conserved free energy corresponding to minimum dissipation is the macroscopic kinetic energy of the Cooper pairs which constitute the supercurrent. In this case the externally applied magnetic field plays the thermodynamic role of the applied stress field. This analogy is suggestive of the possibility that the incredible molecular collaboration involved in negative creep ultimately has a quantum mechanical explanation in much the same way that classical semiconductor theory transforms to a more rigourous quantum mechanical version.

2:25 PM Invited
Thermodynamic Stability of Co/Cu Multilayered Nanostructures: W. W. Cao[1]; Y. Yang[1]; J. Zhu[1]; W. A. Oates[2]; *Y. A. Chang*[1]; [1]University of Wisconsin, Dept. Matl. Sci. & Engrg., Madison, WI 53706 USA; [2]University of Salford, Inst. for Matls. Rsch., Salford M5 4WT UK

When the size of a system decreases to the nanoscale, interfacial energy becomes significant. We can no longer ignore the contribution of the interfacial energy to the total free energy of the system. In deed a recent study suggested that intermixing occurs in multilayered nanostructures of Co/Cu at temperatures lower than the critical temperature for the bulk case. In this presentation, we report the simulation results as a function of the thickness of the metal layers using several thermodynamic models, the regular solution model, the pair approximation of the Cluster Variation Method (CVM) and the cluster/site approximation (CSA). In all these models, the results show that decreases in the thickness of the layer lower the critical temperatures.

2:50 PM Invited
Energy Balances and Stability of Electro-Capillary Systems: *S. J. Burns*[1]; [1]University of Rochester, Mechl. Engrg., Matls. Sci. Prog., Rochester, NY 14627-0133 USA

Wetting between a dielectric liquid and a flat conducting surface is analyzed using energy balances. The wetted surface area, the interfacial surface energy, the voltage, charge, temperature and entropy are considered the state variables in the system. Reversible, equilibrium mechanics are used to obtain the voltage - charge relations on isothermal lines of constant interfacial surface energy. Nonlinear, charged systems are included in these general energy balances. Most frequently, the voltage is linear with charge, i.e., a linear capacitor. For this example, experimental observations shows that the electrostatic energy release rate is proportional to the voltage squared and thus AC or DC voltages create equal driving forces for these systems. The stability of linear systems for a parallel plate capacitor when the capacitance changes linearly with wetted surface area, is considered. This system is neutrally stable at constant voltage, and absolutely stable at constant charge. Measurements establish that the voltage squared is proportional to the interfacial surface energy's driving force using charge control while the charge added to the system is proportional to the total wetted surface area. It is experimentally demonstrated that system stability allows wetting in electro-capillary systems so systematic volumes of liquid can be delivered in micro-liter units. Applications to ink-jet printers and drug delivery systems are explored.

3:15 PM Invited
The Long-Term Stability of Electroplated Thin Films: *Paul T. Vianco*[1]; Garry Bryant[1]; Gary Zender[1]; Paul F. Hlava[1]; [1]Sandia National Laboratories, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA

Electroplated Au films are used with 50In-50Pb (wt.%) solder for level 2 interconnections. Solid-state reactions form the AuIn2 intermetallic compound (IMC) layer at the Au/solder interface. Rate kinetics were measured for thin film Au/In-Pb solder couples having contaminated and non-contaminated Au layers. The AuIn2 layer growth was described by A(t^n) exp(Q/RT). The time exponent, n was equal to one and showed no dependence upon Au layer contamination. The value of Q (50 - 70 kJ/mol) was slightly higher for the non-contaminated Au layer. Formation of the AuIn2 layer was not detrimental to interface integrity. However, contaminants in the Au layer caused voids at the AuIn2/Au interface that degraded adhesion strength. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

3:40 PM Break

3:45 PM Invited
Kinematic and Dynamic Characterization of Plastic Instabilities Occurring in Nano- and Microindentation Tests: *Chinh Quang Nguyen*[1]; András Juhász[1]; Gyõzõ Horváth[1]; György Bérces[1]; János Lendvai[1]; [1]Eotvos University, Dept. of Gen. Physics, Pázmány P. sétány 1/A., Budapest 1117 Hungary

This paper surveys the phenomenon of plastic instabilities occurring in depth sensing indentation measurements, during which a stepwise increase has been observed in the indentation depth vs. load (d-F) curves measured in constant loading rate mode, indicating hardness-oscillations around a nearly constant value of the conventional dynamic microhardness. These oscillations are correlated with Portevin-Le Chatelier type plastic instabilities starting from the contact surface between the sample and the indenter head. Taking into account the experimental observations, a macro-mechanical dynamic model was also proposed for the characterization of indentation instabilities. The characteristics of serrated flow - plastic instabilities - in constant loading rate indentation are compared with those often-observed in conventional, uniaxial compression and tensile tests.

4:10 PM
Stress-Induced Surface Instability of Cylindrical Layer: *Wei Song*[1]; *Fuqian Yang*[1]; [1]University of Kentucky, Chem. & Matls. Engrg., Lexington, KY 40506 USA

The lateral surface stability of a cylindrical layer subjected to a constant uniaxial stress σ_0 and frictionless condition between the layer and cylindrical rigid substrate was analyzed by using the theory of linear elasticity and thermodynamics. The mechanism controlling the surface instability was the gradient of chemical potential associated with surface energy and elastic energy. A new dispersion equation describing the surface evolution was derived. It was found that surface instability depends on the elastic parameter $(2\sigma_0^2 r_1)/(\gamma E)$, Poisson's ratio and film thickness h_0, where r_1 is the radius of the substrate, γ is surface tension, E is Young's modulus and h_0 is the initial thickness of film. For the thin films $h_0/r_1 \to 0$, the critical frequency and the maximum-growth frequency are inversely proportional to the square root of film thickness, while for thick films $h_0/r_1 \to \infty$, they are independent of the thickness.

4:30 PM
Surface Evolution of Crystalline Tubes: Fuqian Yang[1]; *Wei Song*[1]; Jun Zhang[2]; [1]University of Kentucky, Dept. of Cheml. & Matls. Engrg., Lexington, KY 40506 USA; [2]University of Kentucky, Dept. of Computer Sci., Lexington, KY 40506 USA

The surface evolution of an annular tube has been established on the basis of lattice diffusion and linear stability analysis. Without surface disturbance the annular tube will shrink to reduce the surface energy while the cross-sectional area of the tube remains constant. For an annular tube having infinitesimal thickness, the time dependence of the tube radius follows a linear law. When surface energy is significant, a new dispersion relation describing the morphological stability of crystalline tubes due to longitudinal surface perturbation has been formulated. A criterion has been obtained on the dependence of perturbation growth rate on perturbation frequency. The perturbation will grow when the perturbation frequency is less than the critical frequency, which is equal to the inverse of the inner surface radius. To our surprise, the critical frequency for an annular tube of infinitesimal thickness is the same as that given by Nichols and Mullins [Trans. Metall. Soc. AIME 233(1965) 1840] for an infinitely cylindrical rod. A finite spatial frequency for maximum growth rate was also obtained, which depends on the ratio of the inner surface radius to the outer surface radius. The surface instability will lead to the formation of closed end of crystalline tubes.

4:50 PM
Surface Morphology Evolution in Thin Films Via Diffusional Creep: *K. Jimmy Hsia*[1]; [1]University of Illinois, Theoretl. & Applied Mech., 111B Talbot Lab., MC 262, 104 S. Wright St., Urbana, IL 61801 USA

Deformation mechanisms involving mass transport by stress driven diffusion influence a large number of technological problems. We study the formation of undulations on surfaces of stressed films at high temperature by exploring the deformation kinetics governed by volume and surface diffusion. A governing equation is derived that gives the amplitude change of such surfaces as a function of time. A parametric study is then carried out using a range of practically important input values of the film material properties. The results show that at the dominant instability wavelength, under high average stresses (GPa range), only surface diffusion contributes to film surface morphology evolution; whereas under low stress and high temperature conditions, both surface diffusion and volume diffusion contribute to film surface morphology evolution. Furthermore, the contribution of volume diffusion depends on the sign of the film stress, with compressive stress promoting surface roughening while tensile stress promoting surface smoothing.

5:10 PM
Micromechanics Analysis of Elastic Strain and Ferroelectric Domain Texture in Tetragonal PZT Ceramics: *Tsutomu Mori*[1]; David Anthony Hall[1]; Hans Kungl[2]; Philip J. Withers[1]; [1]University of Manchester, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2]University of Karlsruhe, Inst. for Ceram. in Mechl. Engrg., Karlsruhe D-76131 Germany

We demonstrated previously that high energy X-ray diffraction provides a useful technique for determining the elastic strain of a grain 'family' in a poled ferroelectric ceramic as a function of its orientation Ψ relative to the macroscopic polar axis. Specifically, it was

shown that the elastic strain normal to {111} planes and the ferroelectric domain texture, measured by the intensity ratio $I_{(002)}/(I_{(002)}+I_{(200)})$, both exhibit a linear dependence on $\cos^2\Psi$. In the present paper, these observations are analysed on the basis of a simple micromechanical model, which makes use of the unique characteristics of the {111} planes in tetragonal ferroelectrics i.e. that the {111} lattice spacing of a grain is independent of the internal ferroelectric domain population in the 'free' (unconstrained) state. In this approach, individual grains are modelled as spherical inclusions within a surrounding polar matrix, which are characterised in terms of the transformation strain (eigenstrain) associated with the ferroelectric domain fractions. The analysis successfully explains the linear dependence of the elastic strain and the ferroelectric domain texture in a quantitative manner. The results obtained using high energy synchrotron XRD for a range of tetragonal PZT ceramics, having a systematic variation in chemical composition and tetragonality, are evaluated and compared with their macroscopic polarisation-electric field and strain-electric field relationships.

TUESDAY PM

Microstructural Processes in Irradiated Materials: Microstructure Evolution and Segregation

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday PM Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brian Cockeram, Bechtel Bettis Laboratory, W. Mifflin, PA 15122-0079 USA; Yuri Osetsky, Oak Ridge National Laboratory, Computer Sci. & Math., Oak Ridge, TN 37831 USA

2:00 PM
In-Situ TEM Analysis of FePt Ordering Reactions and Grain Growth Via Ion Irradiation: *Nicholas William Morgan*[1]; Gregory B. Thompson[1]; Robert C. Birtcher[2]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., MSD/212, Argonne, IL 60439-4838 USA

We report the use of in-situ TEM analysis of 10 nm and 100 nm thick FePt films irradiated with 500 keV Kr^+ ions. FePt is a potential material for next-generation magnetic recording. When processed, FePt adopts an A1 phase requiring an anneal to order to L10. Irradiation has been proposed to reduce the ordering temperature and maintain small grains. The as-deposited films, grown on SiO, were amorphous and crystallized at room temperature at dosages < $1x10^{14}$ ions/cm^2 with minimal grain growth. The films were then simultaneously annealed and irradiated. Onset of long-range ordering occurred at ~300°C. At elevated temperatures and dosages of ~10^{14} ions/cm^2, the 100 nm film exhibited rapid grain coarsening where as the 10 nm film dewetted and formed an interconnected island pattern. We will address how ion irradiation dosage and temperature can be used to engineer the phase as well as grain morphology of FePt nanostructures.

2:20 PM
Unique Defect Processes at Irradiation-Assisted Stress Corrosion Crack Tips: *Edward P. Simonen*[1]; Larry E. Thomas[1]; Stephen M. Bruemmer[1]; [1]Pacific Northwest National Laboratory, Matls. Div., PO Box 999, Richland, WA 99352 USA

Concurrent point defect processes affect alloy composition in advance of a crack tip during irradiation-assisted stress corrosion cracking (IASCC). Crack-tip corrosion can affect solute redistribution in addition to radiation-induced solute segregation because IASCC occurs at the intersection of the alloy matrix, alloy grain boundary and the aqueous solution. Selective dissolution or oxidation can promote vacancy injection and affect equilibrium concentrations at crack-tip grain boundaries. Competition between radiation-induced segregation, radiation-enhanced diffusion and corrosion-induced vacancy injection has been quantified for conditions relevant to austenitic stainless steels in light-water reactor environments. Corrosion-induced vacancy injection has the greatest potential for changing crack-tip alloy compositions during IASCC propagation. Measured composition changes at crack tips are compared to calculated changes for light-water-reactor component relevant temperatures, doses and dose rates. This work was supported by the Materials Sciences Branch, Office of Basic Energy Sciences and the Office of Nuclear Energy, Science and Technology, U.S. Department of Energy, under Contract DE-AC06-76RLO 1830.

2:40 PM
Phosphorus Interaction with Point Defects in bcc Fe: Electronic Structure Calculation Contribution: Christophe Domain[1]; *Charlotte S. Becquart*[2]; [1]EDF R&D, MMC, Les Renardières, Moret sur Loing F-77250 France; [2]Universite de Lille I, Lab. de Métall. Physique & Génie des Matériaux - UMR CNRS 8517, Bat C6 - 2eme étage, Villeneuve d'Ascq Cedex F-59655 France

The main effect of phosphorus in steels is the possible enhanced embrittlement at grain boundaries. Consequently, the segregation of P under irradiation is an important issue, and furthermore P may influence the evolution of the microstructure under irradiation. The modelling of the P behaviour requires the knowledge of the interaction with point defects created under irradiation, and the possible transport mechanisms. In this work, these interactions have been investigated using ab initio density functional theory. The interactions of P with vacancies and interstitial atoms have been determined and found to be significant. The consequences of these large interactions are discussed.

3:00 PM
Theoretical Investigation of Phosphorus Segregation to Grain Boundaries in Alpha-Iron Under Irradiation: *Alexander V. Barashev*[1]; [1]University of Liverpool, Dept. of Engrg., Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

In pressure vessel steels of nuclear reactors, a big enhancement of the concentration of phosphorus (P) atoms at grain boundaries (GBs) is observed and this leads to a decrease in the GB cohesion and to a shift of the ductile-to-brittle transition temperature. In this paper we present the results of molecular dynamics and Monte Carlo studies of phosphorus atom diffusion in bcc Fe-P alloy in the dilute limit. An EAM type potential set developed by Ackland et al. (2004, J. Phys.: Condens. Matter 16) is used. The diffusion coefficients of point defects and P atoms are evaluated. The rate theory analysis of the P segregation under irradiation conditions is presented. Possible effect of carbon atoms on the P segregation in steels is also discussed.

3:20 PM
Effect of Hafnium Addition on Radiation-Induced Inter-Granular Segregation in Ferritic Steel: Zheng Lu[1]; *Roy G. Faulkner*[1]; N. Sakaguchi[2]; H. Kinoshita[2]; H. Takahashi[2]; Peter E.J. Flewitt[3]; [1]Loughborough University, Inst. of Polymer Tech. & Matls. Engrg., Ashby Rd., Loughborough, Leicestershire LE11 3TU UK; [2]Hokkaido University, Ctr. for Advd. Rsch. of Energy Tech., N. 13, W. 8, Sapporo 060-8628 Japan; [3]Magnox Electric, Berkeley Ctr., Berkeley, Gloucestershire GL13 9PB UK

9%Cr ferritic steels with and without hafnium (Hf) addition were irradiated by 250 keV nickel-ions at 300°C in a ion accelerator attached to the Multi-beam High Voltage Electron Microscopy (ARM1300) to study the influence of Hf addition on radiation-induced segregation in ferritic steel. Grain boundary segregations of phosphorus, silicon, chromium and molybdenum were measured by field emission gun transmission electron microscopy with an energy dispensive analyser. The results show that radiation induces the enrichment of undersized atoms (P, Si) and the depletion of oversized atoms (Cr, Mo) in the materials without Hf addition. The addition of Hf suppressed radiation-induced undersized atom enrichment and oversized atom depletion. A radiation-induced non-equilibrium segregation model is developed to predict radiation-induced oversized atom depletion at grain boundary. The microstructural factors, such as dislocation density, grain size, grain boundary misorientation, and stress effect are taken into consideration in the model. Effect of hafnium on freely migrating defect population is discussed and estimated. The predicted results show a good agreement with experimental data.

Microstructural Processes in Irradiated Materials: Poster Session

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday, 4:00-6:30pm Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: David Hoelzer, Oak Ridge National Laboratory, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; Arthur Voter, Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA

An MEAM Interatomic Potential for the Fe-Cu Alloy System and Cascade Simulation on Pure Fe and Fe-Cu Alloy: *Byeong-Joo Lee*[1]; Junhyun Kwon[2]; Sang Chul Kwon[2]; Jun-Hwa Hong[2]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Engrg., Pohang 790-784 Korea; [2]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. R&D Team, Taejon 305-353 Korea

The microstructural changes occurring during irradiation are essential factors of radiation effects on materials properties, and can be well investigated using atomistic simulation approaches such as molecular dynamics or Monte Carlo simulation. To be able to predict the changes of materials properties more correctly, important is that the interatomic potentials for the atomistic simulation should be more reliable ones and be applicable to practical multicomponent alloy systems. The MEAM interatomic potentials are highly applicable to alloy systems because wide range of elements can be described using a common formalism. In the present study, an interatomic potential for the Fe-Cu binary system was developed using previously developed MEAM potentials of Fe and Cu. The procedure of parameter optimization and transferability of the potential will be presented. Some results of cascade simulations on pure Fe and Fe-0.5at%Cu alloy will also be presented.

Atomic Scale Modelling of the Primary Damage State of Irradiated fcc and bcc Nanocrystalline Metals: *Maria Samaras*[1]; Peter Michael Derlet[1]; Helena Van Swygenhoven[1]; Maximo Victoria[2]; [1]Paul Scherrer Institut, Villigen, Argau 5232 Switzerland; [2]Lawrence Livermore National Laboratory, Livermore, CA USA

Experimental studies of ion irradiated nanocrystalline materials have revealed that that the high density of grain boundaries present in these materials can affect the final damage production. Further differences are seen in defects produced by the different metallic lattice structures, with small vacancy clusters and voids forming in bcc Fe and stacking fault tetrahedra predominately forming in higher energy irradiation of fcc Ni. In the nanocrystalline regime there exists a lengthscale overlap (around the 50nm grain size scale), where experiments and computation are comparable. Large scale molecular dynamics computer simulations of the irradiation of nanocrystalline fcc Ni and bcc Fe with grains sizes in a range of 6- 50nm are presented. The resultant primary damage state after irradiation is discussed in terms of grain boundary structure and grain size, and how these microstructural parameters can affect the primary damage state.

Molecular Dynamics Simulation of Displacement Cascades in α-Fe: A Review: *Lorenzo Malerba*[1]; [1]SCK-CEN, RMO, Boeretang 200, Mol 2400 Belgium

Molecular dynamics has been extensively used to simulate displacement cascades in iron, using different interatomic potentials. The results of this type available from the literature are compared in order to discuss up to what extent they are consistent with each other. It is found that, while generally the number of Frenkel pairs versus recoil energy is the same for most potentials, yielding a defect production efficiency in agreement with experimental estimates, large differences exist concerning the defect clustered fractions. In the case of self-interstitial atoms the criterion used to define clusters is largely responsible for the discrepancies, but sensible differences seem to exist also as a consequence of the different properties of the used interatomic potentials. Very little data have been published concerning vacancy clusters, but in this case too different potentials seem to provide different predictions. These differences may be significant and influence the outcome of, for example, kinetic Monte Carlo studies, depending on what set of input primary damage state is used.

Computer Simulation of Cascade Damage in Alpha-Iron: *Andrew F. Calder*[1]; David J. Bacon[1]; Alexander V. Barashev[1]; Yuri N. Osetsky[2]; [1]University of Liverpool, Dept. of Engrg., Brownlow Hill, Liverpool L69 3GH UK; [2]Oak Ridge National Laboratory, Computer Scis. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA

MD simulation has been applied widely to study displacement cascades in metals. Since it is necessary to use ~1M atoms, the interatomic potentials have simple form and there may be inconsistency between different potentials for the same metal. Iron is of concern because numerous simulations have used potentials for which the <110> dumbbell interstitial is only marginally stable over the <111> crowdion, whereas recent ab initio calculations, (e.g. Fu et al. (PRL 92 (2004)), show that the difference in energy of these defects is ~0.7eV. To investigate this, cascades have been simulated using a new potential (Mendelev et al., Phil. Mag. 83 (2003)) for which the energy difference is close to the ab initio value. A large number of cascades of different energy have been simulated for temperature up to 600K to allow statistical treatment of the number of point defects, the fraction that cluster and cluster properties. Comparisons will be made with previous results.

Similarities and Differences of Point Detect Cluster Formation, Stability and Behaviour in Copper and α-Zirconium: *Roman E. Voskoboinikov*[1]; Yuri N. Osetsky[2]; David J. Bacon[1]; [1]University of Liverpool, Matls. Sci. & Engrg., Dept. of Engrg., Brownlow Hill, Liverpool, Merseyside L69 3GH UK; [2]Oak Ridge National Laboratory, PO Box 2008, MS-6138, Oak Ridge, TN 37831 USA

Atomic-scale simulation of displacement cascades in two metals with close-packed crystalline structures, namely copper (fcc) and α-zirconium (hcp), has been conducted for a wide range of temperature 100 K ≤ T ≤ 900 K and primary knock-on atom (PKA) energy 5keV ≤ E_{pka} ≤ 25 keV. Our study of more than 700 displacement cascades is the largest yet reported for both metals. Using four different identification techniques we identified point defects and their clusters at the end of simulation and carried out comprehensive statistical treatment of the results. For both metals the number of Frenkel pairs and fraction of point defects in clusters versus temperature and PKA energy were obtained. Cluster yield per cascade was also evaluated for both vacancy and self-interstitial atom (SIA) clusters. Typical vacancy and SIA clusters arising in displacement cascades were classified, and the dependence of their mean size on simulation parameters was found. Similarities and distinctions in cluster variety, structure, mobility, thermal stability and transformations in the two metals were investigated in detail.

Copper Precipitates in Alpha-Iron and Their Interaction with Irradiation Produced Defects: *Alexander V. Barashev*[1]; David J. Bacon[1]; Alan C. Arokiam[1]; [1]University of Liverpool, Dept. of Engrg., Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

Precipitation of copper atoms is one of the main reasons for the hardening increase observed in reactor pressure vessel steels during either ageing or irradiation. Theoretical treatment of the experimental data requires knowledge of the diffusion characteristics of copper atoms and interaction properties of copper atoms, vacancies and self-interstitial atom (SIA) clusters with copper precipitates. In this paper, the results of molecular dynamics and Monte Carlo studies of these interactions are presented. The equilibrium concentration of vacancies inside precipitates, the binding energy of a copper atom with a precipitate as a function of precipitate size, the binding energy of a SIA-cluster with a precipitate and transport coefficients are estimated. The calculations were performed using the many-body potential set of Ackland et al. (1997, Phil. Mag. A 75) and ab initio calculations by Becquart and Domain (2003, Nucl. Inst. Meth. B 202).

Mobility of Self-Interstitial Atom Clusters in bcc-Fe: A New Molecular Dynamics Study: *Dmitry A. Terentyev*[1]; Lorenzo Malerba[1]; Marc Hou[2]; [1]SCK-CEN, RMO, Boeretang 200, Mol 2400 Belgium; [2]Université Libre de Bruxelles, Physique des Solides Irradiés & des Nanostructures CP234, Bd du Triomphe, Bruxelles B-1050 Belgium

The mobility of self-interstitial atom (SIA) clusters, characterised by migration energy, prefactor as a function of size and dimensionality of the motion (1D versus 3D) is known to determine largely the microstructural evolution of irradiated metals. The ideal tool to study SIA cluster mobility is molecular dynamics (MD). Yet, so far the description of SIA provided for bcc-Fe by the empirical potentials used in MD studies was at variance with both experimental and ab initio data. Recently, an Embedded Atom Method (EAM) potential capable of

providing a clearly improved description of SIA in á-Fe has been proposed (Mendelev et al., Phil. Mag. 83(35), 2003, 3977 3994). This potential has been used in the present work for dynamical calculations of the diffusion coefficient of SIA clusters in bcc-Fe. The results of the study are expected to help in the definition of the parameter set for kinetic Monte Carlo simulations of radiation damage evolution in ferritic alloys.

Simulations of Elastic Electron Diffuse Scattering from Small Defects: Z. Zhou[1]; S. L. Dudarev[2]; *M. L. Jenkins*[1]; A. P. Sutton[1]; M. A. Kirk[3]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK; [2]EURATOM/UKAEA Fusion Association, Theory & Modlg. Dept., Culham Sci. Ctr., Oxfordshire OX14 3DB UK; [3]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439 USA

We have recently shown that elastic diffuse scattering patterns obtained from single small defect clusters carry information both on the morphology and nature of the defect. Potentially this is an important new method for characterising small clusters. A possible drawback of the our earlier work was the use of kinematical diffraction theory for the simulation of the experimental patterns. A dynamical model of elastic electron diffuse scattering has now been used to calculate the distribution of diffuse scattered electrons in reciprocal space from small dislocation loops and stacking-fault tetrahedra. Simulations were carried out for similar conditions used in experiments to investigate the influence of experimental parameters such as deviation parameters, beam coherence, and the sample conditions, for example, crystal structures, sample thicknesses and the depths of defects. The dynamical and kinematical models will be compared and the validity of the kinematical model assessed. It is shown that the database built from simulated results according to various conditions is essential for the characterization of defects by experimental diffuse scattering patterns, and theoretical analysis and simulations are also very useful for optimizing the experiments.

Effect of the Internal Displacement Cascades Morphology on the Growth of Point Defect Clusters Under Irradiation: *Charlotte S. Becquart*[1]; Christophe Domain[2]; Lorenzo Malerba[3]; Marc Hou[4]; Roger E. Stoller[5]; A. Soudi[6]; [1]Universite des Sciences et Technologies de Lille, LMPGM, UMR 8517, Batiment C6, Villeneuve d'Ascq cedex 59700 France; [2]EDF-R&D Département MMC, Les renardières, Moret sur Loing Cédex F-77818 France; [3]SCK CEN, Reactor Matls. Rsch. Unit, Mol B-2400 Belgium; [4]Physique des Solides Irradiés, CP234, Université Libre de Bruxelles, Bd du Triomphe, Brussels B-1050 Belgium; [5]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS-6138, PO Box 2008, Oak Ridge, TN 37831-6138 USA; [6]Centre Universitaire de Saida, BP138, En-nasr, Saida 20000 Algeria

The influence of the internal cascade structures on the long term evolution of the primary damage is demonstrated by an Object Kinetic Monte Carlo Method and using displacement cascades produced by molecular dynamics as input for long term damage prediction. Displacement cascades obtained in the binary collision approximation as well as random point defect distributions having all similar overall morphologies to the molecular dynamics cascades are used to determine which characteristics of the point defect distributions (spatial extension, the level of agglomeration, etc.) are the most important. Different interatomic potentials are used to generate cascades by MD, allowing the influence of the potential on the results to be discussed.

Kinetic Monte Carlo Simulation of Model Experiments in Iron Based Alloys: *Charlotte S. Becquart*[1]; Christophe Domain[2]; Lorenzo Malerba[3]; [1]Universite de Lille I, Lab. de Métall. Physique & Génie des Matériaux - UMR CNRS 8517, Bat C6 - 2eme étage, Villeneuve d'Ascq Cedex F-59655 France; [2]EDF R&D, MMC, Les Renardières, Moret sur Loing F-77250 France; [3]SCK-CEN Belgian Nuclear Energy Research Centre, Reactor Matls. Rsch. Unit, Boeretang 200, Mol B-2400 Belgium

Object kinetic Monte Carlo methods, based on elementary diffusion and reaction mechanisms, is a very powerful tool to simulate radiation damage and damage accumulation in various configuration (thin slabs, 3D box ?). Model experiments can be used to parameterise the OKMC codes and we have performed ion irradiation simulations of Fe based alloys remaining as close as possible to the model experiments. The purpose is to study the influence of environmental parameters such as temperature, irradiated specimen shape and chemical composition on the microstructure evolution. The model used to simulate the ion irradiation is discussed and the sensitivity of some key parameters is also evaluated.

Exact Kinetic Monte Carlo Simulations Without the Lattice Hops: *Vasily V. Bulatov*[1]; Wei Cai[2]; George Gilmer[1]; Tomas Oppelstrup[1]; Malvin Kalos[1]; Babak Sadigh[1]; [1]Lawrence Livermore National Laboratory, University of California, Livermore, CA 94550 USA; [2]Stanford University, Stanford, CA 94305 USA

This presentation will discuss a new algorithm for kinetic Monte Carlo simulations applicable to a wide range of physical situations where multiple Brownian particles of finite dimensions diffuse, collide and otherwise react with each other. In its spirit, the new approach is reminiscent of the so-called event-based Monte Carlo algorithm (JERK) developed over the years in SACLAY: similar to JERK, it alleviates the need to simulate every single diffusional hop but focuses on more significant changes in the system's configuration. Yet, unlike JERK, the new algorithm is approximation-free and its accuracy is limited only by the quality of the rate coefficients.

Monte Carlo Simulation of Point Defect Recombination During the Initial Stages of Cascade Aging in Fe: *Bor Kae Peter Chang*[1]; Brian D. Wirth[1]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

Structural materials in nuclear energy applications are exposed to intense neutron fields that create atomic displacements leading to microstructural evolution and property changes. At the smallest scales, radiation damage is continually initiated with the formation of energetic primary knock-on atoms through collisions between high-energy neutrons and lattice atoms. Primary damage production in high-energy displacement cascades has been extensively studied by MD simulations and reveals intra-cascade recombination, spatial separation of vacancy and self-interstitial atoms (SIA), and SIA clustering. We have studied the subsequent recombination in the "early" stages of cascade aging as the SIA migrate through and away from the cascade volume. The simulations investigate the effect of PKA energy (from 500 eV to 100 keV), recombination radius (1 to 3 lattice parameters), one dimensional versus three dimensional SIA cluster migration, and temperature (50 - 500°C). The results indicate increasing recombination with cascade energy, which saturates for the highest energy cascades.

Modeling Cascade Aging and Dose Rate Effects in Dilute Fe-Cu Alloys: *Brian D. Wirth*[1]; Jae-Hyeok Shim[1]; G. Robert Odette[2]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]University of California, Santa Barbara, CA USA

Fundamental understanding of defect production in displacement cascades is required to model and predict long-term neutron irradiation induced microstructural evolutions. Defect production is generally treated in terms of primary events, occuring in cascades over time scales of less than 100 ps. We describe the development of advanced kinetic lattice Monte Carlo (KMC) methods to simulate the long-term rearrangement (aging) of displacement cascades as well as cascade aging effects on overall damage accumulation in neutron irradiated dilute Fe-Cu alloys. Special algorithms have been developed to model self-interstitial atom-vacancy recombination in cascades and long range point defect and solute diffusion. The simulations reveal the formation of a continuous distribution of three dimensional cascade vacancy-Cu cluster complexes and demonstrate the critical importance of spatial, as well as short and long-time, correlated processes, that mediate the effect of dose rate on microstructural evolution under conditions relevant to reactor pressure vessel embrittlment.

Kinetic Monte Carlo Simulation of Substitutional He Diffusion in Fe: *Brian D. Wirth*[1]; Jae-Hyeok Shim[1]; Rick J. Kurtz[2]; G. Robert Odette[3]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]Pacific Northwest National Laboratory, Richland, WA 99352 USA; [3]University of California, Santa Barbara, CA USA

Ferritic and martensitic steels will experience severe irradiation induced degradation of many important performance sustaining mechanical properties as well as potential dimensional instabilities in fusion environments, driven by simultaneous production of displacement defects and high concentrations of helium. Thus a key issue is the coupled transport and fate of all defect, gas and solute species, including the effects of radiation enhanced diffusion, segregation and cluster aggregation. As part of a broader multi-scale modeling effort, we present the results of kinetic Monte Carlo (KLMC) simulations of substitutional helium diffusion in iron, based on vacancy jump frequencies in the vicinity of substitutional helium. The jump frequencies are obtained from molecular statics simulations of the potential energy barriers based on semi-empirical Fe-He interatomic potentials. The KLMC results are compared to theoretical descriptions of substitutional solute diffusion in bcc alloys and are also extended to model the diffusion of small helium-multiple vacancy complexes.

Helium and Hydrogen Clustering in Radiation Damaged Iron Studied by Stochastic Simulations: *Chaitanya Suresh Deo*[1]; Michael I. Baskes[1]; Srinivasan G. Srivilliputhur[1]; Stuart Maloy[1]; Michael James[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los

Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, AAA-TDO, MS H809, Los Alamos, NM 87545 USA

Many micro-structural defects are introduced into materials upon irradiation with energetic particles. These defects can cause degradation of mechanical properties and contribute to material failure. Transmutation products such as hydrogen and helium in irradiated stainless steels may exert deleterious effects on material properties. A description of the atomic mechanisms governing the process and their correlation to material properties will result in better understanding of the mechanisms by which iron and iron-based alloys respond to helium and hydrogen implantation by radiation and will suggest methods of alloy improvement to withstand irradiation damage. We have performed kinetic Monte Carlo (KMC) simulations of point defect diffusion and clustering in bcc alpha iron. The model consists of the following entities in bcc iron: interstitial and substitutional helium and hydrogen atoms, interstitial iron atoms, vacancies, vacancy-clusters, and sinks for the trapping of point defects (dislocations and grain boundaries). Input to the simulations includes the migration energies of the point defects (interstitial iron, vacancy, interstitial and substitutional helium and hydrogen), formation energies of the HenVm clusters, dissociation energies of the point defects from the HenVm clusters and initial concentrations and configurations of point defects and defect ratios. These quantities are obtained from experimental data and molecular dynamics (MD) simulations using embedded atom and modified embedded atom potentials. The defect ratios and configurations can be obtained from the post-cascade data of large MD runs. We employ the KMC simulations to investigate the time evolution of the point defect configuration leading to defect clustering and bubble formation. The composition of embryonic defect clusters as a function of time and operating temperatures is determined. It is found that almost all the transmuted helium and hydrogen is trapped in sink configurations (clusters, dislocations or grain boundaries) within a fraction of a microsecond.

The Diffusion of He Atoms and Small He Clusters in Grain Boundaries in Alpha-Fe: *Fei Gao*[1]; Richard J. Kurtz[1]; Howard L. Heinisch[1]; [1]Pacific Northwest National Laboratory, Fundamental Sci., MS K8-93, PO Box 999, Richland, WA 99352 USA

A systematic study of the migration and diffusion mechanisms of He atoms and small He clusters in grain boundaries in α-Fe is presented. Two grain boundaries, the Π11<110> {323}, and the Π<110> {111}, were used for the current investigations. The migration of He atoms and small He clusters were followed from 10 to 30 ns, at temperatures between 400 and 1000 K. The diffusivity and self-diffusion coefficient of He atoms and small He clusters were obtained, and the effective migration energies were determined. Also, the lowest energy paths of He atoms and He clusters were traced out by the Dimer method. He atoms diffuse quickly in the grain boundaries at low temperatures with one-dimensional behavior, but a few directional changes were observed at higher temperatures. The different activation energies and diffusion mechanisms in these two representative grain boundaries suggests that the varying atomic structures of the grain boundaries are important for the diffusivity of He.

Atomistic Modeling of Helium Clustering and Diffusion Along Dislocations in Alpha-Iron: *Howard L. Heinisch*[1]; Fei Gao[1]; Edward A. Le[1]; Richard J. Kurtz[1]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., PO Box 999, P8-15, Richland, WA 99352 USA

An important first step in mitigating helium effects on the mechanical properties of metals is to understand the fate of helium with respect to its interaction with various microstructural features. Molecular statics, molecular dynamics and the dimer method of potential surface mapping are being used to study the fate of helium in the vicinity of a/2<111>{110} edge dislocations in alpha-iron. Interstitial He atoms can easily migrate to dislocations, where they are strongly bound (>2eV) in crowdion positions in the layer of atoms just above the slip plane. A He-divacancy complex can also migrate and be trapped near the core, but with a much lower binding energy (~0.5 eV). These complexes may be a primary mode for the collection of He and its transport along dislocations. Various aspects of the mobility, clustering, and stability of He-divacancy complexes in and near dislocations and their important role in He disposition will be discussed.

Helium Behavior in Metals Characterized by Thermal Helium Desorption Spectroscopy: *Stephen C. Glade*[1]; Brian D. Wirth[1]; Henk Schut[2]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA; [2]Delft University of Technology, Interfaculty Reactor Institute, Mekelweg 15, 2629 JB Delft The Netherlands

The behavior of helium in metals must be understood for their future use in fusion applications. In fusion reactors, the helium concentration in metals increases by direct implantation of helium or by nuclear transmutation reactions. Helium is insoluble in most metals alloys and precipitates in vacancy clusters and voids, forming helium bubbles at high concentrations, causing high-temperature intergranular embittlement. We present the design and initial performance of a thermal helium desorption spectrometer that we are constructing at the University of California, Berkeley. We plan to study microstructural effects on the helium behavior in iron. The information that can be obtained from analyses of the desorption spectra will be discussed.

Positron Annihilation Characterization of Nanostructural Features in High Nickel Copper Free Model Alloys: *Stephen C. Glade*[1]; Brian D. Wirth[1]; G. Robert Odette[2]; Michael K. Miller[3]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]University of California, Santa Barbara, CA USA; [3]Oak Ridge National Laboratory, Oak Ridge, TN USA

Irradiation embrittlement of reactor pressure vessel (RPV) steels results from formation of a high density of nm-scale precipitates. In RPV steels with >0.1%Cu the dominant hardening features are copper-rich precipitates (CRPs) alloyed with manganese, nickel and silicon. But as theoretically predicted long ago, manganese-nickel(-silicon) rich precipitates (MNPs) can form in both copper bearing and copper free alloys, containing large amounts of these elements. Large volume fractions of so-called late blooming MNPs (LBP), cause severe hardening and embrittlement. The presence LBP-MNPs and large hardening in low copper and copper free alloys has been demonstrated recently by a variety of techniques. We present positron annihilation spectroscopy results of neutron irradiated model alloys containing high nickel and low copper. The results provide insight into composition and magnetic properties of the MNPs. The positron results are compared to corresponding small angle neutron scattering, combined electricial resistivity-Seebeck coefficient and atom probe tomography data.

Positron Anihilation in Fe and Fe-Cu After Neutron Irradiation: *Abderrahim Almazouzi*[1]; [1]SCK.CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

In the frame of the european project PERFECT, dealing with the modelling of irradiated materials. Well controlled irrdiations have been performed at 300°C for doses ranging from 0.05 to 0.2dpa. The materials that have been irradiated are ranging from pure Fe to technological steels. In this paper, the Lifetime and coincidence doppler measurements that have been performed on neutron irradiated pure Fe and Fe-Cu containing 0.1 and 0.3% will be reported. the results demonstrate the effect of Cu on reducing the matrix damage and enhancing the Cu-precipitation.

Apt Characterization of High Nickel RPV Steels: *Michael K. Miller*[1]; Mikhail A. Sokolov[1]; Randy K. Nanstad[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The microstructures of high and low copper reactor pressure vessel steels with high nickel contents have been characterized in the local electrode atom probe to investigate the influence of high nickel levels on the response to neutron irradiation. Atom probe tomography revealed nickel-, manganese-, and silicon-enriched precipitates in VVER 1000 base (0.05% Cu,1.26% Ni, 0.46% Mn) and weld (0.07% Cu,1.78% Ni, 0.80% Mn) materials after neutron irradiation. A high number density of copper-, nickel-, manganese-, silicon- and phosphorus-enriched precipitates were observed in the Palisades reactor weld (0.20% Cu, 1.20% Ni, 1.27% Mn) after neutron irradiation. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy.

Precipitation in Neutron Irradiated Fe-Cu-Mn-Ni Model Alloys: *Michael K. Miller*[1]; Kaye F. Russell[1]; Robert Odette[2]; Brian D. Wirth[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA; [2]University of California, Dept. of Cheml. Engrg., Santa Barbara, CA 93106 USA; [3]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

A series of Fe-Cu-Ni-Mn model alloys have been characterized by atom probe tomography and small angle neutron scattering to determine whether any precipitation occurs in low (0.05%) and copper-free alloys during neutron irradiation and to determine the influence of nickel. These alloys contained 0, 0.05 or 0.1% Cu, 0.8 or 1.6% Ni and 1.6% Mn and were neutron irradiated to a fluence of ~1.3 x 10^{23} n m^{-2} (E > 1 MeV) at a temperature of 290°C. After irradiation, precipitates were detected in all alloys. Some 1-2 nm diameter precipitates were

enriched in Ni and Mn and some were enriched in Cu, Ni and Mn. Copper-enriched precipitates were observed in the 0.05% Cu alloys indicating that 0.05% Cu is above the solubility limit under these irradiation conditions. The Cu, Ni and Mn atom distributions were not located at the same center-of-mass in agreement with Monte Carlo simulation predictions. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy. Research at UCSB was sponsored by the U. S. Nuclear Regulatory Commission NRC-04-01-064.

Characterization of Precipitation in MA/ODS Ferritic Steels: *Michael K. Miller*[1]; Kaye F. Russell[1]; David T. Hoelzer[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

Mechanically alloyed, oxide dispersion strengthened (MA/ODS) ferritic steels exhibit excellent high-temperature creep and tensile properties. These ODS ferritic steels are attractive for fusion reactor applications because of their potential for higher operating temperatures and also because the dispersed oxide particles may provide a trap for helium. These ferritic alloys are fabricated by mechanically alloying a pre-alloyed Fe, Cr, Y and Ti powder with a small amount of yttria powder. Atom probe tomography has revealed that these MA/ODS alloys contain a high number density of nanometer scale Ti-, Y- and O-enriched particles in the as-processed condition. These particles were found to be extremely resistant to coarsening at temperatures up to 1300°C (0.85 Tm). Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Irradiated Microstructure of Alloy 800H: *Jian Gan*[1]; James I. Cole[1]; Todd R. Allen[2]; Shuttha Shutthanandan[3]; Suntharampillai Thevuthasan[3]; [1]Argonne National Laboratory, Nucl. Tech., PO Box 2528, Idaho Falls, ID 83403 USA; [2]University of Wisconsin, Dept. of Engrg. Physics, 1500 Engrg. Dr., Madison, WI 53706 USA; [3]Pacific Northwest National Laboratory, Environml. Molecular Sci. Lab., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

Alloy 800H has the same basic composition as INCOLOY alloy 800 (Fe-20Cr-32Ni) but with significantly higher creep-rupture strength. It is one of the high temperature candidate alloys being considered for the Generation IV nuclear reactor system. The radiation resistance of 800H has not previously been studied. This work provides information on the microstructural changes in 800H after irradiation using 5.0 MeV Ni ions at 500°C to 5 and 50 dpa. Following irradiation, changes in microstructure and phase stability were studied using transmission electron microscopy. At the dose of 50 dpa, no voids were found and the density and size of the faulted loops were measured to be 2.3x1016 cm-3 and 8.4 nm, respectively. There are fine precipitates distributed in 800H with an average size approximately 6 nm and a density greater than 9.1x1015 cm-3. The high Ni content and the presence of precipitates are believed to be responsible for the resistance to void formation at dose up to 50 dpa.

Damage Accumulation Under Cascade Damage Conditions with Specific Emphasis on the Evolution of Stacking Fault Tetrahedra: *Stanislav Ilijch Golubov*[1]; Bachu N. Singh[2]; Helmut Trinkaus[3]; Steve J. Zinkle[1]; Roger E. Stoller[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bathel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6138 USA; [2]Risø National Laboratory, Matls. Rsch. Dept., Roskilde DK 4000 Denmark; [3]Forschungszentrum Jülich, Inst. Für Festkörperforschung, Jülich D-2425 Germany

By now it is well established that clusters of vacancies and self-interstitial atoms (SIAs) are produced directly in the displacement cascades. Furthermore, some of these SIAs clusters diffuse one-dimensionally and occasionally may change their direction of diffusion. In addition, they and in addition may diffuse transversely by conservative climb. While diffusing, these clusters are likely to interact with all other defects and their clusters present in the crystal. Over the years, these features of the primary damage production and the ensuing consequences have been incorporated in the production bias model (PBM). During the last ten years or so, various aspects of defect accumulation and microstructural evolution under cascade damage conditions have been treated both analytically and numerically within the framework of the PBM. However the impact of the evolution of stacking fault tetrahedra (SFTs) on damage accumulation in FCC metals, has so far not been subjected to a serious and systematic theoretical treatment. Recently the available experimental data on the evolution of SFTs under irradiation and aging have been reviewed. The review suggests that interactions of SIAs and their clusters with SFTs, transformation of SFTs into Frank loops and direct impingement of cascades/subcascades on existing SFTs may play significant role in determining the evolution of SFTs under different irradiation conditions. These aspects must be taken into account in the theoretical treatment of the microstructural evolution and this is main objective for the present work. The results thus obtained will be implemented to further calculations, for example, of the temperature dependence of microstructural evolution in FCC metals.

The Influence of Fast Neutron Irradiation and Irradiation Temperature on the Tensile Properties of Wrought LCAC and TZM Molybdenum: *Brian V. Cockeram*[1]; Richard W. Smith[1]; Lance L. Snead[2]; [1]Bechtel Bettis Laboratory, PO Box 79, ZAP 05R/MT, W. Mifflin, PA 15122-0079 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA

Molybdenum alloys are generally known to be susceptible to embrittlement following neutron irradiation at temperatures < 800C, which can restrict the broad use of Mo-base alloys in nuclear applications. However, irradiation of molybdenum-base alloys at higher temperatures to relatively low neutron fluence (<60 X 10^20 n/cm^2 (E>0.1 MeV)) can result in little radiation hardening and no embrittlement1. The undesireable changes in the mechanical properties of molybdenum alloys following irradiation can be affected by altering the microstructure and base composition. These variables are evaluated in this work by the irradiation of commercially available wrought Low Carbon Arc Cast (LCAC) and TZM molybdenum in the High Flux Isotope Reactor (HFIR) at 300C, 600C, and 1000C to neutron fluences between 10.5 to 200 X 10^20 n/cm^2. The wrought LCAC sheet has a fine grain size and a high carbon to oxygen ratio, which results in high levels of ductility at room-temperature prior to irradiation. Wrought TZM molybdenum also has a fine grain size with coarse carbides and titanium + zirconium in solid solution that results in higher non-irradiated strength. The change in the Ductile to Brittle Transition Temperature (DBTT), which is based on tensile properties and failure mode, was used as the basis for quantifying radiation embrittlement. Irradiation at 300C is shown to result in evaluation of the DBTT from a pre-irradiated value of -100C to a post-irradiated value of 800C for both LCAC and TZM with a similar increase in fracture stress for both alloys (91% to 25% increase). Irradiation at 600C also resulted in a comparable increase in fracture stress for both alloys (90% to 47% increase), but the post-irradiated DBTT for LCAC (300C) was much lower than observed for TZM (700C). Irradiation of both LCAC and TZM at 935-1100C resulted in little radiation hardening (15% to 0% increase in yield strength) and approximately a -50C DBTT for both LCAC and TZM molybdenum. The finer grain size, low oxygen content, high carbon to oxygen ratio, and absence of coarse carbides may explain the slightly improved embrittlement resistance for LCAC compared to TZM at the 600C irradiation temperature. The post-irradiated fracture modes are shown to correlate with the tensile properties of LCAC and TZM molybdenum. 1. B.V.Cockeram, J.L.Hollenbeck, and L.L.Snead, J. Nuclear Materials, 324 (2004) pp. 77-89.

Strain-Rate Effects on Microstructural Deformation in Irradiated 316 SS: *James I. Cole*[1]; Hanchung Tsai[2]; Todd R. Allen[3]; Tsunemitsu Yoshitake[4]; Naoaki Akasaka[4]; Ichiro Yamagata[4]; Yasuo Nakamura[4]; [1]Argonne National Laboratory, Nucl. Tech. Div., PO Box 2528, Idaho Falls, ID 83403 USA; [2]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [3]University of Wisconsin, Engrg. Physics, Coll. of Engrg., Engrg. Rsch. Bldg., 1500 Engrg. Dr., Madison, WI 53706 USA; [4]Japan Nuclear Cycle Development Institute, Fuels & Matls. Div., Oarai Engrg. Ctr., Oarai-machi, Ibaraki 311-1393 Japan

A series of studies have been performed to investigate the post-irradiation deformation and failure behavior of 12% cold worked 316 stainless steel following irradiation to variety of doses and temperatures. In the current phase of the study, three sets of samples with distinctly different radiation-induced microstructures have been characterized with transmission electron microscopy following tensile testing to failure at a "fast" strain-rate (1 x 10^{-3} s^{-1}) and a "slow" strain-rate (1 x 10^{-7} s^{-1}). The samples were tested at 430°C. The influence of the defect microstructures and strain-rate on deformation and failure behavior is discussed. In particular, mechanisms that might lead to transitions in deformation mode from heterogeneous to localized are analyzed with respect to the irradiation and testing temperatures.

Deformation Mode Maps of Irradiated 316 Stainless Steels in True Stress-Dose Space: *Thak Sang Byun*[1]; Naoyuki Hashimoto[1];

Kenneth Farrell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6151 USA

Deformation modes in type 316 stainless steels after low temperature neutron and ion irradiation have been mapped into the true stress-irradiation dose coordinate system. In the irradiated type 316 stainless steels various deformation microstructure features, such as dislocation tangles and pileups, dislocation channels, stacking faults, twins, and martensite particles, have been observed depending on irradiation and test conditions. With increasing radiation dose the deformation microstructure changed progressively from a dislocation network-dominant to a stacking fault/twin band-dominant or to a dislocation channel-dominant microstructure. Much of this variety in deformation microstructure is believed to result from the low stacking fault energy of the austenitic steels. It was found that the most important external factor for controlling the deformation microstructure was the true stress. In the mapping deformation modes, therefore, the boundaries were drawn by stress-based criteria for deformation modes; the stress criteria for twinning, for channeling, and for macroscopic plastic instability have been proposed and applied in the mapping. Indeed, irradiation hardening and interactions between dislocations and radiation-induced defects plays an important role in determining the deformation mode at high doses; the helium ion-irradiated specimens showed a tendency for twinning due to the non-shearable gas bubbles, while the neutron-irradiated specimens experienced heavy dislocation channeling due to the removable defect clusters. This study also showed the possibility that the stress-based mapping can be expanded to other conditions with minimal experimental efforts.

Molecular Dynamics Simulation of Screw Dislocation Motion in bcc Fe: *Christophe Domain*[1]; Ghiath Monnet[1]; [1]EDF R&D, Dept. MMC, Les Renardieres, Moret sur Loing F-77250 France

At low temperature, the plasticity of steels is controlled by the motion of screw dislocations. Screw dislocation in bcc Fe is studied by classical molecular dynamics using different embedded atom method potentials for Fe. The motion of the screw dislocation under applied strain is presented. It operates through a double kink mechanism. The results are compared to experimental data obtained in extremely pure iron in order to assess the quality of the cohesive models used. Preliminary results regarding the interaction between the screw dislocation and small defects formed under irradiation such as void or interstitial loops is also presented.

Brittle-Ductile Transition in Ferritic Steels Subjected to Irradiation: A Discrete Dislocation Simulation: *Silvester J. Noronha*[1]; Nasr M. Ghoniem[1]; [1]University of California, Mechl. & Aeros. Engrg., 420 Westwood Plaza, #48-121, Los Angeles, CA 90095-1597 USA

Two dimensional discrete dislocation dynamics simulations, incorporating 3D mechanisms are used to study the evolution of plasticity at crack-tip. The result of dislocation reactions: like dislocation annihilation, dislocation junction formation and dynamic source generation are taken into account apart from the long range elastic interaction between dislocations and with the crack. The method is used to determine the brittle-ductile transition in ferritic steels where cleavage propagation from microcracks around hard carbide precipitates or other microcrack initiation sites plays a critical role in the fracture behavior. The dependence of ductile-to-brittle transition temperature (DBTT) with irradiation is studied using the variation of yield stress with irradiation. The micro mechanisms that lead to the sharp increase in the fracture toughness with temperature around DBTT will be discussed. The simulation results are compared with available experimental data.

Dynamics of Edge and Screw Dislocations in Copper in the Environment of Radiation-Induced Clusters: *Yuri N. Osetsky*[1]; David J. Bacon[2]; Bachu N. Singh[3]; [1]Oak Ridge National Laboratory, Computer Scis. & Math., PO Box 2008, MS-6138, Oak Ridge, TN 37831 USA; [2]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK; [3]Risoe National Laboratory, Matls. Dept., PO Box 49, Roskilde DK-4000 Denmark

Commonly, the level of radiation hardening in the post-irradiation tensile tests is related to the density of defect clusters accumulated during irradiation by assuming that these clusters act as obstacles to motion of dislocation produced by applied stress. In a dynamic rector experiment, on the other hand, both defect clusters and dislocations will be generated simultaneously. In an effort to understand the hardening behaviour under these conditions, the resistance to the motion of dislocations due to glissile and sessile defect clusters continuously generated during irradiation is evaluated.

Void Hardening in BCC and FCC Metals Studied by Atomic-Scale Modeling: *Yuri N. Osetsky*[1]; David J. Bacon[2]; [1]Oak Ridge National Laboratory, Computer Scis. & Math., PO Box 2008, Oak Ridge, TN 37831 USA; [2]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

We present results of large-scale atomic modeling of interaction between moving edge and screw dislocations and voids in model crystals of BCC iron and FCC copper. We have considered dislocations with the Burgers vector ½<111> in Fe and ½<110> in Cu, and voids of up to 6nm diameter at T=0. In Fe, where a dislocation is non-dissociated, the critical resolved shear stress (CRSS) is similar to estimates obtained by continuum modeling when dislocation self-stress is taken into account. In Cu, where dissociation occurs, the CRSS for small (<4nm) void is lower than that predicted by the continuum modeling, whereas for larger voids the results are in good agreement. We explain this by the structure of the dissociated dislocation and features of its interaction with small obstacles, when each partial interacts independently, and large obstacles, when the effect of the dissociation becomes negligible. Other atomic-scale features are also demonstrated and discussed.

An Assessment of the Dislocation Obstacle Strength of Precipitates and Defect Cluster: *Takuya Yamamoto*[1]; G. Robert Odette[1]; Brian D. Wirth[2]; [1]University of California, Dept. of Mechl. Engrg., Santa Barbara, CA 93106 USA; [2]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA

Small angle neutron scattering size (r) and volume fractions (f) characterization of copper rich (CRP) and nickel-manganese rich (MNP) precipitates in irradiated RPV steels were combined with yield stress changes to assess the precipitate-dislocation obstacle strength. The database covers a wide range of compositions (Cu, Ni, Mn,..) for RPV steels and model alloys irradiated over a range of flux, fluence and temperature. The yield stress/square root of volume fraction are compared to both the Russell-Brown model (RBM) and recent results of molecular dynamics simulations. Analysis of complex steels requires first de-superimposing the individual precipitate contribution to the overall yield stress. Precipitate strengths are generally consistent with the RBM peaking at 1.2 nm at values from 2800±200 to 4500±400 MPa. The peak hardening increases between pure copper precipitates and MNPs that may be partially ordered. The corresponding strength of matrix defects that harden low copper steels is also assessed.

On the Interactions Between a Vacancy and Interstitial Loops in Metals: M. Angels Puigvi[1]; *Anna Serra*[1]; Nieves de Diego[2]; Yuri N. Osetsky[3]; David J. Bacon[4]; [1]Polytechnic University of Catalonia, Applied Math. III, Jordi Girona 1-3, Barcelona 08034 Spain; [2]Universidad Complutense, Física Materiales, Facultad de Física, Ciudad Universitaria, Madrid Spain; [3]Oak Ridge National Laboratory, Computer Sci. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA; [4]Liverpool University, Matls. Sci. & Engrg., Dept. of Engrg., Liverpool L69 3GH UK

Interactions between point defects and defect clusters and between them and with existing microstructure features cause microstructure evolution and lead to changes in mechanical and physical properties of the irradiated materials. We present results of atomic-scale computer modelling of interactions between a cluster of self-interstitial atoms (SIAs) and a single vacancy in models of bcc, fcc and hcp metals. This type of reaction is considered to be one of the most frequent because formation of SIA clusters, particularly glissile ones, is commonly observed in high energy displacement cascades in all metals. The interaction depends strongly on the dislocation nature of the cluster and is therefore different in the three crystal structures. Vacancy-SIA recombination, in particular, is inhibited by dissociation of the SIA loop on its glide prism.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Diffusion in Oxide Systems

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Tuesday PM Room: 3007
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Afina Lupulescu, Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., Troy, NY 12180-3590 USA; Evan K. Ohriner, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

2:00 PM
Substitutional and Interstitial Diffusion in α_2-$Ti_3Al(O)$: *E. Copland*[1]; D. J. Young[2]; B. Gleeson[3]; [1]Case Western Reserve University, Cleveland, OH USA; [2]University of New South Wales, Sydney, NSW Australia; [3]Iowa State University, Ames, IA USA

The reaction between Al_2O_3 and α_2-Ti_3Al was studied with a series of Al_2O_3 / α_2Ti_3Al multiphase diffusion couples annealed at 900, 1000 and 1100°C. The diffusion-paths were found to strongly depend on α_2-$Ti_3Al(O)$ composition. For alloys with low oxygen concentrations the reaction involved the reduction of Al_2O_3, the formation of a γ-TiAl reaction-layer and diffusion of Al and O into the α_2-Ti_3Al substrate. Measured concentration profiles across the interaction-zone showed "up-hill" diffusion of O in α_2-$Ti_3Al(O)$ indicating a significant thermodynamic interaction between O and either Al or Ti. As O takes interstitial sites in α_2-$Ti_3Al(O)$ the diffusion coefficients for O were determined independently from the interdiffusion of Ti and Al on the substitutional lattice. Diffusion coefficients are reported for α_2-$Ti_3Al(O)$ and γ-TiAl. Finally this is related to the subsequently measured activities of Al, Ti and O in α_2-$Ti_3Al(O)$ in an attempt to better understand the nature of the thermodynamic interaction observed in the measured diffusion-paths.

2:25 PM
Diffusion and Interface Stability During Solid-State Displacement Reactions: *S. N.S. Reddy*[1]; L. B. Wiggins[1]; [1]IBM Corporation, Sys. & Tech. Div., 2070 Rte. 52, B/330, Z/81A, Hopewell Junction, NY 12533 USA

The displacement reaction, A (metal) + BO (oxide) = AO (oxide) + B (metal), at high temperatures is controlled by the diffusion in product phases, namely cation diffusion in AO and oxygen diffusion in B. The stability of the reactive interface is dependent on which of the diffusion step is rate controlling, and was first demonstrated by Rapp, Ezis and Yurek. In this paper, the transition from stable to unstable reactive interface is studied by controlled variation in cation diffusion during the reaction between Copper Oxide and Co-Fe alloys. The transition to interface instability occurs when the product oxide, (Co,Fe)O, can support a cation flux that exceeds the maximum possible Oxygen flux in Copper. The concepts are illustrated using reaction kinetics and product zone microstructure.

2:50 PM
Uphill Diffusion and Zero Flux Planes in Garnets: An Experimental and ATEM Study: *D. Vielzeuf*[1]; A. Lupulescu[2]; A. Baronnet[1]; A. Addad[3]; [1]Université Marseille – CNRS, CRMCN, 5 rue Kessler, 63038 Clermont-Ferrand, France; [2]RPI, Matl. Sci. & Engrg. Dept., Troy, NY 12180 USA; [3]Université de Lille – CNRS, LSPES, France

In order to determine calcium diffusivity in garnets, we carried out self-annealing experiments at 1.3 GPa, 1050-1250°C, from 5 to 36 days, in piston-cylinder apparatus. Polished and calibrated (250-350µm) garnet spheres (Fe51Mg45Ca3Mn1) were incorporated in a powder of clinopyronene and garnet in a graphite and Pt container. At HP and HT, the powder partially melted, recrystallized and developped a calcic overgrowth (Fe28Mg54Ca16Mn2) around the garnet spheres. Long duration experiments were performed to provide measurable relaxation profiles at the interface. Diffusion profiles are narrow (<1µm) and were measured using ATEM techniques. Ca–(Fe,Mg) interdiffusion coefficients in the range 1*10-19 and 1*10-21 m2s-1 were determined at 1250°C and 1050°C, respectively. Some penetration curves are indicative of uphill diffusion. Modelling of these curves indicates the presence of zero flux planes. As far as we know, it is the first time uphill diffusion is evidenced in silicates.

3:15 PM Break

3:30 PM
Internal Displacement Reactions in Multicomponent Oxide Solid Solutions: *S. N.S. Reddy*[1]; D. N. Leonard[2]; L. B. Wiggins[1]; K. T. Jacob[3]; [1]IBM Corporation, Sys. & Tech. Grp., 2070 Rte. 52, B/330, Z/81A, Hopewell Junction, NY 12533 USA; [2]North Carolina State University, Dept. of Matls. Sci., Raleigh, NC 27695 USA; [3]Indian Institute of Science, Dept. of Metall., Bangalore 560012 India

Internal displacement reactions in oxide solid solutions are of the type: A(metal)+(B,C)O(oxide)=B(metal)+(A,C)O(oxide). The oxides AO,BO and CO are iso-structural and form complete solid solutions with A,B and C occupying the same sub-lattice. During reaction, cation B in oxide is displaced by A and B is precipitated as internal metal phase. The cation C does not participate in the chemical exchange reaction. At low concentrations of BO in the starting oxide, the kinetics are controlled by cation diffusion in the product oxide. The diffusion of cations A,B and C in the product oxide are interrelated through cross-coefficient terms in the flux equations. Concentration gradients develop for all cations in product zone. The concepts are illustrated by the following reactions at 1273 K : Fe+(Ni,Mg)O=Ni+(Fe,Mg)O and Fe+(Co,Mg)O=Co+(Fe,Mg)O.

3:55 PM
Oxygen Diffusivity in Nonstoichiometric Cerium Dioxide: *Petrica Cristea*[1]; Marius Stan[1]; [1]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

The diffusion-mediated interactions between intrinsic point defects in nonstoichiometric cerium dioxide is investigated. It is shown that the electric charge carried by the oxygen ions, the presence of reduced Ce(3+) cerium ions, and the orientation of defect pairs influence the frequencies and the local configurations generated by the diffusion jumps. The oxygen self-diffusivity and chemical diffusivity are calculated as functions of temperature and partial oxygen pressure.

4:20 PM
Simulations of High Temperature Internal Oxidation: *Yali Li*[1]; John E. Morral[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., Storrs, CT 06269-3136 USA; [2]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

In order to help design commercial alloys with improved oxidation resistance, mathematical models are being developed to predict internal oxidation as a function of physical properties and boundary conditions. Two methods being used are: Error Function Modeling using Mathematica and Finite Difference Modeling using DICTRA. Mathematica can be applied in the limit of small supersaturations while DICTRA can be applied to more general cases, except when superficial oxide scales form. One limitation is that all simulations assume local equilibrium conditions. Corresponding experimental results on internal oxidation of Cu-Ni alloys will be compared with simulation predictions.

4:45 PM
Anomalous Oxidation of Ferritic Stainless Steels in Air/Hydrogen Fuel Dual Environments: *Z. Gary Yang*[1]; Prabhakar Singh[1]; Jeff Stevenson[1]; Gordon Xia[1]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., 902 Battelle Blvd., Richland, WA 99352 USA

Ferritic stainless steels are among the most promising candidate alloys to construct interconnects in the intermediate temperature (650-800°C) solid oxide fuel cells (SOFCs). During SOFC operation, the interconnects are working in a dual environment, i.e. simultaneously exposed to air at cathode side and a fuel (e.g. hydrogen) at the anode side. Our recent studies found that the oxidation behavior of stainless steels in the dual environments can be significantly different from that in a single exposure, either an oxidizing or reducing atmosphere. The anomalous oxidization is attributed to the hydrogen diffusion flux from the airside to the fuel side under the influence of a hydrogen (as well as water) gradient across the stalinless steel interconnects. This paper will present the details of our study on a number of selected alloys under the dual environments and discuss mechanistic understanding on the anomalous oxidation.

Neutron Diffraction Characterization of Mechanical Behavior: Deformation III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Tuesday PM Room: 3004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sean R. Agnew, University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; Raj Vaidyanathan, University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA

2:00 PM Invited
Measurement and Modeling of Internal Stresses in Directionally Solidified High Volume Fraction Superalloys at High Temperatures: *B. S. Majumdar*[1]; S. Ma[1]; D. W. Brown[2]; [1]New Mexico Tech, Matls. & Metallurgl. Engrg., Socorro, NM 87801 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

Superalloys represent remarkable achievement in the engineering of coherent microstructures, and are being pushed for application at temperatures exceeding 90% of the melting point. Much of the creep life in this regime is dominated by deformation being confined to narrow 50 - 100 nm wide ?× channels, with the 300-500 nm ?×' phase remaining primarily elastic, except at high stresses. The large confinement, and the different elastic/inelastic response of the phases, induce significant internal stresses. These stresses, in turn, play a large role in microstructural stability, and final fracture of the alloy. In order to track the internal stress development, we conducted insitu creep experiments on a columnar grain directionally solidified superalloy using the pulsed neutron source at the SMARTS facility at LANSCE. Tests were conducted at a range of stresses at 900 C, and a rocking technique was successfully used to monitor the elastic strains in the two phases as a function of time. The diffraction measurements showed that the misfit in the loading direction increased fairly rapidly with time, while it decreased in the transverse direction. These results are rationalized in terms of the development of dislocation network at the gamma/gamma-prime interface. TEM analysis was conducted to characterize the interface dislocation structure, and it was observed that these networks formed fairly early in life. However, they reacted into a configuration that appeared to reduce the overall misfit between the gamma and gamma-prime phases at high temperature. The evolution and reaction of these networks will be discussed, and calculations will be presented to show how these networks might influence overall creep rate. Results from finite element method analysis will also be presented, with the overall goal of developing a realistic life prediction methodology based on actual micromechanisms of deformation. We thank Dr. Venkat Seetharaman of Pratt & Whitney for kindly providing us the DS superalloy, and Drs. Bjorn Clausen and Mark Bourke of LANL for their help in accomplishing the neutron diffraction measurements.

2:20 PM Invited
Interaction of Stress on Phase Stability in a Single Crystal Nickel Base Superalloys: S. S. Babu[1]; *Edward Andrew Payzant*[1]; D. W. Brown[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, MS 6064, Oak Ridge, TN 37831-6064 USA; [2]Los Alamos National Laboratory, Lujan Ctr., Los Alamos, NM 87545 USA

Turbine blades used in both land-based gas turbines and in aircraft engines are made up of single-crystal nickel-base superalloys. Their high-temperature strength is related to the presence of coherent, hard L12 ordered γ' precipitates within the γ (fcc crystal structure) matrix. The important microstructural parameters (volume fraction, size, shape, and composition of the γ' phase, as well as lattice mismatch between γ' and γ phases) are affected by the bulk composition, heat-treatment conditions, stress, and time. Prior research suggests that on application of stress at high temperature, the hard γ' phase does not take part in the plastic deformation, which is concentrated in the soft γ phase. A critical requirement is to describe the above phenomenon under in situ thermomechanical conditions and in different crystallographic directions. In this research, the lattice parameters of γ' and γ phases in a single-crystal nickel-base superalloy were investigated by neutron-diffraction while subjected to high-temperature stress during continuous cooling and isothermal conditions using the SMARTS beamline at LANSCE.

2:40 PM Invited
Elevated Temperature Uniaxial and Cyclic Loading of 316 Stainless Steel: *Mark Daymond*[1]; P. John Bouchard[2]; [1]Queen's University, Mechl. & Matls. Engrg., Nicol Hall, Kingston, ON K7L 3N6 Canada; [2]British Energy Generation Ltd., Barnwood, Gloucester GL4 3RS UK

The macroscopic stress-strain response of type 316 stainless steel subjected to cyclic loading at high temperature evolves from the virgin material state to a cyclically hardened state. In practical applications however, the material is not simply cycled, but undergoes cycling with dwell interruptions at various points around the cycle. A knowledge of the start-of-dwell stress alone is insufficient to describe the creep relaxation behaviour observed; the location of the dwell event around the cycle is important. We have carried out experiments investigating the response of the material at a range of elevated temperatures in order to explore the interaction between microscopic internal stresses developed during cyclic loading with the high temperature creep response of the material during dwell periods at different positions around the cycle. The grain orientation strains obtained by diffraction are compared with an elasto-plastic polycrystal deformation model to clarify the role of internal stresses in the material response.

3:00 PM
Creep Relaxation Near a Notch in a Specimen Loaded in Tension: *Nicolas Christodoulou*[1]; Brian W. Leitch[1]; Ronald Rogge[2]; [1]Chalk River Laboratories, AECL, Deformation Tech. Branch, Chalk River, Ontario K0J 1J0 Canada; [2]National Research Council of Canada, Steacie Inst. of Molecular Scis., Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada

A notch is usually defined as a relatively smooth discontinuity or a stress concentration feature that elevates the stress around the region of the notch by a factor of 2 or 3. The stress elevation around a notch is not as severe as that created by the sharp re entrant geometry of a crack. A notch can be a consequence of the manufacturing process or it can be created by an operating event, such as contact with another component. Depending on the far-field stress and the material properties, stress analysis of notches in components subjected to cyclic operating conditions could indicate that the locally elevated stress levels are sufficient to induce cracks that are formed at the notch root and perhaps limit the useful life of the component. However, the incidence of these life-limiting events appears to be relatively low and therefore the high stresses around the notch region must be relaxed in some manner. This paper describes an investigation to ascertain and predict this stress re-distribution with time. Experimental measurements of the stresses around a large radius notch in a tensile specimen were determined using neutron diffraction techniques. A large radius is defined as a radius that is a substantial portion of the specimen cross section. Experimental creep tests on notched, Zr-2.5Nb specimens were carried out at 25 and 300°C. Neutron diffraction measurements were taken during the loading sequence and at regular time intervals during the creep portion of the experiment. The experimental findings are compared with the results of a finite element model where a viscoplastic constitutive law was implemented to simulate the creep relaxation of the notch region stresses. Generalized constitutive creep laws have been developed for un-irradiated and irradiated zirconium materials. Only the un-irradiated material behaviour is considered here and a good agreement is found between the experimental findings and the analytical results.

3:20 PM Break

3:40 PM
Tension/Compression Asymmetry During Fatigue: *Alexandru Dan Stoica*[1]; Xun Li Wang[1]; James W. Richardson[2]; Donald W. Brown[3]; Bjorn Clausen[3]; Hongbo Tian[4]; Michael Lee Benson[4]; Peter K. Liaw[4]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, MS 6474, Oak Ridge, TN 37830 USA; [2]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave., Argonne, IL 60439-4163 USA; [3]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., MS H831, Los Alamos, NM 87545 USA; [4]University of Tennessee, Dept. of Matls. Sci. & Engrg., 319 Dougherty Engrg. Bldg., Knoxville, TN 37996 USA

The residual strain dependence of grain orientation, developing during fatigue life in 316LN stainless steel, was determined by time-of-flight neutron diffraction, as well as longitudinal and transversal lattice

strain dependence of tensile or compressive load on pre-fatigued specimens. The analysis of both types of strain data involves the general concept of strain/stress orientation distribution function, which takes into account of texture symmetry and elastic anisotropy. A phenomenological, model- independent representation of lattice strains was developed for uniaxial loading of cylindrical untextured specimens. The contribution of plastic deformation induced intergranular strains is clearly separated from elastic anisotropy behavior. A marked tension/compression asymmetry was observed to develop during fatigue life. A dramatic difference in residual intergranular strain state measured after the compression and tension half-cycle appears as soon as the fatigue cracks are generated. In late stage, the residual strains vanish for fatigue tests ending in tension and remain high for fatigue tests ending in compression. It is shown that the lattice strain behaviors in incremental in-situ loading experiments match the residual strains in ex-situ samples, if the creep deformation during in-situ loading is taken into account. This research was supported by U.S. Department of Energy, Basic Energy Sciences, Division of Materials Science and Engineering, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. SNS is a partnership of six national laboratories: Argonne, Brookhaven, Jefferson, Lawrence Berkeley, Los Alamos, and Oak Ridge.

4:00 PM

Internal Strain and Texture Development During Cyclic Loading of Haynes® 230®Nickel Based Superalloy: *Tarik A. Saleh*[1]; Hahn Choo[1]; Donald W. Brown[2]; Bjorn Clausen[2]; Peter K. Liaw[1]; Sven K. Vogel[2]; Dwaine L. Klarstrom[3]; Raymond A. Buchanan[1]; [1]University of Tennessee, Dept. of Matls. Sci., 434 Dougherty Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., Los Alamos, NM USA; [3]Haynes International, Inc., Kokomo, IN USA

Haynes 230 is a solid solution strengthened, face centered cubic (FCC), nickel based superalloy. This alloy is frequently used in fatigue intensive applications such as turbine engines. In situ neutron studies can reveal changing internal and residual strains in a material during monotonic or cyclic loading. Additionally, neutron diffraction experiments can reveal the texture of the microstructure, before and after deformation has taken place. Haynes 230 alloy was subjected to monotonic and tension-tension cyclical loading at a σmax of 700 MPa. Internal strains were measured during cycling using in situ neutron diffraction at the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at the Los Alamos Neutron Science Center (LANSCE). Texture was measured before and after cycling using the High Pressure Preferred Orientation (HIPPO) diffractometer at LANSCE. During the fatigue experiment, internal strains were seen to relax over the first 1000 cycles (2.4% of the fatigue life) and then hold steady over the remainder of the fatigue experiment. The initial texture of the fatigue specimens was found to be random. After cycling, the specimen had a texture, 2.5 times random, with the normal to the 111 plane oriented parallel to the loading direction and a two-fold symmetry. The experimental data is being compared to finite element modeling and viscoplastic self-consistent (VPSC) modeling, to develop a mechanistic understanding of the effects of fatigue and deformation on the internal strains and microstructural texture. The present work is supported by the National Science Foundation (NSF), the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program under DMR-0231320, and the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, with Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, C. Huber and Ms. M. Poats as contract monitors.

4:20 PM

Understanding Lüders-Band Evolution During Fatigue by Thermography and Neutron Diffraction Techniques: *Bing Yang*[1]; Yinan Sun[1]; Peter K. Liaw[1]; Choo Hahn[1]; Mark R. Daymond[2]; J. Y. Huang[3]; R. C. Kuo[3]; J. G. Huang[4]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Rutherford Appleton Laboratory, ISIS Facility, Chilton, Didcot OX11 0QX UK; [3]Institute of Nuclear Energy Research, PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, Taipei Taiwan

In tensile testing, yielding, initialized with discontinuous and localized plastic zones or bands, are called the Lüders-band effect, which is commonly observed in steels containing interstitial elements. In engineering applications, Lüders bands often indicate the onset of the plastic deformation at the vicinity of stress concentrations and inclusions, which will contribute to crack initiations - a critical issue in fatigue damage processes. However, up to now, few studies have been performed to understand the Lüders-band evolutions during cyclic loading. In the current study, a detailed investigation on the Lüders band evolution processes during fatigue has been conducted by a combination of the state-of-the-art infrared thermography and neutron scattering techniques. While thermography detection demonstrated in-situ the initiation and propagation of Lüders bands, neutron diffraction technique provides quantitative analyses of the residue stresses in the Lüders band and disclosed the internal mechanism of Lüders band evolution during fatigue, which will be essential to understand the early stages of material fatigue behaviors.

4:40 PM

Measurement and Modeling of the Effect of Hydrogen on the Mechanical Behavior of a Zircaloy-4 Alloy: *E. Garlea*[1]; A. Ionita[1]; H. Choo[1]; D. W. Brown[2]; R. A. Buchanan[1]; P. K. Liaw[1]; C. R. Hubbard[3]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

Hydride formation is one of the main degradation sources of zirconium alloys in hydrogen-rich environments. When sufficient hydrogen is available in these alloys, zirconium-hydride precipitates can be formed. The brittle hydrides near a crack tip can initiate crack growth leading to a premature failure of the material. Hydride formation is believed to be enhanced by the presence of residual or applied stresses. Therefore, the increase in stress field ahead of a crack tip may promote precipitation of additional hydrides. In order to verify this behavior, we investigated the effect of hydrogen charging on the lattice dilation, hydride formation, and mechanical behavior of Zircaloy-4 alloy using neutron diffraction. Spatially-resolved internal strain measurements were made on fatigue pre-cracked compact-tension specimens using in-situ neutron diffraction under applied loads of 667 and 4,444 newtons with or without the presence of hydrogen. The results show that the internal strain profile near the crack tip changes significantly when the specimen is charged with hydrogen. The neutron diffraction results will be compared to the theoretical predictions obtained using finite element modeling.

5:00 PM

Measurement and Modeling of Crack Tip Strains During Tensile Loading and Unloading Cycles: *Y. Sun*[1]; A. Ionita[1]; H. Choo[1]; P. K. Liaw[1]; Y. L. Lu[1]; B. Yang[1]; D. W. Brown[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

The changes in the elastic-lattice strain profiles and plastic zone around a fatigue crack in a compact-tension (CT) specimen were investigated during tensile loading and unloading cycles using neutron diffraction. Spatially-resolved strain measurements were performed to determine the in-plane and through-thickness lattice-strain profiles ahead of the crack tip under a constant tensile load. The strain scanning was repeated under various applied loads ranging from 667 to 6,667 N. Subsequently, overload at 8,889N was applied, and the strain scans repeated. After overload, large compressive strain fields were observed close to the crack tip indicative of the crack-closure phenomena. Diffraction-peak broadening related to the plastic zone was also investigated. The finite element model predictions of the strain (both elastic and plastic) distributions ahead of the crack tip showed good agreements with the experimental data. The results provide fundamental understanding of the micro-mechanics around the crack tip during fatigue deformation.

5:20 PM

Elastic Modulus of B19' Shape-Memory NiTi from Neutron Diffraction, Instrumented Nanoindentation and Extensometry: *S. Rajagopalan*[1]; A. L. Little[1]; M. A.M. Bourke[2]; Raj Vaidyanathan[1]; [1]University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

In situ neutron diffraction during loading and instrumented nanoindentation experiments, using a spherical indenter, were conducted on a B19' shape-memory NiTi alloy to study its elastic behavior. Lattice plane and elastic moduli obtained from the two approaches were comparable but significantly higher than Young's moduli obtained from extensometry in this work and previously widely reported. The lower values from extensometery were attributed to twinning at low stresses that was observed in the diffraction measurements but was suppressed in the spherical nanoindentation experiments. This work was supported by grants from NASA (NAG3-2751) and NSF (CAREER DMR-0239512) to UCF.

Neutron Scattering in Materials Research: Diffraction: Instruments and Nanostructure

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Tuesday PM Room: 3022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109-2136 USA; Brent Fultz, California Institute of Technology, Matls. Sci., Pasadena, CA 91125 USA

2:00 PM Invited
Neutron Scattering Instruments and Opportunities at the Spallation Neutron Source: *R. Kent Crawford*[1]; [1]Oak Ridge National Laboratory, Experimental Facilities Div., Bldg. 8600, Oak Ridge, TN 37830 USA

The unprecedented power and best-in-class neutron scattering instruments of the new Spallation Neutron Source (SNS) facility at Oak Ridge National Laboratory bring many new opportunities for materials science. The SNS has provisions to accommodate up to 24 neutron beam instruments which will cover a broad spectrum of science, and 14 of these have already been funded. Design and construction of these instruments are already well along, and installation of some components is already underway. First instrument commissioning is scheduled for the spring of 2006. This presentation will provide an overview of these instrumentation activities, and will also indicate the performance expected from many of these instruments.

2:30 PM Invited
POWGEN3: A High Resolution Third Generation TOF Powder Diffractometer Under Construction at the SNS: *Jason P. Hodges*[1]; [1]Oak Ridge National Laboratory, Experimental Facilities Div., Spallation Neutron Source, PO Box 2008, Oak Ridge, TN 37831-6474 USA

POWGEN3 is a fundamental departure from previous designs for a time-of-flight powder diffractometer at a spallation neutron source. POWGEN3 may be considered the world's first third-generation time-of-flight powder diffractometer. The combination of a supermirror neutron guide system with a very large modular detector array means POWGEN3 is a very efficient instrument. The high count rates thus achieved together with high-resolution characteristics present a big leap forward in performance over previous diffractometer designs. POWGEN3 will thus provide unprecedented opportunities for new science in the study of polycrystalline materials.

3:00 PM Break

3:20 PM Invited
Nanoscale Structures in Complex Crystals Using Neutron Pair Distribution Function Methods: *Simon J.L. Billinge*[1]; [1]Michigan State University, Physics & Astron., 4268 Biomed. Phys. Sci. Bldg., E. Lansing, MI 48824 USA

We are increasingly interested in complex materials for their unique functional properties. Complex materials often exhibit nanoscale local structures that are important in determining their properties. These come about from defects but often are intrinsic, coming from competing interactions in the materials. It is important to characterize these "nanostructures" but this is difficult because they are not, by their nature, long-range ordered and cannot be studied using traditional crystallographic methods. Imaging probes such as STM and TEM are useful but do not give bulk average information and it is difficult to get quantitative atomic positions in this way. An important complement to these probes are bulk local probes. I will describe the atomic pair distribution function (PDF) analysis method which has proven to be a powerful quantitative probe of nanostructures. I will use examples where neutron diffraction data gives either unique data, or data complementary to x-ray information, resulting in successful understanding of nanostructures in crystals. I will also discuss prospects for the future.

3:50 PM Invited
Medium-Range Atomic Order Studied by Neutron Scattering: *Takeshi Egami*[1]; [1]University of Tennessee/Oak Ridge National Laboratory, MSE/Physics, 208 S. College, 1413 Cir. Dr., Knoxville, TN 37996 USA

Long-range atomic order, or the lattice structure, can readily be studied by diffraction, while the local structure can be accessed by the local probes, such as the EXAFS and NMR. However, there is a dearth of techniques that can probe the medium-range atomic order in the range from 0.5 to 2 nm. Note that TEM sees the structure in this range, but averaged over the sample thickness. We discuss the method of atomic pair-density function (PDF) analysis, which can bridge this gap. In particular, by the upgrading of a pulsed neutron diffractometer, NPDF, of Los Alamos, we now can determine the PDF from zero to up to 20 nm or more. We discuss recent examples, including the nanoscale domains in LiNiO_2. It is also possible to determine the dynamic PDF, by inelastic neutron scattering. An example of local lattice dynamics of relaxor ferroelectric PMN will be discussed.

4:20 PM Invited
Local Atomic Environments in NiPt and SiGe Alloys: *J. Lee Robertson*[1]; [1]Oak Ridge National Laboratory, Ctr. for Neutron Scattering, Bldg. 7962, Oak Ridge, TN 37831-6393 USA

We have investigated two alloys, Ni52-Pt48 and a dilute alloy of 7 atomic percent Ge in Si, using the neutron disk chopper spectrometer (DCS) at NIST. For the Ni-Pt study two crystals were used, a null-matrix crystal using isotopic 62Ni chosen so that the components of the scattered intensity that depend on the average lattice are suppressed leaving only those components that depend on the local atomic configurations that deviate from the average lattice, and a much larger crystal made with natural Ni for studying the dynamics of the alloy. The chemical short-ranged order and static displacement (size effect) scattering are quite prominent for the null-matrix crystal. The Ge-Si crystal showed pronounced fans of diffuse scattering emanating strongly on the low angle side of the Bragg peaks indicating that the Si lattice is expanded around the Ge "defects" and the static displacement field extends to distant neighbors.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Phase Equilibria, Interfacial Energy and Wetting Phenomena in Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Tuesday PM Room: 3016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Herbert Ipser, University of Vienna, Dept. of Inorganic Chmst., Wien A-1090 Austria; Douglas J. Swenson, Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI 49931 USA

2:00 PM
Thermochemistry of the Quaternary System Ag-Cu-Ni-Sn: *Hans Christian Flandorfer*[1]; Christoph Luef[1]; Herbert Ipser[1]; [1]University Vienna, Inorganic Chmst., Waehringer Str. 42, Vienna 1090 Austria

The quaternary intermetallic system Ag-Cu-Ni-Sn and its binary and ternary constituents are of importance because Ag-Cu-Sn is a key systems for lead-free soldering and Ni is a frequently used contact material. The aim is to establish reliable thermochemical and phase relation data from literature and our own experiments. First we determined partial and finally integral enthalpies of mixing of liquid alloys by calorimetric measurements performed at different temperatures.

The experimental results were fitted using the Redlich-Kister-Muggiano formula in order to provide a numerical description of the integral enthalpies of mixing according to the CALPHAD method. The experimental results were also compared with extrapolations and theoretical values from pseudo-potential calculations. Our work is considered as a contribution to the COST-Action 531 "Lead-free solder materials". The main objective of the action is to increase the basic knowledge on possible alloy systems that can be used as lead-free solder materials and to provide a scientific database on possible lead-free solder materials and soldering processes. Experimental thermochemical data are important for the optimization of phase diagram calculations as well as the evaluation of physicochemical properties like e.g., diffusion and surface tension.

2:30 PM

Bi-From Woods Metal to Lead Free Solders: *Sabine Knott*[1]; Adolf Mikula[1]; [1]University of Vienna, Inst. of Inorganic Chmst., Währingerstraße 42, Vienna 1090 Austria

Bismuth, which was discovered as a metal in 1753, offers a wide range of applications. It is a component of Woods metal, named after the American metallurgist B. Wood, which is widely used in electrical fuses and in automatic fire alarm and sprinkler systems. Bismuth is environmentally friendly and non toxic and it is already used as a replacement of lead in solder alloys. The investigated thermodynamic data of the ternary Ag-Bi-Sn and the ternary Cu-Bi-Sn systems will be presented. The Ag-Sn-Bi solder is especially used for the Wave soldering and Reflow soldering processes. No data of the ternary Cu-Bi-Sn system are available within the literature although the knowledge of the properties of this system might be necessary not only for a possible application as a lead free solder but also as a base for the investigation of the quarternary Ag-Bi-Cu-Sn system.

2:50 PM

Interaction of Ag-In-Sn Solders with Palladium Substrates: A Phase Diagram Approach: Adela Zemanova[2]; Ales Kroupa[3]; Jan Vrestal[2]; *Herbert Ipser*[1]; Christoph Luef[1]; Hans Flandorfer[1]; [1]University of Vienna, Dept. of Inorganic Chmst., Waehringerstrasse 42, Wien A-1090 Austria; [2]Masaryk University, Dept. of Physl. & Theoretl. Chmst., Kotlarska 2, Brno CZ-61137 Czech Republic; [3]Institute of Physics of Materials AS CR, Zizkova 22, Brno CZ-616 62 Czech Republic

In order to understand the interaction of lead-free Ag-In-Sn solders with palladium substrates one has to know the quaternary Ag-In-Pd-Sn phase diagram, and for that all the constituent binaries and ternaries. For this purpose, enthalpies of mixing were determined calorimetrically for liquid ternary Ag-Pd-Sn and In-Pd-Sn and quaternary Ag-In-Pd-Sn alloys. These experimental results were taken into account as an input for the optimization in CALPHAD-type phase diagram calculations of the above-mentioned two ternary phase diagrams. These were then combined with an earlier optimization of the Ag-In-Pd system as well as with literature data on the Ag-In-Sn system to obtain a first idea of the phase diagram of the quaternary Ag-In-Pd-Sn alloy system. The calculations were supported by a number of specifically devised experiments.

3:10 PM

Phase Equilibria in the Sn-Rich Corner of the Sn-Cu-Ni Ternary Alloy System: Chia-Ying Li[1]; *Jenq-Gong Duh*[1]; Sue-Yueh Tsai[2]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., No. 101, Sec. 2, Kuan Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Instrument Ctr., No. 101, Sec. 2, Kuan Fu Rd., Hsinchu 300 Taiwan

The interfacial reactions between solders and under bump metallization (UBM) are of highly interests recently in flip chip technology. Intermetallic compounds (IMCs), i.e. $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, formed between solders and UBM. To fully understand the interfacial reactions and phase transformation phenomenon, a suitable phase diagram concerning solder, IMCs and UBM material is required. As a result, Sn-riched phase in Sn-Cu-Ni ternary phase diagram is very critical in determining the concentration tendency of x and y values in $(Ni_{1-x},Cu_x)_3Sn_4$ and $(Cu_{1-y},Ni_y)_6Sn_5$ compounds. In this study, ternary Sn-Cu-Ni alloys were prepared and annealed at 240°C. Three equilibrium phases, Sn, Ni_3Sn_4 and Cu_6Sn_5, were identified by XRD analysis, and also evidenced in BEI micrograph. Using EPMA quantitave analysis, three acme compositions of the ternary region in the Sn-Cu-Ni isotherm near the Sn-riched corner were determined as 98.5 at.%Sn, $(Ni_{0.80},Cu_{0.20})_3Sn_4$ and $(Cu_{0.59},Ni_{0.41})_6Sn_5$. Furthermore, the degree of composit ion homogeneity and the distribution of the forming phases were evaluated by X-ray color mapping and phase analysis technique. By way of software program to convert the concentration measured with EPMA, the exact compositions could be mapped on the Sn-Cu-Ni ternary isotherm. In addition, the solubility of the Cu and Ni in $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_6Sn_5$ compounds was evaluated. Finally, the isothermal section of the ternary Sn-Cu-Ni system at 240°C was proposed on the basis of experimental results in this study. Furthermore, the related phase transformation was also discussed with respect to the formation of the IMCs around 240°C.

3:30 PM Break

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Driving Force of Reactive Wetting in the Au-Sn System: *Liang Yin*[1]; Timothy J. Singler[1]; [1]SUNY Binghamton, Dept. of Mechl. Engrg., 4400 Vestal Pkwy. E., Binghamton, NY 13902-6000 USA

Isothermal sessile drop experiments were performed in a gaseous reducing atmosphere to identify the driving force for the reactive wetting in the Au-Sn system. Two alloys, pure Sn and a eutectic Au-Sn alloy (80 wt.% Au), were used to wet pure Au and an intermetallic compound ζ substrates at 430°C. Spreading on pure Au substrates was accompanied by ζ phase formation and exhibited a near perfect wetting behavior. Spreading on ζ substrates was dissolutive and driven by uncompensated Young's force coupled with solute concentration gradients in the liquid.

4:00 PM

Experimental Wettability Studies Combined With the Related Properties from Data Bases for Tin Based Alloys With Silver, Copper, Bismuth and Antimony Additions: *Moser Zbigniew*[1]; Gasior Wladyslaw[1]; K. Ishida[2]; I. Ohnuma[2]; X. J. Liu[2]; K. Bukat[3]; J. Pstrus[1]; J. Sitek[3]; R. Kisiel[4]; [1]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., 30-059 Kraków, Reymonta Str. 25 Poland; [2]Tohoku University, Grad. Sch. of Engrg., Dept. of Matls. Sci., Obayama 02, Sendai 980-8579 Japan; [3]Tele and Radio Research Institute, 03-450 Warszawa, Ratuszowa Str. 11 Poland; [4]Warsaw University of Technology, Inst. of Microelect. & Optoelect., 00-662 Warszawa, Koszykowa Str. 75 Poland

Starting from binary eutectic Sn-Ag and close to ternary eutectic Sn-Ag-Cu alloys we have investigated the additions of Sb to quaternary tin based Sn-Ag-Cu-Bi alloys. Main aim is to obtain alloys with wettability and T_m closer to 183°C characteristic for traditional Sn-Pb eutectic solders. In this study we use database for Pb-free solders ADAMIS developed in Tohoku University in Japan, to calculate the phase diagram of the Sn-Ag-Cu-Bi-Sb quinary system by means of Calphad method and the melting phenomena, to simulate solidification using Scheil's model and to calculate surface tensions from thermodynamic parameters of the liquid phase by the Butler's model. The resulting calculated surface tensions will be verified with experimental data from maximum bubble pressure method (performed within 250°C to temperatures exceeding 900°C) and with interfacial tensions from meniscographic studies made at 250°C, finally to draw the conclusions important from the point of view of practical application analyzing wettability, mechanical and electrical properties.

4:20 PM

Wettability of Electroplating Ni-P in Under Bump Metallurgy with Sn-Ag-Cu Solder: *Yung-Chi Lin*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

The nickel plating has been used as the under bump metallurgy (UBM) in the microelectronic industry. In this study, the electroplating process was demonstrated to be a good alternative approach to produce the Ni-P layer as UBM. The wettability of several lead-free solders, such as Sn-3.5Ag, and Sn-3.5Ag-xCu (x=0.2, 0.5, and 1.0) solder, on electroplating Ni-P with various phosphorous contents (7wt%, 10wt% and 13wt%) was investigated. The role of phosphorus in the wettability was probed. The surface morphology and surface roughness in electroplating Ni-P was observed with the aid of both field emission scanning electronic microscope and atomic force microscope. The correlation between wettability and phosphorus contents in electroplating Ni-P was evaluated. As the phosphorous contents increased, the nodule size of the Ni-P deposit reduced and surface roughness of Ni-P became smaller. The improvement of surface morphology and surface roughness enhanced the wettability of electroplating Ni-P. The interfacial reaction between lead free solder and electroplating Ni-P UBM was also investigated.

4:40 PM

Interfacial Reactions in the Sn/(Ni,V) Couples and Phase Equilibria of the Sn-Rich Sn-Ni-V System: *Sinn-wen Chen*[1]; Chih-chi Chen[1]; Ching-ya Kao[1]; [1]National Tsing-Hua University, Chem. Engrg. Dept., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

In the Cu/Ni(V)/Al UBM, 7wt% vanadium is added to overcome the magnetic interference of nickel and to enhance the Nickel sputtering.

The interfacial reactions between various solders with the nickel substrate have been investigated extensively; however, the effects of vanadium addition upon the solder/(Ni,V) interfacial reactions have not been investigated. This study examined the interfacial reactions of pure Sn with the pure vanadium and (Ni,V) substrates of various vanadium contents. It is found that besides the Ni3Sn4 phase, a new ternary phase is formed. The phase equilibria of the Sn-rich Sn-Ni-V are studied. The reaction paths are determined based on the determined isothermal section and the reaction couple analysis.

Phase Transformations Within Small-Size Systems: Magnetic and Structural Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee

Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Tuesday PM Room: 3002
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Ramamoorthy Ramesh, University of California, Matls. Sci. & Engrg., Berkeley, CA 94720 USA; Marc DeGraef, Carnegie Mellon University, Matls. Sci. & Engrg., Pittsburgh, PA 15229-3180 USA

2:00 PM Invited
The Phase Diagram of Magnetic Nano-Rings: *Marc De Graef*[1]; Marco Beleggia[2]; June Lau[2]; Marvin Schofield[2]; Shakul Tandon[1]; Yimei Zhu[2]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15229-3180 USA; [2]Brookhaven National Laboratory, Ctr. for Functional Nano-Matls., Building 480, PO Box 5000, Upton, NY 11973 USA

Magnetic nano-rings, circular structures with rectangular cross-section, form the basic building blocks of magnetic random access memory (MRAM). There are three basic magnetization states for such rings when they are sufficiently small: uniform axial, uniform in-plane, and vortex. Most studies of magnetic nano-rings use a micromagnetic computational approach to determine the magnetization state for a given set of shape and materials parameters. In this contribution we present an analytical approach to the complete magnetic phase diagram of such rings. We derive the magnetostatic energy, including demagnetization factors, the magnetocrystalline anisotropy and exchange energy terms, and derive from them a four-dimensional phase diagram. The magnetization state is determined by two shape parameters, (ratio of inner to outer radius and aspect ratio), and two material parameters (ratio of anisotropy to magnetostatic energy density and magnetic exchange length). We will present the detailed phase diagram, as well as confirmation of the diagram by means of micromagnetic simulations and electron holography observations.

2:35 PM Invited
Microstructural Evolution and Interfaces in Ferromagnetic Semiconductor Films Based on p-Type Ge-X (X=Mn,Fe,Cr) and n-Type CdCr2Se4: *Ramasis Goswami*[1]; G. Kioseoglou[2]; A. T. Hanbicki[2]; B. T. Jonker[2]; G. Spanos[2]; [1]Geo-Centers Inc., Naval Rsch. Lab., 4555 Overlook Ave., Metall., Code 6320, Washington, DC 20375 USA; [2]Naval Research Laboratory, Code 6300, 4555 Overlook Ave., Washington, DC 20375 USA

Considerable efforts have been made at the Naval Research Laboratory to understand the basic mechanism(s) of ferromagnetic order in Ge-X semiconductors (X = Mn, Fe, Cr), and in hetero-epitaxial thin films based on CdCr2Se4/ZnSe/AlGaAs. It is observed that the Ge transition metal films are not single crystalline and consist of transition metal rich particles in a Ge matrix; small amounts of transition metal are often still retained in solution in the Ge matrix between the particles. Likewise, relatively complex microstructural evolution has been observed in the hetero-epitaxial thin film systems. It is therefore essential to understand the phase evolution and characterize the microstructure and interfaces in these films in order to put forward successful theoretical models which explain the underlying mechanism(s) of ferromagnetic order. Phase transformations, defect evolution, and interfaces have thus been studied in detail in Ge-X (X= Mn, Fe, Cr), CdCr2Se4/ZnSe/AlGaAs/GaAs(LED), and CdCr2Se4/AlGaAs/GaAs(LED) films prepared by molecular beam epitaxy at different substrate temperatures. The microstructural evolution in all three Ge-based systems will be discussed first, and correlated with the magnetic properties. The effect of substrate temperature on the microstructure and interfaces of CdCr2Se4/ZnSe and CdCr2Se4/AlGaAs will be shown next, and related to the observed spin polarized electron injection into the AlGaAs/GaAs LED structures.

3:10 PM
Cu-Fe Interdiffusion During Severe Plastic Deformation of a Nanoscaled Composite: *X. Sauvage*[1]; P. Pareige[1]; [1]Université de Rouen, Groupe de Physique des Matériaux - UMR CNRS 6634, 76801 Saint-Etienne-du-Rouvray France

As reported in the literature, the ball milling of Fe-Cu powder mixtures usually leads to the formation of supersaturated solid solutions. The aim of this work was to induce such a mechanical mixing in a bulk Cu-Fe nanocomposite by continuous severe plastic deformation. The nanostructure of a Cu-Fe(10%vol.) composite processed by High Pressure Torsion (HPT) was investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), and 3D atom probe analysis (3D-AP). This latter technique gives the evidence of Fe-Cu that the smaller the Fe nanoclusters the stronger the interdiffusion. Thus, the driving force for the mechanical mixing is thought to be the result of a high capillary energy. The mechanism of Fe-Cu enhanced diffusion during the HPT process is then discussed. The formation of excess vacancies would dramatically increase the atomic mobility.

3:35 PM Break

3:50 PM Invited
Self-Assembled Single Crystal Ferromagnetic Iron Nanowires formed by Decomposition: *Ramamoorthy Ramesh*[1]; Ladan Mohaddes-Ardabili[1]; [1]University of California, Hearst Memorial Mining Bldg., Berkeley, CA 94720 USA

We report a novel and simple approach to create self-assembled ferromagnetic α-Fe nanowires, which involves spontaneous phase decomposition of a single-phase perovskite oxide during film growth. The growth of $La_{0.5}Sr_{0.5}FeO_{3-x}$ films by pulsed laser deposition under reducing oxygen conditions leads to spontaneous formation of an array of single crystalline α-Fe nanowires embedded in an antiferromagnetic $LaSrFeO_4$ matrix, which grow perpendicular to the substrate and span the entire film thickness. The diameter, shape, density and spacing of these α-Fe nanowires depend systematically on growth conditions. At high growth temperature, square shaped α-Fe pillars with a lateral width of 40-50 nm are formed. As the deposition temperature is reduced the shape evolves progressively into octahedral and then circular section. The diameter of the nanowires reduces to 4-6 nm for the growth at 560ºC. The large remanence and sizable coercivity of the nanowires make them desirable for high-density data storage and other magnetic device applications.

4:25 PM Invited
Self-Assembled Growth of DySi2 Nanowires and Nanostructures on Si(100): Gangfeng Ye[1]; Jun Nogami[1]; *Martin A. Crimp*[1]; [1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48823-1226 USA

Dysprosium silicide nanostructures have been grown by metal deposition on to Si(100) substrates at 600°C with coverages ranging from 0.3 to 3.0 monolayers. The resulting DySi2 nanostructures have been characterized using scanning tunneling microscopy (STM) and high resolution transmission electron microscopy (HRTEM). STM has revealed two general types of structures. Large aspect ratio nanowires, with heights up to 0.7 nm and widths up to 6 nm were found to grow in the <110> Si directions. Larger rectangular 3-D islands (nanostructures) with heights ranging from 1.2 to 7.5 nm (depending on growth condition) and widths up to 50 nm were found interspersed amongst the nanowires. HRTEM and associated fast Fourier transform (FFT) analysis indicates that in addition to the expected hexagonal form (AlB2 type) of the DySi2, orthorhombic (GdSi2 type) and/or tetragonal (ThSi2 type) co-exist on the Si(100) substrates. Cross-sectional HRTEM reveals a number of different interfacial structures, with varying degrees of strain. The observed structures will be discussed in terms of the mismatch between the different observed phases and the Si(100) substrate. This work has been supported by the National Science Foundation grant number DMR-0305472.

5:00 PM
Phase Stability in $Ce_{1-x}Zr_xO_{2-y}$ Nanocrystals: Feng Zhang[1]; Chih-Hao Chen[1]; Jonathan C. Hanson[2]; Caliebe Wolfgang[2]; Richard D. Robinson[1]; Irving P. Herman[1]; *Siu-Wai Chan*[1]; [1]Columbia University, Matls. Sci. & Engrg. Dept. of Applied Physics & Applied Math., 1136 Mudd Bldg., MC 4701, 500 W. 120th St., New York, NY 10027 USA; [2]Brookhaven National Laborataory, Upton, NY USA

Cubic $Ce_{1-x}Zr_xO_{2-y}$ is the oxygen storage constituent in three-way catalyst and will loose its functionalities when it transforms to either the tetragonal or the monoclinic phase. It is therefore important to know the stability range of the cubic phase (c') of $Ce_{1-x}Zr_xO_{2-y}$. We employed x-ray diffraction (XRD), time-resolved high temperature XRD, transmission electron microscopy, Raman spectroscopy, and X-ray absorption near edge spectroscopy (XANES) for the investigation. With decreasing particle size, the c'-tetragonal phase boundary shifts to a higher zirconium concentration. A clear relationship between the phase stability of c' phase with Ce3+ concentration has been found from Ce LIII edge in XANES. We will report different phase stability ranges for nanoparticles in different redox environments as well as the differences from those predicted by traditional bulk phase diagrams.

Refractory Metals in Electronic Applications: Joint Session with Texture and Microstructure in Thin Films and Coatings: Texture and Thin Films

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee, EMPMD-Thin Films & Interfaces Committee, SMD-Refractory Metals Committee, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Gary A. Rozak, Fabricated Products, Cleveland, OH 44117 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA

Tuesday PM Room: 3010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Christopher Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA

2:00 PM
Development of Texture in Asymmetric Processing of Tantalum Plate: *David P. Field*[1]; Jeff Yanke[1]; Eva V. McGowan[1]; Christopher A. Michaluk[2]; [1]Washington State University, Mechl. & Matls. Engrg., Box 642920, Pullman, WA 99164-2920 USA; [2]Williams Advanced Materials, 2306 Cassard Cir., Gilbertsville, PA 19525 USA

Sputtered tantalum and TaN are employed as barrier layers in modern integrated circuits to enable reliable use of Cu as an interconnect material. The directional properties of sputtering Ta can result in non-uniform film thicknesses (from heavily textured plate) and unpredictable sputtering rates (from plates with through thickness texture gradients). This results in film thicknesses larger than necessary because of the sputtering being unpredictable. This presentation reports on an effort to increase textural and grain size uniformity in Ta by deforming the plate under conditions that simulate asymmetric rolling. This is accomplished by using a channel die configuration with uneven friction on the top and bottom platens so a strong shear component is added to the plane strain condition enforced by the channel dies. Results indicate a trend towards less severe texture banding and more uniform structure in the plate processed by asymmetric friction conditions.

2:25 PM
Control of Texture in Plates for Sputtering: *Peter R. Jepson*[1]; [1]H.C. Starck Inc, Fabricated Products, 45 Industrial Pl., Newton, MA 02461 USA

For some applications, achievement of excellent uniformity of thickness of a sputtered film is important: for example, tantalum when used as a barrier layer under copper interconnects in integrated circuits. The crystallographic texture of the target plate affects sputter yield and must therefore be carefully controlled, if excellent uniformity is to be achieved. H.C. Starck Inc. has worked since 2000 to establish itself as the technology leader in the supply of plate for this application, and in so doing has studied the effects of target texture on the thickness of the thin film, and also developed manufacturing processes which consistently produce top-quality product at a competitive price. Innovative R&D techniques (Finite Element Modelling, and Electron Back-Scattering Diffraction) will be described, as well as the important features of the products which have been launched since 2002, including crystallographic texture control.

2:50 PM
Tantalum Angular Sputter Emission Distributions: *Charles E. Wickersham*[1]; Zhiguo Zhang[1]; [1]Cabot Corporation, Thin Films, 1275 Kinnear Rd., Columbus, OH 43212 USA

Sputtering target crystallographic orientation affects the film deposition uniformity by controlling the emission trajectories of the sputtered atoms. This effect is well known having been first reported by Wehner et al. in 1956. More recently, this effect has been observed to affect film uniformity for magnetron sputtered aluminum alloy films. Understanding the relationship between the crystallographic orientation of the sputtering target and the emission trajectory for the sputtered atoms is important for being able to accurately model film thickness uniformity and step coverage. In this paper we report measurement of the relationship between the crystallographic orientation of tantalum single crystal sputtering targets and the tantalum atom sputter trajectory. We use a quartz crystal monitor to detect sputter atom flux as a function of angle. The intensity of the sputtered atom flux as a function of angle show that the bcc tantalum single crystals strongly emits sputtered atoms along the <111> close packed direction as well as the <100> direction. Emission from the <110> direction is significantly less than that from the <111> direction. This fundamental property of tantalum sputtering is then used to accurately model thin film uniformity produced by magnetron sputtering from polycrystalline tantalum sputtering targets with strong (110), (111) and (100) orientations.

3:15 PM
Effect of Tungsten Sputtering Target Density on PVD Thin Film Properties: *Chi-Fung Lo*[1]; Peter McDonald[1]; Darryl Draper[1]; Paul Gilman[1]; [1]Praxair Electronics, Deposition Matls., 542 Rte. 303, Orangeburg, NY 10962 USA

Tungsten is commonly used for diffusion barrier, via and gate materials in semiconductor devices. Using physical vapor deposition (PVD), tungsten is deposited on to the silicon substrate. In order to achieve the desired film properties and minimize particle generation, control of the sputter target properties, which include density, purity, grain size and orientation, are essential. This study focused on the effect of sputtering target density on the thin film properties and defect generation. By controlling the powder sintering process, three sputtering targets from 80% to 100% of theoretical density were prepared. Using a sputtering tool, thin films were deposited on to the silicon wafers. The deposited films were then evaluated by four-point probe, XRD and SEM. In addition to film properties, the re-deposition behavior of the test targets during sputtering was also investigated by SEM. The re-deposited structures are known to be a potential particle source from the target. By monitoring the formatio n of re-deposition structure on the sputtered surface as function of target life, a correlation between target density and re-deposition behavior was established.

3:40 PM
MOCVD Ultra Thin Tungsten Nitride Films as Barrier Metal for Interconnect Applications: *Wei Pan*[1]; Robert Barrowcliff[1]; David Russell Evans[1]; Sheng-Teng Hsu[1]; [1]Sharp Labs of America, Inc., IC Process Tech., 5700 NW Pacific Rim Blvd., Camas, WA 98607 USA

This paper discusses the MOCVD processes, thin film properties, and barrier metal application for interconnects of the ultra thin W2N films deposited from a solid source W(CO)6 along with the gaseous NH3 through thermal decomposition. W(CO)6 vapor was carried into the deposition chamber by hydrogen to mix with NH3. The wafer was heated to the temperature between 350¢XC and 450¢XC, where the precursor vapor decomposed and reacted with NH3 to form W2N films. The as-deposited ultra thin W2N films, with thickness around 8nm, had resistivity as low as 275?Ýohm-cm and density as high as 17g/cm3. X-ray diffraction and X-ray reflectivity were used to characterize phase, density, and thickness. Growth kinetics was also studied. Standard bias-temperature-stress method was used to evaluate the barrier properties. Furthermore, the contact resistance of CVD Cu/CVD W2N/CVD Cu was measured on a Kelvin structure. Results indicate that CVD W2N has a great potential for barrier applications.

4:05 PM
The Effect of Texture of TiN Diffusion Barrier for Cu Metallization: Insoo Kim[1]; Dong Young Sung[1]; Min Gu Lee[1]; *No Jin Park*[1]; Beelyong Yang[1]; Jun Mo Yang[2]; Jung Kyu Ko[2]; [1]Kumoh National Institute of Technology, Sch. of Advd. Matls. & Sys. Engrg., 188 Shin Pyung Dong, Kumi, Kyung Buk 730-701 Korea; [2]Hynix Semiconductor Inc., Memory R&D Div., San 136-1, Ami-ri, Bubal-eub, Ichon-si, Kyoungki-do 467-701 Korea

TiN coated films have a good mechanical and conductivity properties, high thermal properties, and strong erosion and corrosion resistance. Therefore, TiN coated films have been used as a surface modification method in parts and as a diffusion barrier in semiconductors. The uniform and dense structure of thin films is influenced by the texture of films. It was good to have uniform and dense structure and bad to have an open columnar structure in TiN thin films. Therefore, the property of diffusion barrier of the TiN films in semiconductor also is related to the texture and microstructure of TiN coated layers. In this study, the relationships between the textures and the properties of TiN diffusion barrier for Cu metallization on SiO2/Si wafer semiconductor were investigated under different processing methods (PVD and MOCVD). The property of diffusion barrier for Cu metallization of RF sputtered (PVD) TiN is better than that of metal organic chemical vapor deposited (MOCVD) TiN thin films. Also the property of diffusion barrier for Cu metallization of PVD (111) textured TiN is better than that of PVD (100) textured TiN thin films on SiO2/Si wafer.

4:30 PM
Factors Affecting the Sputtering Performance of Magnetic Materials: *Christopher A. Michaluk*[1]; Henry L. Grohman[2]; David P. Field[3]; [1]Williams Advanced Materials, 2306 Cassard Cir., Gilbertsville, PA 19525 USA; [2]Williams Advanced Materials, 42 Mt. Ebo Rd. S., PO Box 1950, Brewster, NY 10509-8950 USA; [3]Washington State University, Dept. Mechl./Matls. Engrg., 239C Dana Hall, PO Box 642920, Pullman, WA 99164-2920 USA

D.C. magnetron sputtering relies on a magnetic field usually imparted by a fixed or rotating permanent magnet located behind the sputtering target, to trap free electrons within the plasma. The spiral trajectory of the contained electrons acts to increases the frequency of collisions with gaseous atoms, subsequently increasing the density of the plasma and the sputtering efficiency of the system. However, the magnetron sputtering of magnetic metals and alloys present unique challenges; the target's low magnetic permeability significantly hinders the magnetic flux from the permanent magnet to the plasma. This paper reviews how a combination of texture control and innovative target design is employed to enhance the sputtering performance of magnetic materials. Recently developed data revealing the correlation between global texture, characterized by Electron Backscatter Diffraction (EBSD), and the Pass-Through Flux (PTF) of a Co-4.5Zr-4.5Ta (CZT) alloy is also presented.

4:55 PM
Structure and Soft Magnetic Properties of Fe-Co-Ni-Based Multi-Component Thin Film: *Hung-Kai Chen*[1]; Shih-Hai Li[1]; Jenq-Gong Duh[2]; [1]National Tsing Hua University, Dept. of Engrg. & Sys. Sci., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Dept. of Matl. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Soft magnetic with suitable uniaxial anisotropy and high saturation magnetization are required for the high frequency application. Multi-components of Fe-Co-Ni-based soft magnetic thin films were deposited on the Si substrate by RF magnetron sputtering with different Ar/N_2 ratios at room temperature. The composition, crystal structure and surface morphology were analyzed by using electron probe microanalyzer (EPMA), X-ray diffraction (XRD), atomic force microscope (AFM) and magnetic force microscope (MFM). Without nitrogen doping, the domain distribution of the magnetic thin film arranged orderly and the domain thickness was about 1μm. The effect of N_2 content in the thin film on the magnetic properties was further discussed. Magnetic properties, such as saturation magnetization and coercivity, were determined with a vibrating sample magnetometer (VSM) and Magneto-Optics Kerr Effect (MOKE). The saturation magnetization (M_s) of the un-doped magnetic thin film is around 1.3T. It is expected that the derived magnetic thin film is a promising candidate for using in high frequency inductor.

5:20 PM
Growth Processes, Structure Peculiarities and Intergranular Magnetic Interaction in Electrodeposited Magnetic Nanostructures: *Vladimir Grigor'evich Shadrow*[1]; Anatolii Vasil'evich Boltushkin[1]; Ludmila Vas'vna Nemtsevich[1]; [1]Academy of Science, Inst. Solid State Physics, P. Brovki,17, Minsk, Belrus 220072 Belarus

Growth processes, structure peculiarities and magnetic properties of electrodeposited magnetic nanostructures have been investigated by means of EM,XRD,AFM,VSM and AGFM. Mechanisms of columnar and fine grained structure formation, regularities of content modulated FeCu and CoCu nanophase particles formation in the pores of aluminium anodic oxide are proposed, which accounts for the observed structure peculiarities.Intergranular magnetic interaction and magnetization reversal processes are investigated through the remanence and delta M curves analysis, magnetic viscosity measurements as well as rotational hysteresis loss and angular variations of hysteresis parameters measurements. The influence of the intergranular interaction on magnetic characteristics of the above model structures is discussed as well as evaluation of structural and magnetic films inhomogenities on the basis of the above techniques.

Shape Casting — The John Campbell Symposium: Structure and Properties

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM Room: 2008
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Qigui G. Wang, General Motors, Advd. Matls. Engrg., Pontiac, MI 48340 USA

2:00 PM
Effect of Oxide Films and Other Defects on the Fatigue Properties of Aluminium Castings: *Qigui G. Wang*[1]; Paul N. Crepeau[1]; John R. Griffiths[2]; Cameron J. Davidson[2]; [1]General Motors, Matls. Engrg., Powertrain, M/C 483-710-251, 895 Joslyn Ave., Pontiac, MI 48340 USA; [2]CSIRO Manufacturing Science and Technology, PO Box 883, Kenmore, QLD 4069 Australia

The fatigue properties of aluminum castings strongly depend on casting defects including oxide films and other defects. In this paper, the effect of oxide films on the fatigue properties of the most commonly used sand-cast, lost-foam-cast, squeeze-cast, and semi-solid-cast aluminum alloys are reviewed and also compared with the effect of porosity in these castings. In the presence of defects, the fatigue life of these alloys is dominated by defect sizes and the effect of other microstructural features becomes negligible on crack initiation. In comparison with pores of same size, oxide films have a less adverse effect on fatigue life.

2:25 PM
Estimating Ideal Tensile Properties of Cast Al-7%Si-0.6%Mg Alloys via Hybrid Mechanical Testing: *Murat Tiryakioglu*[1]; [1]Robert Morris University, Engrg., 6001 Univ. Blvd., Moon Twp., PA 15108 USA

Cast Al-Si-Mg alloys are in-situ metal matrix composites used in a variety of applications. During tensile deformation, cracks are initiated at major structural defects, such as porosity and bifilms. In the absence of major structural defects, however, Si particles in these alloys crack during tensile deformation. When damage to Si particles reach a critical value, fracture occurs. It has been suggested n the literature that ideal properties of these alloys are obtained when there is no damage to Si particles, as in compression. A new hybrid testing method built on the Kocks-Mecking work hardening model, that uses the entire tensile curve and Rockwell B hardness number will be introduced. The effect of Si particle shape and size on the ideal properties obtained by the new method will be discussed.

2:50 PM
The Influence of Strontium and TiB2 Addition on the Formation of Porosity in Aluminum Castings and its Effect on Final Fatigue Properties: *Peter D. Lee*[1]; Trevor C. Lindley[1]; [1]Imperial College London, Matls., Prince Consort Rd., London SW7 2BP UK

The influence of altering both the processing conditions and minor alloying additions upon the formation of microporosity in aluminium castings was investigated using traditional metallographic and microtomographic techniques. The confounding effects of eutectic modifiers (Sr), grain refiners (TiB2) and solidification rate upon the size, distribution and complex three dimensional shape of the pores formed

was characterised. For each condition tensile and S-N fatigue properties were measured in the heat treated condition. Using scanning electron microscopy, fractographic examination was performed, revealing that the fatigue cracks initiated at pores in almost all cases. The final fatigue performance was correlated to changes in microporosity and hence to casting conditions. The results of the study illustrate that the interaction of strontium and TiB2 is complex, with the as-cast pore morphology changing during heat treatment.

3:15 PM
A Study of Crack Initiation Sites in High Cycle Fatigue of 319 Aluminum Alloy Castings: *Glenn E. Byczynski*[1]; [1]Nemak, Canadian Ops., 4655 G N Booth Dr., Windsor, ON N9C 4G5 Canada

High cycle fatigue performance of aluminum castings is of high importance for the casting designer. Several recent fatigue studies in Al-Si-Mg alloys have found that in addition to porosity, oxide films are viable fatigue crack initiators. The current study analyzed the high cycle fatigue performance of 319 (Al-Si-Cu-Mg) alloy test bars produced in filtered and unfiltered gravity poured sand moulds. The fatigue life was found to be related to the size of defect acting as the crack initiation site. Scanning electron microscopy and energy dispersive spectroscopy were used to identify the nature of crack initiation sites. A linear elastic fracture mechanics crack growth model was found to be particularly successful at predicting the life of fatigue samples that initiated at oxide films. Having crack-like geometry, and a minute crack tip radius, oxide films effectively acted as preformed cracks. Consequently there was an absence of crack nucleation time, explaining the correlation of predicted propagation life to fatigue life.

3:40 PM Break

3:50 PM
The Effect of Si Content on the Ductility of Al-Si-Cu-Mg-(Fe,Mn) Casting Alloys: *Carlos H. Caceres*[1]; John A. Taylor[1]; [1]University of Queensland, Co-op. Rsch. Ctr. for Cast Metals Mfg., Sch. of Engrg., Brisbane, QLD 4072 Australia

The strength-ductility behaviour of a number of experimental Al-Si-Cu-Mg-(Fe,Mn) alloys has been compared to assess the effect of different levels of Si, Cu and Fe/Mn. Increased Si content improves the ductility of most alloys. This is particularly significant when the Fe content is high, but the effect is also important when either Cu alone or both Cu and Fe are present at high levels. A study of the intermetallics formed at different Si levels shows that the length of b-Al5FeSi platelets is reduced at high levels of Si. Similarly, Cu-rich intermetallics, which form extensive, interconnected particle clusters at lower Si content, appear as smaller, more isolated clusters at higher Si levels. It is thought that this refining of intermetallics leads to the observed increase in ductility at high Si content. Possible mechanisms for the refinement of the intermetallics during the solidification of high Si alloys are discussed.

4:10 PM
Hard Spot Defects in Al-Si Cast Alloys: *Xinjin Cao*[1]; John Campbell[2]; [1]Institute for Aerospace Research, Aeros. Mfg. Tech., 5145 Decelles Ave., Montreal, Quebec H3T 2B2 Canada; [2]University of Birmingham, Sch. of Metall. & Matls., Edgbaston, Birmingham B15 2TT England

Hard spot inclusions are one of the main machining defects encountered in cast aluminium alloys. The term "hard spot" designates an inclusion, generally of high hardness and may cause great trouble in machining operations. Basically there are mainly four types of "hard spot" inclusions in Al-Si-Mg cast alloys: oxides, intermetallics, refractory particles and cold drops. The oxides in Al-Si-0.4Mg cast alloys include Al2MgO4, Al2O3 and MgO. SiO2 inclusions are entrained sand or refractory material. Iron-rich inclusions are perhaps the most common of intermetallic hard spots in Al-Si alloys containing Fe and Mn. Other intermetallic hard spot inclusions are composed of TiAl3 and Ti(AlSi)2 in Al-Si-0.4Mg alloy containing Ti, and Al2Si2Sr or Al4Si2Sr in Al-Si-0.4Mg alloy containing Sr. Some intermetallic compounds may originate from the use or presence of grain refiners in the charges (added titanium boride/titanium carbide particles), or other nitrides, carbides, borides, etc. Cold drops are the small droplets of cast alloy themselves formed during excessively violent pouring.

4:30 PM
High Pressure Die Casting of Aluminium and Magnesium Alloys - Comparison of Microstructure Formation: *Hans I. Laukli*[1]; Otto Lohne[2]; Lars Arnberg[2]; [1]Hydro Aluminium, R&D Matls. Tech., Sunndalsøra 6600 Norway

Cold chamber high pressure die casting, (HPDC), is an important commercial process for the production of complex near net shape aluminium and magnesium alloy castings. The solidification characteristics related to the process and the alloys control the formation of grains and defects. This again has a significant impact on the mechanical properties of the castings. Significant amounts of pre-solidified metal flow into the die cavity during cold chamber HPDC of aluminium and magnesium alloys. The solid fraction commonly consists of single crystals which are termed externally solidified crystals (ESCs). The semi-solid metal fills the die cavity and, eventually, the microstructure consists of coarse ESCs and fine grains solidified in-situ. The prevalence of ESCs in the castings takes a variety of distributions and morphologies. Additionally, banded defects commonly form in the final stages of die filling. The nature of the bands depends on different parameters but tend to consist of segregation in Al die castings and porosity (and segregation) in Mg die castings. In the present work the microstructure formation in HPDC of aluminium (A356) is compared with magnesium (AM60B). The major similarities and differences are discussed with particular focus on the physical properties and solidification behaviour of the two alloys.

4:50 PM
Influence of Ca, Be and Mn on the Structure and Properties of 356 Alloy: *Sreeja Kumari*[1]; Raman Marimuthu Pillai[1]; Ballembettu Chadrasekhar Pai[1]; [1]Regional Research Laboratory (CSIR), Metals Procg. Div., Industl. Est. PO, Pappanamcode, Trivandrum, Kerala 695 019 India

Iron, the most harmful impurity in cast aluminium alloys, forms platelet iron intermetallic phase (β), which is detrimental to the mechanical properties and fracture toughness. Among the various methods available to neutralize its ill-effects, trace elements addition is commonly practiced. Effects of Ca, Be and Mn additions on the microstructure and properties of 356 alloy containing 0.6% Fe have been investigated. The alloy without Ca, Be and Mn additions exhibits poor mechanical properties due to the presence of long intercepting platelet iron intermetallics. Addition of Ca (300 ppm) has modified the eutectic Si structure and reduced the size of platelike Fe-intermetallic phases, while Be (0.2%) and Mn (0.3%) have changed the platelet morphology to Chinese script form. Mechanical properties have been improved with Ca, Be and Mn additions. The mechanisms for the microstructural changes have also been discussed.

5:10 PM
Fluidized Bed Heat Treatment of Cast Al Alloys: *Sujoy Chaudhury*[1]; Diran Apelian[1]; [1]MPI - WPI, Matls. Sci. and Engrg., 100, Inst. Rd., Worcester, MA 01609 USA

Improved mechanical properties in cast Al alloys are often achieved through heat treatment comprising of solution heat treatment, quenching, and ageing successively. With the impetus to be cost effective, it is imperative to reduce the long heat-treating times needed (i.e., up to 15-20 hours) without any reduction in performance. Fluidized beds provide an attractive heat treating technology for cast components with more efficient energy transfer and thereby reducing the net heat treatment time and enhance productivity. However, typical fluidized bed units are batch systems; therefore, further reductions in cycle time may be possible by using in-line continuous fluidized beds. In this paper, we will review the fundamentals of fluidized beds, highlight their advantages and examine applications of fluidized beds to heat treat cast Al-Si-Mg, and Al-Si-Cu-Mg alloys. Mechanical properties data along with the resultant microstructure as a result of extensive trials with fluidized bed technology will be reviewed and discussed.

Superalloys and Coatings for High Temperature Applications: Ceramic Materials for TBCs

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Tuesday PM — Room: Nob Hill A/B
February 15, 2005 — Location: San Francisco Marriott

Session Chairs: Sammy Tin, University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK; Roger C. Reed, University of British Columbia, Dept. of Metals & Matls. Engrg., Vancouver, BC V6T 1Z4 Canada

2:00 PM Invited
Stability Issues in Current and Emerging TBC Materials: Stephan Kraemer[1]; Ashutosh S. Gandhi[1]; Noemi R. Rebollo[1]; Rafael M. Leckie[1]; Felicia M. Pitek[1]; *Carlos G. Levi*[1]; [1]University of California, Matls. Dept., 1355 Engrg. II, Santa Barbara, CA 93106-5050 USA

Insulating layers for present thermal barrier systems comprise a metastable t' structure with composition ZrO_2-7.6%$YO_{1.5}$ (7YSZ) which exhibits remarkable stability under current operating conditions, but are deemed inadequate to satisfy the demands of upcoming engine designs. The current 7YSZ can also be severely degraded by chemically aggressive environments, e.g. those associated with molten silicate and/or sulfate/vanadate deposits. Emerging alternate compositions involve co-doping 7YSZ with RE oxides or its replacement by RE zirconates. Preliminary evidence suggests that both approaches can enhance thermal resistivity as well as the morphological stability of the microstructure at higher temperatures. As new oxides are incorporated, however, concerns arise about the phase stability of the coatings, their response to corrosive environments and their thermo-chemical compatibility with the underlying thermally grown alumina. These issues are examined, highlighting the thermodynamic and kinetic fundamentals determining the long-term stability of the coatings.

2:30 PM
Development of Ion Plasma Deposition Process for Turbine Coating Applications: *Don M. Lipkin*[1]; Joseph Rigney[2]; [1]GE Global Research, Ceram. & Metall. Tech., Bldg. K1, Rm. MB207, One Rsch. Cir., Niskayuna, NY 12309 USA; [2]GE Transportation, Aircraft Engines, 1 Neumann Way, MD M85, Cincinnati, OH 45215 US

The Ion Plasma Deposition process has proven to be a robust, versatile approach to depositing a broad range of coatings for use in high-temperature applications. We describe GE's experiences, including the application of Ion Plasma Deposition to advanced turbine airfoil coatings. The process is shown to exhibit excellent capability to transfer multi-component alloys with reproducible chemistry, coating thickness, adhesion, and microstructural quality. In developing the Ion Plasma Deposition process, GE adapted the cathodic arc process widely used for thin-film tool and decorative coating applications, thus enabling practical deposition of thick, complex-alloy metallurgical coatings. Using coating hardware specifically designed to optimize the uniformity of coverage on complex-geometry components, these modifications have proven key to the demonstration of Ion Plasma Deposition for turbine coating applications. Several examples of coatings deposited using the Ion Plasma Deposition process will be described, with special emphasis on the high-pressure turbine airfoil coatings.

2:55 PM
Cold-Shock Spallation Mechanism in Thermal Barrier Systems Subject to CMAS-Infiltration: *Sabine Faulhaber*[1]; Chris Mercer[1]; Anthony G. Evans[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

Three principle mechanisms govern the spallation of thermal barrier coatings (TBC) - stresses due to the formation of thermally grown oxide (TGO), erosion due to particle impact and foreign object damage (FOD) and infiltration with CMAS (calcium-magnesium-alumino-silicate) at high temperatures. A new mechanism for the spallation of the CMAS infiltrated layers has been identified: it involves cold-shock during shut-down of the engine. Calculations predict a critical penetration thickness that, if exceeded, makes the TBC layer susceptible to cold-shock in the presence of a vertical separation. The model is consistent with the experimental observations of an airfoil removed from service, gained from scanning electron microscopy (SEM) on metallographic cross-sections.

3:20 PM
Non-Destructive and Microstructural Characterization of Thermal Barrier Coatings: *Yongho Sohn*[1]; Balaji Jayaraj[1]; Sankar Laxman[1]; Barb Franke[1]; David Miranda[1]; Jaewon Byeon[1]; Vimal H. Desai[1]; [1]University of Central Florida, AMPAC & MMAE, Box 162455, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA

The durability and reliability of thermal barrier coatings (TBCs) play an important role in the service reliability and maintainability of hot-section components in advanced turbine engines for aero and utility applications. Photostimulated luminescence spectroscopy (PSLS) and electrochemical impedance spectroscopy (EIS) are being concurrently developed as complimentary non-destructive evaluation (NDE) techniques for quality control and life-remain assessment of TBCs. The evolution the residual stress, polymorphic constituents and impedance of TBC constituents with an emphasis on the thermally grown oxide were examined by NDE techniques as a function of thermal cycling. Results from PSLS and EIS were correlated to the microstructural development of TBCs examined by using a variety of microscopic techniques including focused ion beam in-situ lift-out, transmission and scanning transmission electron microscopy (TEM and STEM). TEM/STEM revealed many features of microstructural development that can influence the failure mechanisms of TBCs, and in turn affect the durability and reliability.

3:45 PM Break

4:05 PM
Simulation of the High Temperature Indentation of TBC with Columnar Microstructure: *Tao Xu*[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106 USA

The phenomena governing the durability of thermal barrier coatings (TBCs) are affected by their high temperature mechanical properties: especially the mechanisms of material removal upon particle impact. Some high temperature properties can be explored using an impression test. The utility of the test is contingent upon a method for deconvoluting aspects of the stress/strain response from load-displacement measurements. A numerical procedure having this attribute is described, and applied to TBCs with a columnar microstructure. The method elucidates the extent of the plastic deformation and densification as well as the column distortions caused by the impression. It is also capable of exploring the deformation heterogeneities observed experimentally, such as shear bands, by embodying salient constituent properties, such as the column width, contact friction, and inter-columnar porosity. Comparisons with measurements provide some understanding of the plastic response of several thermal barrier systems.

4:30 PM Invited
Thermal Barrier Coatings Under In-Service TMF Conditions: *Jeffery W. Brooks*[1]; Bernd Vermeulen[1]; [1]QinetiQ Ltd., Rm. 2008, Bldg. A7, Cody Tech. Park, Ively Rd., Farnborough, Hampshire GU14 0LX UK

Thermal Barrier Coatings (TBC) are fast becoming mainstream technology for use in gas turbine engines. The work to be presented assesses the effects of thermo-mechanical fatigue (TMF) on the degradation of the TBC. The inherent problem, when using standard induction heating during TMF tests, is that the specimen is heated from the inside and hence the temperature profile seen is the reverse of that experienced in service. An alternative heating system is described, which achieves temperature distributions close to in-service conditions. The test cycle profiles were derived from finite element analyses of a typical gas turbine blade using an anisotropic deformation code to compute the substrate strain field. The TMF tests were conducted with single crystal CMSX4 specimens coated using plasma vapour deposition (PVD) with an MCrAlY bond coat and a zirconia based TBC. The degradation and adhesion of the TBC, assessed using microscopy, will be compared with results from TMF tests performed using standard induction heating.

4:55 PM Invited
Thermal Barrier Effect and Thermal Cyclic Behavior of YSZ Coating with Rare Earth Oxide Addition: *Huibin Xu*[1]; Shengkai Gong[1]; [1]Beijing University of Aeronautics and Astronautics, Sch. of Matls. Sci. & Engrg., Xueyuan Rd. 37, Beijing 100083 China

The present TBC system with Y2O3 stabilized ZrO2 (YSZ) top coat exhibits an excellent high temperature performance, however, its thermal barrier effect has been greatly limited due to the thermal conductivity of YSZ top coat prepared by EB-PVD. Low conductivity thermal barrier coatings have been increasingly demanded to obtain thin coating configurations which still could achieve sufficient temperature reductions at higher engine operating temperatures. In this study, lattice distortion has been calculated by ab initio method on the system of YSZ added by various rare earth oxides to study the change of atomic oscillation frequency and thermal conductivity as well. Furthermore, some of YSZ with rare earth oxides have been deposited onto MCrAlY bond coat by EB-PVD method. The thermal barrier effect and thermal cyclic behaviors have been investigated as a function of the amount of the different kinds of rare earth oxides.

5:20 PM
Nondestructive Evaluation of Thermal Barrier Coatings Thermal Properties: *Ted D. Bennett*[1]; Fengling Yu[1]; [1]University of California, Mechl. & Environml. Engrg., Santa Barbara, CA 93106 USA

A nondestructive technique for determining thermal properties of thermal barrier coatings is developed. The measurement reveals both the thru-plane thermal diffusivity and the effusivity contrast between the film and substrate. From these measurements both the thermal conductivity and volumetric specific heat of the film may be determined. Because the measurement is nondestructive, this technique may be applied to evaluate film properties over the lifetime of a serviceable part. The measurement can be used to resolve the effects of structural variability in the film, high temperature sintering of pores, and localized delamination of the film from the substrate.

5:40 PM
Enhancing the Environmental Stability of Thermal Barrier Coatings: *Felicia Marie Pitek*[1]; Stephan Kraemer[1]; Carlos G. Levi[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

The hot corrosion of yttria stabilized zirconia (7YSZ) thermal barrier coatings by molten sulfate/vanadate deposits is discussed. The ensuing reaction of V_2O_5 with Y leads to the depletion of stabilizer from the initially "non-transformable" t' phase and rendering it susceptible to the deleterious monoclinic transformation upon cooling. The presence of a stable non-transformable tetragonal region in the ZrO_2-$YO_{1.5}$-$TaO_{2.5}$ phase diagram provides an opportunity to enhance the corrosion resistance of the coating. In principle, the addition of Ta reduces the activity of Y in the solid solution and presumably its susceptibility to reaction. Moreover, coating compositions can be selected to be tolerant to depletion without de-stabilization. Selected Y and Y+Ta doped compositions were made by precursor methods, consolidated into pellets and tested at 900°C using a Na_2SO_4-30mole%$NaVO_3$ corrodent. The resulting changes in microstructure and composition were characterized by XRD, SEM and TEM to compare the relative stabilities and differences in corrosion mechanisms.

Surface Engineering in Materials Science - III: Thin Films

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Tuesday PM Room: 2022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. & Mechl., Oviedo, FL 32765-7962 USA; Yip-Wah Chung, National Science Foundation, Civil & Mechl. Sys. Div., Arlington, VA 22230 USA

2:00 PM Invited
Stable Four Terminal Solar Cells Using Thin Film Silicon Technology: *Arun Madan*[1]; [1]MVSystems, Inc., 17301 W. Colfax Ave., Ste. 305, Golden, CO USA

For photovoltaics (PV) to be competitive with other forms of energy generation, a consensus is that thin film technologies could provide the route to lower costs. One of the most likely contender is thin film Silicon which has been commercialized (with efficiency,h, of 6-8%) by several companies. However, the intrinsic light induced degradation in amorphous Silicon (a-Si:H), remains an impediment. The degradation is strongly dependant on the thickness of the device used and can be reduced by the use of multi-junction (MJ) at the expense of complexity in fabrication. MJ type devices use several cells stacked on top of each other with differing band gaps (and thickness) to absorb a wider portion of the solar spectrum (e.g. a-SiH/a-SiGeH/a-SiGeH). As this two terminal (2-T) MJ device structure requires the same current from each constituent cell, it necessitates the use of relatively thick a-SiH junctions (~2000A) and the device generally degrades by ~20%. Further, the fabrication of a-SiGe:H requires GeH4 gas which is prohibitively expensive and since the gas utilization during production is normally <10%, the cost reduction of such PV panels may be difficult to realize. Hence the use of 2-T MJ solar cell with stable micro-(or rather nano-) crystalline Si (nc-SiH) as the bottom cell and a-Si:H as the top cell is attracting attention (termed as "micro-morph"). Such MJ (or tandem) solar cells can produce an initial h of ~14.5% in a small area (3cm2) and ~12% in large area modules. However, this structure also contains a thick (~ 4000A) a-Si:H junction (due to the current matching) and as a result majority of the power (~ 70%) emanates from the unstable thick a-Si:H portion with an inevitable degradation under light. We have developed a simpler 4 terminal (4-T) thin film Si based MJ solar cell configuration in which the current matching constraint is released from each constituent cell, e.g. two cells (a-SiH and stable low band gap material, such as nc-SiH)) are separated via an insulator. This allows the use of ultra-thin (<1000A) a-Si:H solar cell where instability is no longer an issue. This stable solar 4-T MJ design, has the potential to attain h >16%. We discuss the steps that are needed to go from the current efficiency of ~10% to >16%. We discuss the use of a modified pulsed PECVD (plasma enhanced chemical vapor deposition) technique, which provides a powerful way to improve the nc-SiH materials by altering the growth via a layer-by-layer technique. With an improvement in grain size to > 1000-2000A, as in fine grained polycrystalline-Si, that higher carrier mobility's (especially for holes) could be attained leading to longer minority carrier diffusion length and to a substantial increase in the open circuit voltage (from 480 mV to >650mV) thus opening the route to high efficiency (>16%) low cost stable thin film Si solar cells.

2:25 PM Invited
Coatings for Extreme-Condition Applications: *Yip-Wah Chung*[1]; [1]National Science Foundation, Civil & Mechl. Sys. Div., 4201 Wilson Blvd., Rm. 545.25, Arlington, VA 22230 USA

Coatings used in certain mechanical components operate under extremely demanding conditions. For example, coatings used in protecting cutting tools have to endure high contact stresses and tempera-

tures; nanometer-thick overcoats in computer hard disk drives have to provide the necessary wear and corrosion protection for extended periods; bearings and gears operate under high cyclic stresses, and the applied coatings must be fatigue-resistant and provide low friction/traction performance. This talk presents selected examples to illustrate some general principles in the selection and synthesis of such protective coatings.

2:50 PM
Enhanced Adhesion of Polyimide with Sputtered Copper by Argon Plasmas for Microelectronic Flex Substrates: *Yung-Sen Lin*[1]; Huang-Ming Liu[1]; Hsuan-Ta Chen[1]; [1]Feng Chia University, Dept. of Chem. Engrg., No. 100, Wenhwa Rd. Seatwen Dist., Taichung, Taiwan 407 China

Enhanced adhesion of polyimide films such as Kapton E(N) and Upilex S with sputtered copper by argon plasmas were investigated. Peel tests demonstrate this improvement. The enhanced adhesive strengths of sputtered coppers to polyimide films by argon plasmas were strongly affected by surface morphology and surface energy of polyimide films. The surface morphology of polyimide film was investigated by atomic force microscopy (AFM) and the surface energy of the polyimide film was calculated by the sessile drop method that indicated the surface roughness and the surface energy of polyimide films were much increased by argon plasmas that results in much increased peel strengths of sputtered coppers to polyimide films. Electron spectroscopy for chemical analysis (ESCA) observed the increased surface energy on polyimide films by argon plasmas were due to the increased surface compositions of O and N and the increased chemical bonds of C-O, C=O and C-N.

3:05 PM
Magnetic Semiconductor Thin Films - Magnetic and Transport Properties: *Lidia Jadwiga Maksymowicz*[1]; Maria Lubecka[1]; Beata Teresa Ciêciwa[1]; Zbigniew Stanislaw Sobkow[1]; Rita Szymczak[2]; Marek Sikora[3]; Czeslaw Kapusta[3]; [1]University of Science & Technology, Dept. of Elect., al. Mickiewicza 30, Kraków 30-059 Poland; [2]Polish Academy of Sciences, Inst. of Physics, al.Lotników 32/46, Warszawa 02-668 Poland; [3]University of Science & Technology, Dept. Solid State Physics, Reymonta 19, Kraków 30-059 Poland

Thin films of magnetic semiconductor (Cd1-yIny)[Cr2-2xIn2x]Se4 have been investigated as the perspective element of near-infrared detector. It is soft magnetic material, the energetic structure is modyfied by In dilution level and magnetic interaction. Also the increase of lattice parameter and changes of the local atomic environments with increasing the amount of In is detected. For CdCr2Se4, the magnetic state with reentrant transition (REE) is achieved. When In substitites Cr or Cd, we have the spin glass state (SG). The more complex type of magnetic ordering, the randomly canted (RC) was found when In substitutes Cr and Cd. The basic photoconductivity parameters as spectral voltage responsivity and detectivity were determined by lock-in measurement technique. It was found that for thin films in the state with REE the value of detectivity is the same order as for semiconductor materials.

3:20 PM
Surface Modification Through Inter Electrode Mass Transfer - A Phenomenon Intrinsic to Electro Discharge Machining: *T. A.J. Reddy*[1]; S. R.M. Voleti[1]; N. N. Ramesh[1]; [1]Osmania University, Mechl. Engrg., Coll. of Engrg., Hyderabad, Andra Pradesh 500 007 India

A significant observation in electro discharge machining is the occurrence of inter electrode material transfer. This phenomenon can be exploited for surface modification by suitable selection of process parameters, which can promote significant diffusion of selected electrode material into the machined surface for improved tribological properties. Microscopic analysis of such processed surface exhibits the presence of hard resolidified stratum with significant diffusion of electrode material revealed from Energy Dispersive X-ray Analysis. Consequently the micro hardness of resultant surface also increases significantly. Shaped electrodes fabricated by powder metallurgy technique and wire electrode with coating of selected materials are more effective in such mass transfer along with positive and negative polarity respectively. This increases electrode erosion and higher diffusion of electrode material into the processed surface. A major attraction of this approach is the simultaneous occurrence of machining and surface modification.

3:35 PM
Mechanically Induced Modification of an Al Surface and the Synthesis of Ceramic Coatings by Mixing in SiO_2 Particles: *Aghasi R. Torosyan*[1]; Laszlo Takacs[2]; Nshan H. Zulumyan[1]; Ashot A. Tataryan[1]; [1]National Academy of Sciences, Inst. of Gen. & Inorganic Chmst, 2-tup., Argutyan St. 10, Yerevan, Yerevan 375051 Armenia; [2]University of Maryland, Dept. of Physics, 1000 Hilltop Cir., Baltimore, MD 21250 USA

Surface modification of an Al plate and its interaction with amorphous and crystalline SiO2 powders during mechanical processing has been investigated. The Al plate was placed into a vibratory ball mill where it was exposed to the impact of the balls and mechanochemical interaction with SiO_2 powder for intervals of 20, 40, 60 and 120 min. Heat treatment at temperatures from 300°C to 600°C was applied to induce or continue the chemical reaction between the Al substrate and the SiO_2 particles forced into its surface layer. The mixing of the SiO_2 powder particles into the substrate, the evolution of the microstructure and the progress of the mechanochemical reaction between Al plate and SiO_2 were investigated by XRD and SEM. It was established that the products depend on the structural modification of the used SiO_2.

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4:00 PM Invited
Expansive Breadth Surface Engineering to Revolutionize the Energy Industry: *J. DeBarro*[1]; K. E. Rea[2]; [1]Mitsubishi Power Systems, Orlando Service Ctr., 2287 Premier Row, Orlando, FL 32809 USA; [2]University of Central Florida, AMPAC, MMAE, 4000 Central Florida Blvd., Orlando, FL 32816 USA

The materials required for the energy systems industry has and will be dominated with the modification of surfaces or surface science engineering. With the principal requirements of the materials' properties constantly shifting to resist higher temperature regimes and more corrosive environments to augment energy generation and efficiency, the surfaces of the materials become the essential building block of the entire industry. Enabling the manipulation and control of the materials surface through the processes of plasma forming, heat treating, and combinatorial alloy materials is paramount for development and progression of the energy systems and turbine repair manufacturing. Plasma spray and its relative process High Velocity Oxygen Fuel (HVOF) have lead the way in surface engineering breakthroughs in the recent decades and are becoming increasingly automated for manufacturing. New techniques of process and manufacturing evaluation are essential for overall control of strenuous requirements with a broad capacity of revolutionizing the industry.

4:25 PM
Texture and Microstructure Characteristics of Aluminium Thin Film on Stainless Steel: *Zhongliang Shi*[1]; *Hualong Li*[1]; Reza Bateni[1]; *Jerzy A. Szpunar*[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., Wong Bldg., 3610 Univ., Montreal, Quebec H3A 2B2 Canada

Aluminium thin film is ductile with good electrical and thermal conductivities. Particularly, it can be oxidized easily by artificial oxidation or anodizing process to provide an advantageous combination of a good conducting layer and a completely insulating layer which can be applied in integrated circuit technology, micromachining, optical devices and for the development of a rechargeable aluminium battery system. There are several processes available for coating aluminium on work pieces such as (1) thermal spraying coating; (2) hot dipping or galvano-forming; (3) roll binding; (4) PVD/CVD process, for example, sputter and plasma processes and (4) electrodeposition of aluminium. Except for electrodeposition process, any other techniques are rather expensive or run at high temperature or are limited by the arbitrary shape of work pieces. Aluminium coating obtained by electrodeposition has very good quality. However, the electrodeposition process of aluminium is much more complicated than many other metals from aqueous electrolyte as it is less noble than hydrogen, so it cannot be deposited from aqueous solution. In this paper, an organic nonaqueous electrolyte based on tetrahydrofuran with AlCl3 and LiAlH4 dissolved in is employed for electrodeposition of aluminium thin film on stainless steel at room temperature and its texture and microstructure on stainless steel substrate is characterized in details. The measured texture results show that the aluminium thin film has weak (210) and (111) fibre textures and their intensity maximum is 1.7. The observation of the microstructures under different current densities and coated time illustrates that nano-size aluminium particles (10~50nm) are packed together after initial nucleation and the obtained homogenous aluminium thin film is attached to the stainless steel substrate well and no cracks were observed in the film.

4:40 PM
Formation and Characteristics of Oxidation Film Produced by Heat Treatment of Ti and Ti Alloys: *Kimihiro Ozaki*[1]; [1]Advanced Industrial Science and Technology, Matl. Rsch. Inst. for Sustainable

Dvlp., Anagahora 2266-98, Shimoshidami, Moriyama, Nagoya 463-8560 Japan

An oxidation layer of a pure titanium and Ti alloys has interference colors. Especially, it is famous to make a design by an anodic oxidation method. The anodic oxidation layer has various colors, but the layer has disadvantage in erosion resistance and in heat resistance. An oxide film by heat treatment in the atmosphere has higher erosion resistance and heat resistance than that by anodic oxidation. However, the former has fewer kinds of color and worse controllability of the thickness than the latter. Then in this, the formation of oxide films produced by heat-treating Ti and Ti-Si alloys and characteristics of interference color of the film were investigated for obtaining stability and variety of color.

4:55 PM
Microstructures and Ferroelectric Properties of Barium Strontium Titanate Thin Films: Chen Hong Wei[1]; [1]University of Electronic Science & Technology of China, Inst. of Microelect. & Solid State Elect., Chengdu, Sichuan 610054 China

Barium strontium titanate (BST) thin films were prepared by RF magnetron sputtering. The microstructures and ferroelectric properties of Ba0.6Sr0.4TiO3 films were investigated. The composition of BST thin film with tetragonal perovskite structure was near to BST ceramic target. Nanometer-sized domains were observed by piezo-response force microscopy (PFM). The critical size is found to be between 28nm to 33nm from a multi-domain to a mono-domain. The dielectric constant-electric field curves and polarization hysteresis loops of BST films have been measured. At 1kHz the dielectric constant, tunability and dielectric loss of the Ba0.6Sr0.4TiO3 film with thickness 280nm are 562,25.6% and 0.016, respectively. The remanent polarization (Pr) and the coercive electric field (Ec) of the BST film are 1.2iC/cm2 and 42.9kV/cm at room temperature, respectively. The år-V and P-V curves show a voltage shift toward the negative side and asymmetry, which may be caused by asymmetric potential barriers at the upper and bottom interfaces.

5:10 PM
A Model for Dielectrics Nonlinearity of Ferroelectric Thin Films: Fu Chun Lin[1]; [1]University of Electronic Science & Technology of China, Inst. of Microelec. & Solid State Elect., Chengdu, Sichuan 610054 China

Dielectric nonlinearity is an important characteristic of ferroelectric thin films. Based on the characteristics of hysteresis loops and å-E curves in thin films, a novel model for dielectric nonlinearity is established. Many parameters, intensively influencing the ferroelectric properties of thin films, such as grain size, film thickness and the barrier height at the interface are involved. In this model, the barrier layers were modeled in terms of p-n junction equivalent circuits. The accuracy of the model predictions is quantitatively verified with the data from reference.

Texture and Microstructure in Thin Films and Coatings: Joint Session with Refractory Metals in Electronic Applications: Texture and Thin Films

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Tuesday PM Room: 3010
February 15, 2005 Location: The Moscone Center

Session Chair: Christopher Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA

See Refractory Metals in Electronic Applications Symposium on page 214 for schedule.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session IV

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Tuesday PM Room: 3003
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Ken R. Elder, Oakland University, Physics, Rochester, MI 48309 USA; Jong K. Lee, Michigan Tech, MSE, Houghton, MI 49931 USA

2:00 PM Opening Remarks

2:05 PM Invited
Phase Transformations in Ferroic Materials: *David E. Laughlin*[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Ferroic materials are those materials which on cooling, transform from a high symmetry phase into a lower symmetry phase without any reconstruction of the underlying crystal structure. The low symmetry phase is composed of two or more orientation variants, or domains. An important additional feature of ferroic materials is that their domains can be transformed into each other by the application of an appropriate external field. Ferroic materials are being used extensively as sensors, actuators, shape memory alloys and in other "smart material" applications. In this paper an overview will be presented of the various phase transformations and symmetry changes that are involved in selected ferroic materials. A general classification of ferroic phase transformations in terms of symmetry changes will also be given. This research has been supported in part by the Data Storage Systems Center at CMU.

2:30 PM Invited
Thermodynamics and Evolution of Ferroic Domain Structures: J. X. Zhang[1]; Y. L. Li[1]; S. Choudhary[1]; *L. Q. Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 102 Steidle Bldg., Univ. Park, PA 16802 USA

One of Khachaturyan's seminal contributions to theoretical materials science is his mesoscopic elasticity theory developed about 35 years ago. It has been the basis for the prediction and simulation of precipitate morphologies and microstructures during various phase transformations. In this presentation, the applications of Khachatuyran's elasticity theory and phase-field approach to ferroic domain structures will be discussed. Emphasis will be on systems that are simultaneously ferroelectric and ferroelastic or ferromagnetic and ferroelastic. Examples to be discussed include PbZrxTi1-xO3 (PZT) piezoelectric thin films and the ferromagnetic shape memory alloy Ni2MnGa. While PZT is a primary ferroelectric and secondary ferroelastic, Ni2MnGa is both a primary ferromagnetic and a primary ferroelastic. Similar approach can be applied to domain structures of ferromagnetoelectric multiferroics that are simultaneously ferromagnetic and ferroelectric.

2:55 PM
The Strain-Induced Paramagnetic to Ferromagnetic in Intermetallic Compounds: *Ian Baker*[1]; Markus W. Wittmann[1]; [1]Dartmouth College, Thayer Sch. of Engrg., 8000 Cummings Hall, Hanover, NH 03755 USA

A strain-induced paramagnetic to ferromagnetism transition has been observed in a number of intermetallic compounds containing magnetic moment-bearing elements. This phenomenon has been quantitatively modeled in FeAl, using the local environment theory, based on the idea that the ferromagnetism arises from APB tubes, where Fe atoms can have >=3 like nearest neighbors. It is shown that the saturation magnetization, Ms, depends on both the Fe:Al ratio, the ternary atoms present and on the degree of deformation. The behavior of both

ternary FeAl-based alloys and of the L21-structured intermetallic compound Fe2AlMn will be explained by considering the site preferences of ternary atoms on the Fe and Al sublattices. TEM observations of APB tubes in lightly-strained single crystals of FeAl and Fe2AlMn will be presented. The effects of plastic strain on the magnetic properties of L12-structured Co3Ti are also outlined. Research sponsored by NSF grant DMR 9973977 and NIST grant 60NANB2D0120.

3:10 PM
Effects of Si Addition on the Microstructure and Magnetic Properties of Permalloys Fabricated by Melt Drag Casting: *Kyoung-Mook Lim*[1]; Chan-Gyung Park[1]; Jung Namkung[2]; Mun-Chul Kim[2]; [1]Pohang University of Science & Technology, Matls. Sci. & Engrg., San 31, Hyojadong, Namgu, Pohang, Kyungbuk 790-784 Korea; [2]Research Institute for Science and Technology, Casting Process Rsch. Team, San 31, Hyojadong, Namgu, Pohang, Kyungbuk 790-785 Korea

Effects of Si addition on the microstructure and magnetic properties of 79Ni-Fe based permalloys, fabricated by melt drag casting, has been investigated. The Permalloy strips of 50mm width obtaining various Si contents were successfully prepared by melt drag casting, which was newly applied to fabrication of the permalloy strips in this study. In order to understand the relationship between magnetic properties and Si contents, microstructure and texture were extensively analyzed by TEM and EBSD. Increasing Si contents improved permeability of the permalloys in low Si content region below 2% due to the enlarged grain size. In high Si content region more than 2%, the formation of Ni3Fe ordered phase was revealed remarkably, which caused the drastic reduction of permeability. In addition, the formation of Ni3Fe was promoted by higher temperature annealing at fixed Si content. From these results, the ordering kinetics of Ni3Fe in Si added permalloys will be discussed.

3:25 PM
Effect of High Magnetic Field on Microstructural Evolution in Fe-1%Si: *T. A. Bennett*[1]; R. A. Jaramillo[2]; J. B. Wilgren[2]; R. Kisner[2]; G. Mackiewicz-Ludtka[2]; G. M. Ludtka[2]; P. N. Kalu[3]; A. D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 3325 Wean Hall, Pittsburgh, PA 15217 USA; [2]Oak Ridge National Laboratory, Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6065 USA; [3]FAMU-FSU College of Engineering, Mechl. Engrg. Dept., 2525 Pottsdamer St., Tallahassee, FL 32310-6046 USA

Microstructural and texture evolution and the change in grain boundary character distribution have been studied in Fe-1%Si as a function of field strength. Magnetic fields varying from 1.5T to 30T have been applied parallel to the sample rolling direction at 600°C and 787°C, i.e. below and above the Curie temperature. When annealed above the Curie temperature for 1 hour at 1.5T, samples experienced a drastic increase in average grain size from 50 microns to 220 microns. No significant texturing occurred but there was an increase in the cube texture component. The cube component persisted up to 30T but its volume fraction decreased relative to that observed for lower fields. The grain boundary character distribution also exhibited a higher percentage of low angle boundaries with increasing field strength; hence, a correlation was observed between the magnetic field strength and the microstructural changes in Fe-1%Si.

3:40 PM Break

4:05 PM Invited
Applications of Numerical Simulations of Displacive and Diffusional Transformations: *Greg Olson*[1]; [1]Northwestern University/ QuesTek, Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60015 USA

Application of a numerical "phase-field" approach based on rigorous Landau-Ginzburg models of martensitic transformations has quantified the boundary between classical and Cahn-Hilliard nonclassical behavior in both heterogeneous and homogeneous nucleation. This has allowed confident predictive application of classical approximations in computational materials design of steels and shape-memory alloys. Application of precipitation simulation to the accelerated development and qualification of new alloys requires higher computational efficiency provided by sharp-interface models. The new PrecipiCalc code grounded in multicomponent thermodynamics and diffusion has demonstrated an optimal combination of fidelity and efficiency for both process optimization at the component level and prediction of part-to-part variation in manufacturing.

4:30 PM Invited
Quantitative Phase Field Modeling: *Alphonse Finel*[1]; Yann Le Bouar[1]; Quentin Bronchart[1]; Guillaume Boussinot[1]; [1]ONERA-CNRS, LEM, BP 72, 29 Ave. Div. Leclerc, Châtilllon 92322 France

Phase Field methods are intensively used to predict the mesoscopic behavior of complex materials and its range of applicability has been extended to a wide range of physical phenomena. One of the directions which are today the subject of an intensive activity is its ability to be quantitative for a specific system. We will address this last aspect, and discuss different points of view for devising a quantitative Phase Field modeling.

4:55 PM Invited
A Comparison of the Phase Field and Monte Carlo Simulations of the Interaction Between a Grain Boundary and a Coarse Particle: *Bala Radhakrishnan*[1]; Gorti Sarma[1]; Yunzhi Wang[2]; [1]Oak Ridge National Laboratory, Computer Sci. & Math., Bldg. 5600, MS 6008, Oak Ridge, TN 37831-6008 USA; [2]Ohio State University, Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

The paper presents large scale, three-dimensional, Potts model simulations of the interaction between an initially straight grain boundary driven by a bulk stored energy difference across the boundary and a coarse grain boundary particle. The Potts model simulations show that the interaction energy is significantly affected by the choice of the lattice temperature and the driving force. The boundary velocity responds appropriately to changes in the magnitude and direction of the driving force only at high lattice temperatures or high driving forces where the boundary is flexible enough to bend locally near the particle. At lower lattice temperatures or low driving forces, the boundary response is "stiff" and this leads to an increase in the initial energy before it starts to decrease as the boundary approaches the particle. The Potts model simulations are compared with the predictions of the phase field model under identical initial conditions.

5:20 PM
Study on the Abilities of the Phase Field Method in Simulating Grain Growth in Materials Containing Second-Phase Particles: *Nele Moelans*[1]; Bart Blanpain[1]; Patrick Wollants[1]; [1]K. U. Leuven, Dept. of Metall. & Matls. Engrg., Kasteelpark Arenberg 44, B-3001 Heverlee Belgium

A Phase Field model, together with a convenient implementation technique, has been worked out for grain growth in materials containing inert, immobile particles. Simulations were performed for several sizes and volume fractions of the second-phase particles. Properties frequently measured in grain growth experiments were calculated for the simulated systems and compared with experimental values. Two main difficulties arose. (1) The essential microstructural features, viz. grains, second-phase particles and grain boundaries, range in size over very different scales. This leads to long computational times and huge computer memory requirements. (2) It is difficult to reproduce grain boundary energy and thickness, which are important parameters determining the interaction between grain boundary and particle. Both difficulties are general drawbacks of the Phase Field method in its current state of development. This work can bring new insights in grain growth in multi-phase materials and contributes to a further development of the Phase Field simulation technique.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Equal Channel Angular Pressing

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Tuesday PM Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sai V. Raj, NASA Glenn Research Center, Matls., Cleveland, OH 44135 USA; Marco J. Starink, University of Southampton, Matls. Rsch. Grp. Sch. of Engrg. Sci., Southampton SO17 1BJ UK; Patrick B. Berbon, Rockwell Scientific, Thousand Oaks, CA 91360 USA; Kenji Higashi, Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Sakai, Osaka 599-8531 Japan

2:00 PM
Interactions Between Microstructure and Texture Evolution in the Early Stages of ECAE: *Irene J. Beyerlein*[1]; David J. Alexander[1]; Saiyi Li[1]; Carl T. Necker[1]; Qing Xue[1]; [1]Los Alamos National Laboratory, Theoretical Div., MS B216, Los ALamos, NM 87545 USA

Through severe plastic deformation and strain path changes, ECAE imparts substantial microstructural changes in the material at multiple length scales. Our pursuit involves developing a multi-scale model that couples the mechanics starting at the subgrain level, increasing to the grain and aggregate levels and eventually ending at the macroscopic length scale of the sample. Understanding the impact of the strain path changes associated with various ECAE routes requires appreciation of the evolution of microstructure at these length scales in each pass. Combining multi-scale modeling (substructure evolution, polycrystal models, and finite element simulation) and experimental techniques (TEM, OIM, neutron diffraction, and mechanical testing), we investigate the relationship between substructure and texture evolution in the early stages of the ECAE process for routes Bc and C. We present our findings on how the grain morphology, substructure, and texture developed in the new strain path (either a mechanical test or subsequent ECAE pass) interact with those generated in the previous pass.

2:15 PM
Anisotropy in Mechanical Properties of High-Purity Copper Processed by Equal Channel Angular Extrusion: *David J. Alexander*[1]; Irene J. Beyerlein[2]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, T-3, B-216, Los Alamos, NM 87545 USA

High-purity oxygen-free electronic copper has been processed by equal channel angular extrusion for up to 4 passes at room temperature, using 90° tooling, by either route Bc or C. Small tensile specimens, as well as compression cubes, were sectioned along the length of the billet by electrodischarge machining, and tested in the as-processed condition. The ECAE-processed material was anisotropic, with the strength in the direction perpendicular to the plane formed by the inlet and outlet channels being greater than that in the other directions. Also, the yield strength in compression was greater than the yield strength in tension. The results for routes Bc and C will be compared, and viscoplastic self-consistent modeling will be used to help interpret these results.

2:30 PM
Texture Development During the Equal Channel Angular Pressing of Magnesium Alloys AZ31, AZ80, Mg-4wt%Li, WE43, and ZK60: *Sean R. Agnew*[1]; P. Merhotra[1]; T. M. Lillo[2]; G. M. Stoica[3]; P. K. Liaw[3]; [1]University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; [2]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID USA; [3]University of Tennessee, Matls. Sci. & Engrg., Knoxsville, TN USA

In recent years, Terry Langdon has contributed to the literature of severe plastic deformation more than anyone. One notable contribution has been his group's (and collaborators') exploration of the effect of various processing routes possible via equal channel angular pressing (ECAP), i.e. A, Ba, Bc, C, etc., on the microstructure and properties of many metals and alloys. Defining and describing these various strain paths has helped to clarify many findings in recent years. The effect of ECAP processing on the properties of magnesium alloys originally focused on the potential for superplastic behavior. However, a report of tremendously improved room temperature ductility stressed the possible importance of the texture developed through ECAP. As a result, the texture development of 5 magnesium alloys representing 4 major classes of alloys (Mg-Al, Mg-Li, Mg-Rare Earth, and Mg-Zn based) has been explored. Each class exhibits a distinct texture evolution, which may be linked to distinctions in the activities of the various underlying deformation mechanisms. These connections may be made more quantitatively through the use of computer simulation of the texture using polycrystal plasticity models. The implications for properties will be discussed and demonstrated, in select cases.

2:45 PM
Creep Processes in Pure Aluminium Processed by ECAP Technique: *Vaclav Sklenicka*[1]; Jiri Dvorak[1]; Petr Kral[1]; Milan Svoboda[1]; [1]Academy of Sciences of the Czech Republic, Inst. of Physics of Matls., Zizkova 22, Brno CZ-616 62 Czech Republic

Creep tests were conducted on pure aluminium processed by ECAP at temperatures 423-523K and a 10-25MPa. Specimens were examined by transmission and scanning electron microscopy equiped with an EBSD facility. The amount of grain boundary sliding was experimentally determined. Based on the results, it is suggested that creep of the ECAPed aluminium occurs by diffusion-controlled movement of dislocations and by grain boundary sliding. The coexistence of a dislocation climbing process and grain boundary sliding may explain the observed decrease of the creep resistance with increasing number of ECAP passes. Since high-angle grain boundaries are necessary in order to achieve grain boundary sliding, an increase in the fraction of high-angle boundaries with increasing number of ECAP passes will essentially lead to an increasing contribution of sliding to the total creep strain as it was observed experimentally.

3:00 PM
Ultrafine Grained Steels Processed by ECAP: *Kyung-Tae Park*[1]; Young-Kook Lee[2]; Dong Hyuk Shin[3]; [1]Hanbat National University, Div. of Advd. Matls. Sci. & Engrg., Taejon 305-719 S. Korea; [2]Yonsei University, Dept. of Metall. Engrg., Seoul 120-749 S. Korea; [3]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Kyunggi-Do 425-791 S. Korea

ECAP is very useful to modify the microstructure of ferrite-pearlite steels, resulting in considerable enhancement of their mechanical properties. The microstucture and mechanical properties of ECAPed ferrite-pearlite steels are reviewed, specifically focusing on fabrication of ultrafine grained (UFG) dual phase steels and their mechanical properties. In addition to ultrahigh strength, the UFG dual phase steels fabricated by ECAP exhibited extensive strain hardening unlike other UFG metallic materials.

3:15 PM
A Quantitative Study of Cavity Development in the Tensile Testing of an Aluminum Metal Matrix Composite Processed by ECAP: *Megumi Kawasaki*[1]; Yi Huang[1]; Cheng Xu[1]; Minoru Furukawa[2]; Zenji Horita[3]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan

An Al-6061 metal matrix composite, reinforced with 10 vol. % of fine Al_2O_3 particulates, was subjected to equal-channel angular processing (ECAP) through one pass at 298 K and an additional 11 passes at a temperature of 473 K. The mechanical properties were investigated through tensile testing of both as-received and as-pressed materials over a range of strain rates at 873 K. The results show that higher elongations are achieved in both materials at the faster strain rates. A detailed examination was conducted to analyze the extent of the internal cavitation in these materials after tensile testing to failure. This paper compares the development of cavities in the composite material in both the as-received and as-pressed conditions.

3:30 PM Break

3:45 PM
An Investigation of the Deformation Process During Equal-Channel Angular Pressing of an Aluminum Single Crystal:

Minoru Furukawa[1]; Yoshiyasu Kawasaki[2]; Yuichi Miyahara[2]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Fukuoka University of Education, Dept. of Tech., 1-1 Akama-Bunkyomachi, Munakata, Fukuoka 811-4192 Japan; [2]Kyushu University, Dept. of Matls. Sci. & Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg., Los Angeles, CA 90089-1453 USA

This investigation examines the deformation process when an aluminum single crystal is subjected to equal-channel angular pressing (ECAP) using a die having a channel angle of 90°. The crystal within the entrance channel was oriented so that the (111) slip plane and the [110] slip direction were inclined by 20?a in a counter-clockwise direction from the theoretical shear plane and direction, respectively. The crystal was pressed through one pass at room temperature and examined by orientation imaging microscopy. After passing through the shear plane, the initial orientation remained in the upper portion within the exit channel but there was a rotation by ~60?a in a counter-clockwise direction near the lower die wall. These observations show pure shear occurs in the upper portion of the specimen but there is a rotation in the lower portion due to the curvature at the point of intersection of the two channels.

4:00 PM

Effect of Acute Tool-Angles on Equal Channel Angular Extrusion/Pressing: *Anumalasetty Venkata Nagasekhar*[1]; Yip Tick-Hon[1]; Li Suixiang Sean[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Singapore 639 798 Singapore

Materials processed by equal channel angular extrusion/pressing (ECAE/ECAP) are influenced by factors like amount of strain induced, and uniformity of strain distribution. These factors in turn depend on parameters like tool angles (channel angle and outer corner angle), material properties, and friction between the die and the sample. Acute tool-angles can increase the strain induced in the material for minimum number of passes. Such increased strains can yield ultrafine grains and high fraction of high angle grain boundaries, which enhance mechanical and superplastic properties. However, with acute tool angles the deformation and punch pressure requirements will be more stringent. Hence, to study the effect of the parameters, finite element simulations of ECAE were carried for channel angles of 60°, 75° and 90°, and with various outer corner angles by using Abaqus/Explicit. Factual phenomena like strain hardening of material and friction were considered to obtain realistic deformation behavior.

4:15 PM

Influence of Severe Plastic Deformation on Aging of Al-Mg-Si Alloys: *Emanuela Cerri*[1]; Leo Paola[1]; [1]University of Lecce, Dept. Ingegneria dell'Innovazione, via Arnesano, Lecce 73100 Italy

The influence of SPD induced by ECAP on microstructural modifications and aging effect was studied in two different Al-Mg-Si alloys. The microstructure of both alloys in different heat treated and deformed state was characterized by X-Ray diffraction, SEM and optical microscopy. The effect of artificial aging was studied after SPD in the as received state and after SPD performed in the solution condition. The aging effect was followed by microhardness and electrical conductivity measurements. At higher aging temperature (i.e 170°C and 190°C) the alloys showed an increasing softening with time and number of pressing due to recovery or/and grain coarsening effect. At low temperature of aging (110°C) only the solutioned alloy exhibited an hardness increasing with time due to an enhanced precipitation, induced by an higher dislocation density present at low temperature, and a reduced recovery phenomena.

4:30 PM

Creep and Superplasticity in a Spray-Cast Aluminium Alloy Processed by Equal-Channel Angular Pressing: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

A commercial spray-cast aluminium alloy having a composition of Al-11.5% Zn-2.5% Mg-0.9% Cu-0.2% Zr was processed by equal channel angular pressing (ECAP) at a temperature of 473 K. The alloy achieved a grain size reduction from ~2.1 μm to ~0.3 μm and it was observed that there is a breaking of precipitates during ECAP. The ultrafine-grained structure of the as-pressed alloy was reasonably stable up to temperatures of the order of ~670 K. Tensile and creep tests were conducted using the alloy in both the unpressed and the as-pressed conditions. The results show that the alloy subjected to ECAP achieved superplastic elongation to failure of >1000% at high stain rates of $\geq 10^{-2}$ s^{-1} when testing at 673 K. Also, faster creep rates were observed in the as-pressed material when testing at the same applied stress and testing temperature.

4:45 PM

Microstructural Evolution in a Spray-Cast Aluminium Alloy During ECA Pressing and in Subsequent Heat Treatment: Nong Gao[1]; *Marco Jan Starink*[1]; Minoru Furukawa[2]; Zenji Horita[3]; Cheng Xu[4]; Terence G. Langdon[4]; [1]University of Southampton, Matls. Rsch. Grp., Sch. of Engrg. Scis., Highfield, Southampton SO17 1BJ UK; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-4192 Japan; [4]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The microstructures of a spray-cast Al-7034 (Al-Zn-Mg-Cu) alloy after processing through equal-channel angular pressing (ECAP) were studied using electron back-scatter diffraction (EBSD) and differential scanning calorimetry (DSC). The ageing response and recrystallisation softening post ECAP were studied by hardness testing and DSC. The results demonstrate there is a relatively rapid increase in the fraction of high-angle boundaries during the initial ECAP passes and a subsequent more gradual increase in further passes. The crystallographic textures introduced by ECAP are analysed. For the Al-7034 alloy, the DSC analysis identifies the occurrence of several thermal effects during heating involving the formation, coarsening, dissolution and melting of the eta phase.

5:00 PM

Scaling Up of Equal Channel Angular Pressing (ECAP) and its Effect on Mechanical Properties, Microstructure, and Hot Workability of AA 6061: *Prabir K. Chuadhury*[1]; Raghavan Srinivasan[2]; [1]Materials and Processing Resources, Inc., 4120 Foxtail Ln., Plano, TX 75024 USA; [2]Wright State University, Mechl. & Matls. Engrg. Dept., Dayton, OH 45435 USA

Equal Channel Angular Pressing (ECAP) at various cross sectional areas were performed on aluminum alloy 6061 to study the effect of scaling up on the mechanical properties, microstructure, and the hot workability of the alloy. In this study, annealed AA 6061 was subjected to severe plastic deformation at room temperature by ECAP (Route BC), producing 12.5 mm (0.5 inch), 50 mm (2.0 inch), and 100 mm (4.0 inch) square billets. The mechanical properties and microstructure of as pressed alloy were examined as a function of the extent of deformation and the cross sectional area of the billets. Also hot workability was determined by forging industrial parts at various temperatures. Results indicate that the average grain size after ECAP is of the order of 0.5 mm. This grain refinement resulted in enhanced hot workability at 315 C (600 F), indicating applicability of ECAP processed material in Forging Industry.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Poster Session

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Tuesday, 5:15-7:00pm Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Intermediate-Temperature Equal Channel Angular Extrusion of Electron-Beam Melted Crystal Bar Zirconium: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA

Electron-bean melted crystal bar zirconium was processed by equal channel angular extrusion at temperatures from 200 to 450°C, for 4 passes with tooling with a 105° angle. The deformed material showed evidence of deformation by twinning, and was finely subdivided. Annealing was performed at temperatures from 400 to 550°C. Full recrystallization occurred at annealing temperatures of 500°C or higher; only partial recrystallization occurred below this temperature. The recrystallized grain size increased as the annealing temperature in-

creased. The effect of the deformation temperature on the resultant microstructure and texture will be discussed.

Processing of Tantalum by Equal Channel Angular Extrusion with the Aid of Electroplated Surface Layers: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA

Tantalum is difficult to process by conventional extrusion as it has a marked tendency to gall on the tooling. Equal channel angular extrusion of Ta was successfully performed at room temperature by using thick electroplated layers of Cu, Ni, or Ni + Cu to prevent contact between the Ta billet and the tooling. The electroplated layers successfully withstood 4 passes for tooling with a 105° angle between the channels. The processed material was annealed in vacuum at temperatures ranging from 800 to 1200°C. The recrystallized grain size increased as the annealing temperature increased.

An Upper Bound Analysis in ECAE: *Burhanettin Semsi Altan*[1]; [1]Michigan Technological University, Mechl. Engrg. & Engrg. Mech., 1400 Townsend Dr., Houghton, MI 49931-1299 USA

An upper-bound analysis has been carried out to investigate the plastic deformation behavior of the material during the 90-ECAE process. Material is assumed to be rigid-plastic Von Mises material. The deformation model proposed in this study allows plastic deformation to occur in a region symmetric with respect to plane formed by the intersection of the two extrusion channels. Formation of the plastic deformation region is investigated as a function of three parameters: the radius of the outer corner of the die (actually the interface between the dead metal zone and the deformation zone), the radius of the inner corner of the die, and the friction on the die walls. For simplicity, the inner and outer corners of the die are taken to be concentric circles. The relationship between the radius of the inner corner and the friction coefficient has been developed which governs the size of the deformation region.

Analytical Electron Microscopy of Reaction Ball-Milled Y-Ni-O Nanostructures: *James Bentley*[1]; David T. Hoelzer[1]; Laurent Chaffron[2]; Dorothy W. Coffey[1]; Kathy A. Yarborough[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]CEA Saclay, Service de Reserches de Métallurgie Physique, Gif sur Yvette 91191 France

Powders of NiO and Ni2Y were ball-milled under vacuum at 30 and 100°C for 144h in an instrumented mill at CEA, Saclay to induce the reaction 3 NiO + 2 Ni_2Y —> 7 Ni + Y_2O_3. Quantitative compositional mapping at ~1 nm resolution has been performed at ORNL on the ~10-nm-scale microstructures by energy-filtered transmission electron microscopy (EFTEM) and spectrum imaging techniques. Electron microscopy specimens were made from as-milled powders by focused ion beam (FIB) milling. The atomic diffusion necessary to accomplish the almost complete reduction of NiO and form the observed nanostructures cannot occur at the 30 or 100°C. Temperature spikes, from either localized exothermic chemical reactions or the conversion of ball kinetic energy upon impact, and the role of vacancies injected during the severe plastic deformation (the required activation energy is then just the vacancy migration energy, not migration plus formation energies) are considered as enabling mechanisms.

Applicability of the Groove Pressing Technique for Grain Refinement in Commercial Purity Copper: *Krishnaiah Arkanti*[1]; *Uday Chakkingal*[1]; P. Venugopal[1]; [1]Indian Institute of Technology, Metal Forming Lab., Metallurgl. & Matls. Engrg., Madras, Chennai, Tamil Ladu 600 036 India

There is currently a lot of interest in producing bulk ultra-fine grained materials by imposing severe plastic deformation. This interest arises because a reduction in grain size leads to increases in the strength and toughness of the material at ambient temperatures. In the current study, specimens of commercial purity copper were subjected to large strains using the groove pressing technique. Dies were designed and fabricated with the groove angle (q) of 45° so that a single pressing yields a shear strain of 1, at deformed region. This is equivalent to an effective true strain of 0.58 and each series of four pressings yields a homogeneous effective strain of 1.16 throughout the sample. Copper specimens were subjected to several passes using these techniques at room temperature and at cryogenic temperature. The mechanical properties and microstructure were studied as a function of the number of passes. The Vickers hardness values increased from 44 VHN to 98 VHN. The microstructure of the copper samples reduced to 32 mm from 87 mm as the starting grain size after 3 passes. Cell sizes approximately 500 nm in size were obtained.

Creep Behavior in a Cryomilled Ultrafine-Grained Al 5083 Alloy: *Manish Chauhan*[1]; Indranil Roy[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

The creep behavior of an ultrafine-grained (UFG) Al 5083 alloy, prepared by a cryomilling with an average grain size of 300 nm, was investigated at three temperatures: 573, 623 and 673 K. The results show the presence of three creep regions: Low-stress region that exhibit a low stress exponent of less than 10, intermediate-stress region that is characterized by a high stress exponent of approximately 47, and a high-stress region that is characterized by an intermediate stress exponent of approximately 17. An analysis of the data of the low-stress region suggests the presence of a threshold stress, which strongly depends on the temperature. Possible creep mechanisms are discussed in cryomilled Al 5083 alloy and compared with those proposed for dispersion strengthened alloys.

Mechanical Behavior of a 6061 Al Alloy and Al2O3/6061 Al Composite Processed by ECAP: *Lijia Chen*[1]; Chunyan Ma[2]; *G. M. Stoica*[1]; *Peter K. Liaw*[1]; *Cheng Xu*[3]; *Terence G. Langdon*[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Rm. 427-B, Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Shenyang University of Technology, Sch. of Matls. Sci. & Engrg., 58 Xinghua S. St., Tiexi Dist. Shenyang 110023 China; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Olin Hall of Engrg. 430, Los Angeles, CA 90089-1453 USA

A commercial 6061 Al alloy and an Al2O3/6061 Al composite were subjected to 8 passes of equal-channel angular pressing (ECAP) using the strain path of route BC. The tension and stress-controlled fatigue behavior of the alloy and composite at room temperature were investigated and compared. For the commercial 6061 Al alloy, it was found that after ECAP both the tensile and fatigue strengths of the alloy decreased while the elongation to failure showed a minor reduction. However, the tensile and fatigue strengths of the Al2O3/6061 Al composite subjected to ECAP were significantly although the elongation to failure of the composite was lower than before ECAP. In addition, the fracture surfaces were observed using scanning electron microscopy to determine the fracture mode.

A Comparison of the Properties of SPD Processed AA-6061 by ECAP, Multi-Axial Compressions/Forgings (MAC/F) and Accumulative Roll Bonding (ARB): *Balakrishna Cherukuri*[1]; Teodora S. Nedkova[1]; Raghavan Srinivasan[1]; [1]Wright State University, Mechl. & Matls. Engrg. Dept., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA

Commercially available AA-6061 in the annealed condition was subject to Severe Plastic Deformation (SPD) processing by Equal Channel Angular Pressing (ECAP), Multi Axial Compression/Forgings (MAC/F) and Accumulative Roll Bonding (ARB) at room temperature to approximately the same accumulated strain (~4). Room temperature and elevated temperature tensile and compression tests were carried out to evaluate the flow behavior of the as-SPD materials. In comparison to the as received material, the SPD processed materials show high strain rate sensitivity, and hence potentially enhanced formability in the temperature range of 300C-350C. Results presented will include the as-processed microstructure, the stability of the microstructure at elevated temperatures, microhardness and the flow behavior of the various materials at different strain rates and temperatures. This work was supported by the US Department of Energy through Grant No. DE-FC36-01ID14022.

Superplastic Behavior of Coarse-Grained Aluminum Alloys: *A. R. Chezan*[1]; J. Th.M. De Hosson[1]; [1]University of Groningen and Netherlands Institute for Metals Research, Dept. of Applied Physics, Nijenborgh 4, Groningen 9747AG The Netherlands

We report on the superplastic behavior and the microstructural evolution of two coarse-grained Al alloys: Al-4.4w/oMg and Al-4.4w/oMg-0.4w/oCu. The maximum tensile elongations obtained by deformation at 450°C and at a strain rate of 10^{-2}/s were 260 and 315%, respectively. The value for the strain rate sensitivity index of 0.3 and a sharp peak stress at small plastic strain suggest that solute drag on dislocation motion is an important phenomenon in these materials. Orientation imaging microscopy (EBSP) observations show a decrease of the average grain size with increasing plastic strain. The deformed microstructure is characterized by an increased density of low angle grain boundaries and by a pronounced texture as compared to the initial grain structure. These microstructural changes trigger instabilities in the plastic flow resulting in necking followed by fracture and act as limiting factors of the superplastic performance in coarse-grained aluminum alloys.

Creep Behavior of Extruded Al-6Mg-1Sc-1Zr-10SiC Alloy: *S. P. Deshmukh*[1]; R. S. Mishra[1]; K. L. Kendig[2]; [1]University of Missouri, Dept. of Metallurgl. Engrg., 1870 Miner Cir., Rolla, MO 65409 USA;

[2]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright Patterson AFB, OH 45433 USA

Creep tests were performed on fine grained Al-6Mg-1Sc-1Zr-matrix alloy with and without SiC particulates in the temperature range of 150 to 260°C. The composite was fabricated by direct extrusion of billets made from helium atomized alloy powder and SiC particulate. The composite exhibits high value of apparent stress exponent and apparent activation energy for creep at low temperatures. The flow behavior of these composite is characterized by the presence of a threshold stress. Incorporation of threshold stress into analysis reduces the high values of apparent stress exponent and activation energy to those anticipated form the creep of solid solution alloys. Results shows increased creep resistance in composite compared to unreinforced alloy at higher temperatures.

Tensile and Compressive Creep Behaviour of Al2O3 (Saffil)-Short Fiber Reinforced Magnesium Alloy AE42: *Hajo Dieringa*[1]; Yuanding Huang[1]; Petra Maier[1]; Norbert Hort[1]; *Karl Ulrich Kainer*[1]; [1]GKSS Research Center, Ctr. for Mg. Tech., Max-Planck-Str. 1, Geesthacht 21502 Germany

Development of metal matrix composites (MMCs) is a possibility to overcome the disadvantages of poor high temperature creep proiperties of common magnesium alloys. It is well known that short fiber reinforcement improves the high temperature creep resistance of magnesium alloys. Tensile and compressive creep properties of magnesium alloy AE42 (4 wt.-% aluminum and 2 wt.-% rare earths) reinforced with 20 vol.-% alumina (Saffil)-fibres were investigated in the temperature range of 175-300°C and stress range of 40-140 MPa. The MMC was manufactured by direct squeeze casting. The materials investigated show different behaviour depending on type of loading. From the stress dependence of the minimum creep rate stress exponents were calculated in order to find indications for the possible mechanisms of deformation. The need of correcting the stress dependence of minimum creep rate by a threshold stress was found. Electron microscopy was performed to investigate the surface of fracture of a tensile creep specimen.

The Effect of Testing Temperature on Stress-Strain Relationships in Pure Aluminum Over a Wide Range of Strain: *Nguyen Quang Chinh*[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Eötvös University, Dept. of Gen. Physics, 1117 Budapest, Pázmány P. sétány 1/A., Budapest Hungary; [2]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The stress-strain relationships of high purity aluminum were investigated over a wide range of strain by combining data obtained in conventional testing of annealed samples with data obtained after processing by equal-channel angular pressing (ECAP) to high imposed strains. The results show the nature of the macroscopic stress-strain relationship changes characteristically in different temperature regions. In the low temperature region the macroscopic stress-strain behavior exhibits a monotonously increasing tendency over the entire range of strain whereas in high temperature testing the flow stress increases only to a critical strain. It is demonstrated that the stress-strain relationship in the positive strain-hardening region may be described by an exponential-power law constitutive equation which reduces to the conventional Hollomon power-law relationship at low strains and to the Voce exponential relationship at high strains. On the basis of this new equation, it is possible to define low and high temperature deformation regions.

Synthesis of Bulk Nanostructured Materials by Repeated Cold-Rolling: *Guru Prasad Dinda*[1]; Harald Roesner[1]; Gerhard Wilde[1]; [1]Forschungszentrum Karlsruhe, Inst. of Nanotech., Eggenstein-Leopoldshafen, Karlsruhe 76021 Germany

Recently, bulk nanostructured materials synthesized by severe plastic deformation (SPD) have attracted increased attention because of their improved and rather unusual mechanical properties. Repeated cold-rolling with intermediate folding represents an alternative SPD-technique to produce extremely fine nanocrystalline materials at ambient temperature. In the present work, massive nanocrystalline samples of Ti and Zr with average grain sizes below 100 nm and Ni with grain size less than 10 nm have been synthesized at ambient temperature by repeated cold-rolling. The evolution of the microstructure and texture at different stages of plastic deformation was investigated by X-ray diffraction, scanning and transmission electron microscopy. Mean grain sizes less than 10 nm in pure Ni have not been obtained by any SPD techniques. The main key to produce such fine nanocrystalline structure seems to be the requirement of very large plastic strain but not necessarily a high imposed pressure in the range of several GPa.

Finite Element Modeling of Equal Channel Angular Pressing: Effect of Material Properties, Friction and Die Geometry: Stephane Dumoulin[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Trondheim 7491 Norway

Equal channel angular pressing (ECAP) is an efficient process for obtaining ultra-fine grained materials. To be industrially applicable, it is of great importance to create a homogeneous microstructure in order to have constant material properties in the whole work-piece i.e. to have homogeneous strains during the process. However, the strain distribution developed during pressing strongly depends on material properties (yield strength, strain hardening, strain rate sensitivity), die geometry (channel angle, die corner angle) and friction. Therefore, finite element modeling of one pass ECAP at room temperature of an aluminum alloy is performed in order to investigate and characterize the effects of these parameters on strain distribution and some selected parameters of the process such as the work-piece shape in the die angle region. Simulations are performed using the commercial finite element code DEFORM. The results are validated by comparison with experimental results.

Improving the Superplastic Properties of a Two-Phase Mg-8% Li Alloy Through Processing by ECAP: *Mitsuaki Furui*[1]; Cheng Xu[2]; Tetsuo Aida[3]; Makoto Inoue[4]; Hiroshi Anada[1]; Terence G. Langdon[2]; [1]Toyama University, Dept. of Sys. Engrg. of Matls. & Life Sci., Faculty of Engrg., 3190 Gofuku, Toyama 930-8555 Japan; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [3]Toyama University, Dept. of Mechl. & Intelligent Sys. Engrg., Faculty of Engrg., Toyama 930-8555 Japan; [4]Toyama National College of Technology, Dept. of Ecomatls. Engrg., Toyama 939-8630 Japan

Grain refinement was achieved in a cast Mg-8mass% Li alloy through processing by equal-channel angular pressing (ECAP) using a die having an internal channel angle of 110° and a pressing temperature of 473 K. Following extrusion and subsequent ECAP through 2 passes, the alloy exhibited superplasticity over the temperature range from 423 to 473 K. A maximum fracture elongation of >950% was attained at 473 K with an initial strain rate of 1×10^{-4} s^{-1} and the strain rate sensitivity was of the order of 0.6. This elongation is high by comparison with other Mg alloys tested in tension at the same temperature and strain rate. The activation energy for superplastic flow in this alloy was essentially equal to the value for grain boundary diffusion of Mg. The use of ECAP gave a significant improvement in strength and ductility at room temperature.

An Investigation of Crack Growth Behaviour by Reverse Bending Cycling Under Creep-Fatigue Condition: *Nong Gao*[1]; [1]University of Southampton, Matls. Rsch. Grp., Southampton SO17 1BJ UK

An investigation of short crack growth behaviour of AISI type 316 stainless steel under creep-fatigue conditions at 550 within high strain ranges of 0.9-2.5% and 60 minutes hold time was undertaken on a high temperature reverse bending rig. Throughout the tests, surface crack initiation and growth on both tensile and compressive sides were monitored by means of a plastic replication technique. These analyses revealed that the behaviour of individual initiation and growth of many minor cracks in Stage I, and their subsequent coalescence in Stage II are the dominant characteristics for the failure of the specimens. Increasing the strain range causes an increasing number of minor cracks and a promotion of the process of minor crack coalescence. Predominantly intergranular long cracks on the tensile side and transgranular short cracks on the compressive side are the prominent feature. The different crack morphology and crack length on the two sides of a specimen indicates that a compression-only dwell is much less dangerous than a tension-only dwell.

Microstructure and Mechanical Properties of Highly Deformed Ti-6Al-4V: *Mehmet N. Gungor*[1]; Lawrence S. Kramer[1]; Ibrahim Ucok[1]; Hao Dong[1]; Nicholas R. Martin[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

Various deformation processes, including beta extrusion, alpha + beta extrusion, rotary piercing and flowforming, were utilized to produce seamless Ti-6Al-4V tubular structures. Microstructures and mechanical properties of deformed Ti-6Al-4V tubes were studied. Microstructure development in tubes was investigated utilizing metallography, texture analysis, and scanning electron microscopy techniques. While extruded and rotary pierced tubes were annealed, flow formed tubes were stress relieved prior to mechanical testing. The mechanical testing matrix included tensile and fatigue tests. In this paper, the study results for different tube manufacturing methods are presented, compared and discussed. This work was prepared by the National Center for Excellence in Metalworking Technology, operated by Concur-

rent Technologies Corporation (CTC), under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the Navy Manufacturing Technology Program.

Improvement of High-Temperature Behavior in a Mg-0.55% Zr Alloy Through Equal-Channel Angular Pressing: *Bing Q. Han*[1]; Terence G. Langdon[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing was performed on a Mg-0.55% Zr alloy at a temperature of 513 K. The grain size was refined from 75 mm to 8.6 mm by ECAP. Tensile tests were performed at temperatures of 473 to 773 K and at strain rates from 10-5 to 10-1 s-1. It was found that the tensile elongation in the as-pressed state was higher than in the as-received state. A largest elongation of 384 % was observed at a temperature of 773 K using a strain rate of 4 x 10-5 s-1. The stress exponent at all temperatures was estimated to be about 0.17. The activation energy of deformation of the pressed Mg alloy was estimated to be close to that of grain boundary diffusion while the activation energy of the as-received Mg alloy was close to that of lattice self-diffusion. The possible deformation mechanisms at elevated temperatures were discussed.

Superplasticity of a Commercial 6061 Al Composite Processed by Severe Plastic Deformation: *Bing Q. Han*[1]; Terence G. Langdon[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Severe plastic deformation of a commercial 6061 Al composite was performed through equal-channel angular pressing at a temperature of 533 K. A microstructure with ultrafine grains was present after 8 passes. Superplasticity was observed in the ultrafine-grained 6061 Al composite at a temperature of 853 K and a strain rate of 2.5×10-4 s-1 due to this grain refinement. The effects of severe plastic deformation on the microstructure and superplasticity of 6061 Al composites are discussed in the present study.

New Developments in Geometric Dynamic Recrystallization: *M. E. Kassner*[1]; [1]University of Southern California, Aeros. & Mechl. Engrg., OHE 430, Los Angeles, CA 90089-1453 USA

The concept of geometric dynamic recrystallization (GDX) originated in 1985 with work on elevated-temperature deformation pure aluminum to large strains. In this case, substantial grain refinement occurs through a process of grain elongation and thinning leading to a dramatic increase in grain boundary area. The grain boundaries become serrated as a result of subgrain (low angle) boundary formation. Pinching off and annihilation of high-angle grain boundaries occurs as the original grains thin to about twice the subgrain diameter to and a ?steady-state structure. About 1/3-1/2 the subgrain facets remaining high angle boundaries, with this geometric dynamic recyrstallization. This concept has since been carefully verified in pure Al, as well as Al-Mg alloys deforming in the 3-power regime. Large strain deformation of Al single crystals are consistent with the concept. Recent experiments on alpha zirconium are show that GDX applies to this low SFE hcp metal. Also, data in the literature on large strain deformation of a bcc iron alloy is consistent with GDX. Thus, it appears that GDX is a general phenomena that can lead to grain refinement in the absence of any discontinuous dynamic recrystallization (DRX) or continuous dynamic recrystallization (CDX).

Microstructure and Anisotropic Mechanical Properties of Pure Ti Produced by Equal Channel Angular Pressing: *Ho-Kyung Kim*[1]; Woo-Jin Kim[2]; [1]Seoul National University of Technology, Dept. of Auto. Engrg., 172 Kongnung-dong, Nowon-gu, Seoul 139-743 S. Korea; [2]Hong-Ik University, Dept. of Matls. Sci. & Engrg., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 Korea

Microstructure, texture analysis and anisotropic tensile properties of the 1, 2 pass ECAPed and unECAPed pure Ti were investigated. The unECAPed coarse microstructure became refined to finer grains after ECAP. Tensile properties in the transverse and longitudinal directions of Ti billets processed by route Bc were investigated. Work hardening was observed in all the samples. After ECAP, yield strength increased by 141, 103, 123, 129% for 1 passed transverse and longitudinal and 2 passed transverse and longitudinal direction samples, respectively. However, tensile ductility decreased by 68, 34, 37, 38%, respectively. The strength differential between the samples pulled along the transverse and longitudinal directions and its dependence on pass number is believed to be related to texture modification during ECAP. The microstructure and texture development in the Ti during ECAP process, and their relation with the strength are discussed.

Substructural and Phase Transformations During Plastic Deformation of Materials Obtained by Severe Plastic Deformation: *Nina A. Koneva*[1]; Eduard V. Kozlov[1]; [1]Tomsk State University of Architecture and Building, Dept. of Physics, Solyanaya sq. 2, Tomsk 634003 Russia

The paper summarizes the results of the studies of structural transformations that take place in UFG metals and alloys during deformation in tension or compression at room temperature. The studies of UFG Cu and its alloys are performed using TEM methods. Quantitative analysis of changes in (1) grain structure (grain size, density and types of boundaries), (2) dislocation structure (substructural transformations, scalar and excess dislocation density), (3) phase content (decomposition of the solid solution, deformational dissolution of small particles) and (4) the spectrum of stress fields from various sources is performed. The role of dislocational mass transfer, migration of grain boundaries and movement of triple junctions is emphasized. Contributions from thermally activated processes at moderate temperatures are considered.

High Strength and Ductility of UFG Metals and Alloys, Subjected to Severe Plastic Deformation: Nikolay Krasilnikov[1]; Alfred Sharafutdinov[2]; Witold Lojkowski[3]; Ruslan Z. Valiev[2]; [1]Ulyanovsk State University, 42, L. Tolstoy str., Ulyanovsk 432700 Russia; [2]Institute of Physics of Advanced Materials, UGATU, Ufa 450000 Russia; [3]Polish Academy of Sciences, High Pressure Rsch. Ctr., Warsaw Poland

The Ni and Al-based alloy 2024 after different methods of severe plastic deformation (SPD) are investigated. The equal channel angular pressing (ECAP) and cold rolling nickel, subjected also to high-pressure torsion (HPT), is characterized of large strain degree, homogeneous structure with grain size 120 nm and shows at room temperature the record strength with ultimate tensile stress (UTS) (1270 MPa), but limited plasticity (6%). The annealing at 200°C of Ni after ECAP and rolling leads to UTS about 900 MPa and ductility 12%. Al-based alloy 2024 with grain size 70 nm was obtained using HPT at room temperature. The nanostructured alloy at room temperature demonstrated record UTS above 1100 MPa, and superplastic behavior at temperature higher then 300°Ñ. The microhardness of nanostructured alloy after superplastic deformation (1.5 GPa) was more than after standard treatment of coarse-grained alloy (1.2 GPa). Opportunity of achievement in metals and alloys of combination high strength and good ductility opens perspectives of its application in industry, particularly, for mirco-systems and for details with complex geometry, obtained due to superplastic forming.

Significance of Grain Boundary Sliding in the Zn-22% Al Alloy After Processing by Equal-Channel Angular Pressing: *Praveen Kumar*[1]; Cheng Xu[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Specimens of the Zn-22% Al eutectoid alloy were processed by equal-channel angular pressing (ECAP) through a total of 8 passes at 473 K using processing route BC. Following ECAP, the grain size was measured as ~1.0 μm. Tensile specimens, having gauge lengths of 4 mm, were cut from the as-pressed billets and tested at strain rates from 10^{-2} to 10^{-1} s^{-1}. Marker lines were placed on the specimens prior to testing and these markers were used to take measurements of the grain boundary sliding offsets at an elongation of 30%. Similar sets of measurements were also taken at a total elongation of 230%. Separate sliding measurements were taken for the three types of interfaces: Zn-Zn, Zn-Al and Al-Al. This paper describes the results obtained in this investigation and estimates the contributions of grain boundary sliding to the total strain.

Bulge Forming Characteristics of AZ 31 Sheet at Elevated Temperature: *Yong-Nam Kwon*[1]; Y.-S. Lee[1]; J.-H. Lee[1]; [1]KIMM, Matls. Procg. Dept., Sangnam 66, Changwon 641-010 Korea

Magnesium alloys have a huge potential for the structural applications due to the light weight and high specific strength. Until recently, die casting has been considered as the best way to fabricate components in practice since magnesium has such a low plastic formability. For more wide application of magnesium alloys, the plastic forming needs to be developed to ensure the high productivity with the reliability of the products. In the present study, superplastic behavior of AZ31 was studied on the respect of temperature and strain rate. Blow forming used for the investigation of the forming characteristics of AZ31 alloy with uni-axial tensile test. Formability was observed to enhance greatly with temperature increment. Strain rate sensitivity became over 0.5 over 400C below the strain rate of 10-3/s. Deformation assisted grain growth occurred in the superplastic deformation condition, while grains got refined below 250C due to dynamic recrystallization.

Development of Particle Free Zone During Superplastic Deformation in 7075 Al Alloy: *Yong-Nam Kwon*[1]; Y.-S. Lee[1]; J.-H. Lee[1]; Y.-W. Chang[2]; [1]KIMM, Matls. Procg. Dept., Sangnam 66, Changwon 641-010 Korea; [2]POSTECH, CAAM, Hyoja, San 31, Pohang 790-784 Korea

Particle free zones(PFZs) were observed to occur during the high temperature creep in many alloys containing second phase particles and often considered as a direct evidence for diffusional creep. These PFZs were also reported to develop during superplastic deformation of Al alloys. Therefore PFZ formation was suggested as a clear evidence of diffusional creep for superplastic deformation. However, it is still unclear how diffusion takes a role during superplastic deformation of Al alloys. The major features of the present study are to explain on the physical mechanism of a superplastic deformation and its corresponding PFZ development. The flow curves were separated into grain boundary sliding and accommodating parts. Deformed specimens on several deformation conditions were observed to investigate the evolutions of PFZ formation. Some new insights are presented on the development of PFZ during the superplastic deformation.

Cavitation and Failure in a Fine-Grained Superplastic Inconel 718 Sheet: Yi Huang[1]; *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Olin Hall of Engrg., Rm. 430G, 3650 McClintock Ave., Los Angeles, CA 90089-1453 USA

Tensile tests were carried out at 950C over a range of strain rates to failure using the superplastic IN718 sheet. Cavity morphology observations and quantitative analysis (area fraction and average cavity size) were determined. The results are discussed including the possible mechanism of cavity development and the factors contributing to failure.

Structure and Mechanical Properties of Long-Sized Titanium Rods Processed by Severe Plastic Deformation: *Vladimir Vladimirovich Latysh*[1]; Irina P. Semenova[2]; Gulnaz Sadikova[2]; Ruslan Z. Valiev[2]; [1]Scientific Design Technical Office "Iskra", 81 Pushkina Str., Ufa, Republic Bashkortostan 450025 Russia; [2]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx Str., Ufa, Republic Bashkortostan 450000 Russia

Recently we have developed the technology consisting of equal-channel angular pressing (ECAP) and thermomechanical treatment (TMT) for fabrication of nanostructural Ti rods 6-12 mm in diameter and up to 1.5 meter long. The present work considers the homogeneity of microstructure and mechanical properties distribution along the length of CP Ti (Grade 2) rods 8 mm in diameter and up to 1 meter long, processed by the described technique. It was established that the scatter of mechanical properties along the length of a rod subjected to the treatment was within ±5%. It is shown that the processed long-sized Ti billets possess a homogeneous ultrafine-grained structure with a- grain size of 150 nm and highly homogeneous mechanical properties with yield stress value exceeding 900 MPa, which is almost two times as much as the same value of the initial annealed alloy with ductility constituting d•10.

Investigation of Precipitation and Deformation in Cryomilled Bulk Nanocrystalline Al-Mg Alloys Using Transmission Electron Microscope: *Zonghoon Lee*[1]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; [2]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Bainer Hall, Davis 95616-5294 USA

The precipitation and deformation mechanism in cryomilled bulk nanocrystalline Al-Mg alloys were investigated using analytical and high resolution transmission electron microscope. Grain refinement was achieved by cryomilling of atomized Al-Mg powders, and then cryomilled nanocrystalline powders were consolidated by hot isostatic pressing followed by extrusion to produce bulk nanocrystalline alloys, resulting in high strength. The precipitations such as aluminum nitride and oxide were not easily resolvable in microscope because those precipitations were pretty small and often not fully crystallized. Thus the grown crystalline precipitations were investigated using both analytical and high resolution transmission electron microscope in this study. The enhanced strength of Al-Mg alloy might arise from the combination of ultrafine grain size and the existence of precipitates. The oxide and nitride content is also responsible for the unusual thermal stability and resistance to grain growth. This process demonstrates a novel approach to designing and producing bulk nanocrystalline metals that exhibit exceptionally high strength and acceptable toughness.

Microstructural Influence on Low-Temperature Superplasticity of UFG Ti-6Al-4V Alloy: *Young Gun Ko*[1]; Chong Soo Lee[1]; Dong Hyuk Shin[2]; [1]Pohang University of Science and Technology (POSTECH), Dept. of Matls. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Gyeongbuk 790-784 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Gyunggi-do 425-791 S. Korea

Microstructural influence on low-temperature superplastic behavior of ultra-fine grained Ti-6Al-4V alloy fabricated by ECA pressing was investigated in this study. For this purpose, a series of tensile tests was carried out on samples with equiaxed and lamellar microstructures at temperature range of 873K - 973K and at strain rates of 10-4/s - 10-2/s. After 4 ECA pressings, lamellar microstructures exhibited higher elongation than that of equiaxed microstructures at the same temperature and strain rate, which was attributed to the finer grain sizes of alpha and beta phases resulted from fragmentation of alpha/beta lamellae. When increasing ECAP strain from 4 to 8, elongation was significantly increased, which was associated with higher potion of high-angle grain boundary and lower activation energy. Finally, deformation mechanisms of ultra-fine grained microstructure were analyzed and discussed in the context of inelastic deformation theory.

A Crystal Plasticity Finite Element Analysis of Texture Evolution in Equal Channel Angular Extrusion: *Saiyi Li*[1]; Surya R. Kalidindi[2]; Irene J. Beyerlein[3]; Mark A.M. Bourke[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS H805, Los Alamos, NM 87545 USA; [2]Drexel University, Dept. of Matls. Sci. & Engrg., Philadelphia, PA 19014 USA; [3]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA

A 3-D crystal plasticity finite element (CPFE) approach [Kalidindi et al., J. Mech. Phys. Solids, 40 (1992) 537] is applied to simulate the texture development during equal channel angular extrusion (ECAE) of metallic materials. With this approach, polycrystal plasticity constitutive equations are incorporated in a FE code. The material is represented by thousands of grains with each grain represented by one or several FEs, and each grain is assigned an orientation randomly selected from a set of orientations discretized from the initial texture. The performance of this approach is evaluated through quantitative comparison of the simulated textures with experimental results for a number of materials (aluminum, copper and interstitial-free steel) reported earlier. They are also compared to those predicted by the full constraints Taylor and viscoplastic self-consistent models. From the CPFE simulations, information about the local variations of deformation as a result of grain interactions is extracted from the deformation gradient components and discussed in relation to the differences in simulated textures compared to the other models.

Enhancing Ductility of AL6061+10wt% B4C Through ECAE Processing: *Thomas Martin Lillo*[1]; [1]Idaho National Engineering and Environmental Laboratory, Matls., MS 2218, PO Box 1625, Idaho Falls, ID 83415-2218 USA

Additions of high modulus particles to aluminum alloys offer the potential developing a high stiffness, lightweight composite. Powder metallurgy was used to create a metal matrix composite of aluminum alloy, 6061, and 10 wt% boron carbide particles. Characterization of the as-fabricated material showed the presence of agglomerates of B4C particles and some residual porosity. Evaluation of the mechanical properties showed little improvement to the elastic modulus, low tensile strength and no significant amount of ductility. The material was ECAE processed using a variety of parameters, e.g. temperature, number of passes through the die, route, intermediates anneals, etc. Subsequent microstructural characterization revealed redistribution of the B4C particles with evidence of B4C particle size reduction. ECAE processing also eliminated residual porosity and improved the elastic modulus. The biggest improvement was observed in the elongation to fracture which increased to >10%.

Cyclic Stress-Strain Response and Low-Cycle Fatigue Damage in Ultrafine Grained Copper: *Hans J. Maier*[1]; Pawel Gabor[1]; Ibrahim Karaman[2]; [1]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Pohlweg 47-49, Paderborn 33098 Germany; [2]Texas A&M University, Dept. Mech. Engrg., 326 Engrg. Bldg., Coll. Sta., TX 77843-3123 USA

We report on the fatigue behavior of ultrafine grained copper obtained by equal channel angular extrusion (ECAE). Cyclic stress-strain response and fatigue life data were determined in low-cycle fatigue (LCF) tests. The early stages of the fatigue process were examined in a scanning electron microscope equipped with a small-scale load frame that allowed for in-situ fatigue observations. The actual ECAE route employed had a substantial effect on macroscopic cyclic stress-strain response. Despite these differences, all ECAEed samples displayed deviation from Masing behavior in LCF tests, but showed nearly perfect Masing behavior in incremental step tests. As expected, fatigue life was observed to depend on the actual ECAE process conditions. The in-situ studies helped to rationalize the effect of the microstruc-

ture on LCF life data, and it is concluded that a microstructure that leads to a more tortuous crack path will decrease small crack growth rate, which in turn increases fatigue life. The effects of the actual ECAE process routes on fatigue performance will be discussed, and the ramifications of the results obtained with respect to optimization of the microstructure will be addressed.

Ultra Fine Grain Interstitial Free Steels: *Joel Malaplate*[1]; Sandrine Guérin[1]; Jean-Pierre Chevalier[1]; [1]CECM-CNRS, 15, rue Georges Urbain, Vitry Cedex 94407 France

The development of steels with high specific strength is of major interest for the automotive industry both for energy saving. A much studied method of increasing yield stress is to reduce the grain size and rely on the Hall-Petch relation. For example, reducing the grain size of an interstitial free (IF) steel from 15 to 1 micron increases the yield stress by a factor of 3 to 4. Furthermore, since the steel composition is unchanged, recyclability is maintained. Here we present results on ultrafine grain IF steels produced by severe plastic deformation using equal channel angular extrusion and controlled recrystallisation. Special attention has been paid to the recrystallisation conditions in order to yield fine grain sizes, leading to high yield stress, together with acceptable work hardening and ductility. The microstructures obtained (grain size, dislocation structures) before and after deformation, as well as after recrystallisation have been examined by electron microscopy.

Unified Terminology for Strain Induced Boundaries: *Hugh J. McQueen*[1]; Enrico Evangelista[2]; Marcello Cabibbo[2]; [1]Concordia University, Mechl. Engrg., 1455 Misonneuve W., Montreal, QC H3G 1M8 Canada; [2]University Polytechnica della Marche, Mech., Via Brecce Bianche, Ancona I-60131 Italy

The terminology of strain ε induced or altered boundaries should reflect the mechanisms that create them and the function performed; both are related to the regions they surround or separate. Dislocation glide is the primary mechanism in high σ creep and hot, warm and cold working, being less influenced by recovery (dynamic DRV, static SRV) as temperature T falls and ε rate rises. Dependent on σ, boundaries contain dislocations that are geometrically necessary for the misorientations Ψ as well as dipoles that are more dense in cold working; cell walls are rather incidental whereas higher angle block walls enclose cell clusters having different slip systems. After SRV, or DRV in hot deformation, polygonized walls consist almost entirely of regularly arrayed, low-energy, geometrically-needed dislocations; such subgrain boundaries (SGB) continually rearrange during steady-state ε in a stable, σ-defined substructure. Due to Taylor-defined multiple-slip in poly-crystals, grains divide into deformation bands slipping on different systems and rotating into different texture components. The transition boundaries between bands are permanent and increase in Ψ with strain to become indistinguishable from grain boundaries (GB). Original GB extended with εare disturbed by lattice dislocations; at high T, they migrate locally at SGB to become serrated. Equilibrium GB, possibly from high Ψ dislocation walls, are able to migrate causing recrystallization (SRX or DRX).

Severe Plastic Deformation of Copper, Aluminum and Titanium Alloys: *Anuj Mishra*[1]; Marc A. Meyers[1]; Bimal Kad[2]; Robert Asaro[2]; Franck Grignon[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0418 USA; [2]University of California, Dept. of Structural Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0085 USA

Equal Channel Angular Processing (ECAP) is being carried out with two goals: a) to understand the micro/nanostructural evolution during intense plastic deformation b) to produce Ti alloys with reduced grain size and superior mechanical properties. Mechanical test results are presented along with TEM pictures to examine structural evolution.

High Temperature Creep Behavior and Microstructure Analysis of Binary Ti-6Al Alloys with Trace Amounts of Ni: *Jun Ho Moon*[1]; S. Karthikeyan[1]; G. B. Viswanathan[1]; R. W. Hayes[2]; S. P. Fox[3]; M. J. Mills[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Metals Technology Inc., 19801 Nordhoff, Northridge, CA 91324 USA; [3]Timet Inc., America Techl. Lab., W. Lake Mead & Atlantic Ave., Henderson, NV 89015 USA

High temperature creep behavior of two binary, equiaxed Ti-6Al alloys with varying trace amounts of Ni (<3ppm and 3ppm) was studied. Uniaxial compression tests were performed over temperature range 510 to 593°C at applied stresses ranging 150 to 300MPa. Stress and temperature jump tests were performed to obtain the stress exponents and the creep activation energy. Results show that increased amounts of Ni increased the minimum creep rates at all stress levels. Stress exponent values of ~5.0 were obtained for both samples. Detailed TEM analysis of the deformation structure was performed on samples crept monotonically at 200~300MPa upto 0.2~4.5% plastic strain. Results are explained with reference to the recently reported trends associated with lattice self-diffusion in alpha-titanium in the presence of fast diffusing impurities. From the TEM analysis of the dislocation structures, a modified jogged-screw model has been developed that provides quantitative predictions of the observed creep rates.

Microstructural Evolution During Simple Heavy Warm Compression of a Low Carbon Steel: *Susarla Venkata Surya Narayana Murty*[1]; Shiro Torizuka[1]; Kotobu Nagai[1]; [1]Metallurgical Processing Group, Steel Rsch. Ctr., Natl. Inst. for Matls. Sci., 1-2-1, Sengen, Tsukuba Sci. City, Ibaraki Ken 305-0047 Japan

The microstructure development in a low carbon steel (0.15% C) during heavy warm deformation (HWD) was studied using field emission scanning electron microscopy (FESEM) and electron back scattering diffraction (EBSD). Plane strain compression tests have been conducted in the temperature range of 773-923K at strain rates of 0.01/s and 1/s and the specimens were deformed to 25% of the original thickness. Ultrafine grains were noticed when the strain attained a critical value and varied with the Zener-Hollomon parameter (Z). In order to understand the combined effect of strain rate and temperature on the strain required for the formation of ultrafine ferrite grains, the variation of Z parameter was plotted against compressive strain. The evolved microstructures were classified into three regions based on EBSD data, namely (a) elongated grains; (b) mixture of elongated grains with newly generated grains, and (c) newly generated ultrafine grains. The microstructures in these three regions were further studied by transmission electron microscopy (TEM).

Structural Superplasticity of an Al Alloys in Low Strain Rate Regime - An Internal Variable Approach: *Ji Eon Park*[1]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Matl. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea

On the superplastic deformation behavior the existing researches using the external variables such as total stress and strain have failed to explain the role of grain size and test temperature quantitatively. Recently, however, quantitative analysis for structural superplasticity has been progressed considerably with the use of the concepts of internal deformation variables. Nevertheless the effect of grain size and test temperature on flow characteristics in low strain rate region has not been elucidated precisely and several fundamental characteristics, such as the validity of threshold stresses and the role of grain size, are yet to be resolved or still in controversy in the superplasticity community. In this study, a series of load relaxation and tensile tests has been conducted to obtain the flow curves, which were consequently analyzed based on the internal variable theory for structural superplasticity, focusing especially on the low strain rate region.

Comparison of the Microstructure and Thermal Stability of an AZ31 Alloy Processed by Different Severe Plastic Deformation Processing Routes: Mohamed Eddahbi[1]; *Jorge Antonio del Valle*[1]; María Teresa Pérez-Prado[1]; Oscar Antonio Ruano[1]; [1]CENIM,CSIC, Physl. Metall., Avda. Gregorio del Amo, 8, Madrid, Madrid 28040 Spain

The aim of this work is to compare the microstructure, the texture, as well as the thermal stability of an AZ31 Mg alloy processed via two different severe plastic deformation (SPD) processing techniques, namely large strain hot rolling (LSHR) and equal channel angular pressing (ECAP). The microstructure was characterized by optical microscopy and the texture was measured both by X-ray diffraction (XRD) and electron backscatter diffraction (EBSD). The processing conditions were chosen to achieve similar strain levels using both routes. The microstructure obtained via LSHR is finer, with average grain sizes around 3 mm, but quite heterogeneous. During large strain hot rolling a well define basal texture develops. The sample processed by ECAE is comparatively more homogeneous and slightly coarser, with an average grain size of 7 mm. Simultaneously, a shear-type texture develops. It was found that the sample processed via LSHR was prone to heterogeneous grain growth and secondary recrystallization after annealing at 250°C for 15 h whereas the sample processed via ECAE underwent homogeneous grain growth under the same annealing conditions. The influence of the microstructure on the thermal stability is explored.

Severe Plastic Deformation Associated with [001] Single-Crystal W and W-Ta Alloy Ballistic Rod Penetration into Steel Targets: *C. Pizana*[1]; E. V. Esquivel[1]; L. E. Murr[1]; M. T. Baquera[1]; C. Y. Pina[1]; I. A. Anchondo[1]; L. S. Magness[2]; [1]University of Texas, Metallurgl.

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Examples of severe plastic deformation (SPD) associated with [001] oriented, single-crystal W and W-5%Ta alloy ballistic rod penetration into steel targets were observed in this study. Both zone-melt and CVD grown single-crystal rods were studied. Examination of in-target residual cross-sections of rod heads and rod erosion fragments was performed utilizing scanning and transmission electron microscopy (TEM) as well as optical metallography. Consistent with prior related studies, rod penetration (at 1.2 to 1.4 km/s) was facilitated by SPD induced solid-state flow, cleavage cracking, shear instabilities, and overlapping adiabatic shear bands characterized by dynamic recrystallization (DRX); observed by both optical metallography and TEM. The erosion of the [001] penetrators as a consequence of the solid-state flow creates an erosion tube of primarily DRX penetrator material which flows from the penetrator head and can particulate behind the penetrating rod, within the penetration channel. Some zones within the penetrator cleave and/or flow as large crystalline blocks on thin DRX zones. Microhardness evidence suggests that the penetrator head is heavily deformed by the initial shock compression and penetration, and the penetrator flows by DRX in a surface zone. For example, the initial CVD W [001] rod Vickers microhardness was 417 in contrast to average values of 550 near the embedded rod head center, and values of 450 in the surface-related flow zone. This research was supported by the U.S. Army Research Laboratory (Aberdeen Proving Ground, MD) under contract No. DATM 05-02-C-0046 (TO#6).

Developments in Nanostructured TiNi-Based Shape Memory Alloy: *Vladimir G. Pushin*[1]; Vladimir V. Stolyarov[2]; Ruslan Z. Valiev[2]; Terry C. Lowe[3]; Yuntian T. Zhu[3]; [1]Ural Division of Russian Academy of Sciences, Inst. of Metal Physics, 18 S. Kovalevskaya St., Ekaterinburg 620219 Russia; [2]Ufa State Aviation Technical University, Inst. of Advd. Matls., 12 K. Marx St., Ufa 450000 Russia; [3]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

In this paper, we present the processing, microstructure and properties of nanostructured TiNi-based shape-memory alloys synthesized by severe plastic deformation (SPD) methods, including torsion under high pressure (HPT), equal-channel angular pressing (ECAP), and complex combined deformations (SPD plus rolling or drawing). It is found that the SPD processing altered the phase transformation sequence of the alloy as well as the morphology of the martensite. Also, we found that the mechanical and shape memory properties can be enhanced by forming nanostructures in these alloys. More specifically, SPD processing renders higher reactive stress (up to 1.5 GPa) and reverse deformation (up to 10%) of shape memory, which are desired for various medical and other engineering applications. Several examples of medical and engineering applications of nanostructured shape memory TiNi-based alloys will be presented.

Equal-Channel Angular Pressing in Parallel Channels: *Georgy Iosifovich Raab*[1]; [1]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx St., Ufa, Republic Bashkortostan 450000 Russia

The efficiency increase of ultrafine-grained (UFG) structure formation in metallic materials is directly connected with the number of passes, type of the processing route as well as with the level of hydrostatic pressure in deformation zone. In this connection, the scheme of equal-channel angular pressing (ECAP) in parallel channels seems to be the most appropriate one. In this scheme, the material is two times subjected to successive shear localized deformation during one press stroke, which allows to decrease the number of processing cycles, to increase structure homogeneity as well as hydrostatic pressure in deformation zone of the 1st shear process due to thrust deformation, provided by the 2nd shear process. These factors contribute to the formation of more refined and homogeneous structure. The advantages of the processes have been studied on copper M1 subjected to ECAP in parallel channels.

Nanostructure and Thermal Stability of Two AlXSc Alloys Processed by SPD: *Hans J. Roven*[1]; Hakon A. Nesboe[1]; Borge Forbord[2]; Jens C. Werenskiold[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway; [2]SINTEF, Matls. & Chmst., Trondheim N-7491 Norway

Severe plastic deformation (SPD) has been used to create ultra-fine grains in two different AlXSc alloys, i.e. one alloy with a high diffusivity and the other with a low diffusivity ternary element. Both alloys are pressed in the same ECAP tool in two conditions (homogenized and not homogenized). Ultra fine grains are formed after ECAP in both alloys and an even finer distribution of Sc and/or ScZr disperoids are sub-imposed to the grain structure. The dispersoids are effectively locking down the grain boundaries. Also, the nano-structured grains embed some nanodomains with crystallographic twin character. The alloys are subjected to ordinary mechanical testing, superplastic tensile testing, calorimetric investigations and thermal stability annealing. Post ECAP thermal recrystallization is strongly dependent on the solution temperature for disperoids. Finally, the roles of the Sc containing dispersoids in regard to properties and nanostructures are briefly discussed.

Fracture Properties of Nanostructured Materials Processed by Severe Plastic Deformation: *Ilchat Sabirov*[1]; Vladimir V. Stolyarov[2]; Ruslan Z. Valiev[2]; Reinhard Pippan[1]; [1]Erich Schmid Institute of Materials Science, Jahnstrasse, 12, Leoben 8700 Austria; [2]Institute of Physics of Advanced Materials, K. Marx str., 12, Ufa 450000 Russia

In recent years, bulk nanostructured materials processed by methods of severe plastic deformation (SPD) have attracted the growing interest of specialists in materials science. The mechanical properties of these materials have been intensively studied. But in spite of a vast amount of publications devoted to this topic, the fracture properties of nanostructured materials processed by SPD have not been investigated so far. This work deals with the effect of SPD on the fracture behavior of materials. The object of this investigation is a Ti subjected to equal channel angular pressing (ECAP) for 2 and 8 passes. The microstructure of the material in all investigated conditions is studied. Standard mechanical tensile tests are performed to determine the tensile mechanical properties. Disk compact tension specimens are tested to determine the J-Äa curves. A great effect of ECAP on the morphology of the fracture surface is observed. The value of the crack tip opening displacement at the moment of fracture initiation (CODi) is measured by a fracture surface analysis system. The evolution of the fracture properties and fracture behavior, namely, the fracture toughness, the slope of the J-Äa curves dJ/d(Äa), the maximum extension of the stable crack propagation, etc., with the number of ECAP passes is studied and will be discussed.

Analysis of High Strength State of Ultrafine-Grained TI-6AL-4V ELI Alloy Processed by Severe Plastic Deformation: *Irina Petrovna Semenova*[1]; Yuntian Theodore Zhu[2]; Terry C. Lowe[3]; Georgy Iosifovich Raab[1]; Lilia Rashitovna Saitova[1]; Ruslan Zufarovich Valiev[1]; [1]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx St., Ufa, Republic Bashkortostan 450000 Russia; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [3]Metallicum, 1207 Callejon Arias, Santa Fe, NM 87501 USA

As we showed earlier, equal-channel angular pressing (ECAP) and further extrusion of two-phase titanium alloy Ti-6Al-4V contributes to a considerable enhancement of strength properties up to UTS [3]1400-1450 MPa as a result of grain refinement. The current work presents the detailed study of the microstructure of medical Ti-6AL-4V ELI alloy subjected to the described treatment. It was established that the morphology of a- and b-phases in the initial state before ECAP had great effect on the microstructure refinement and the alloy mechanical behavior. The influence of various structural parameters (ultrafine grains, disperse phase precipitates, supersaturated solid solution, etc.) on the alloy strengthening was analyzed. It was found that strength and fatigue properties could be enhanced by means of the control of a grain size and phase morphology of the initial structure before ECAP as well as after ECAP processing and further thermal and thermomechanical treatment.

Characteristics of Aluminum 6061-T6 Deformed to Large Strains by Machining: *M. Ravi Shankar*[1]; Srinivasan Chandrasekar[1]; W. Dale Compton[1]; Alexander H. King[2]; [1]Purdue University, Sch. of Industrial Engrg., IE, GRIS, 315 N. Grant St., W. Lafayette, IN 47907-2023 USA; [2]Purdue University, Sch. of Matls. Engrg., 315 N. Grant St., W. Lafayette, IN 47907-2023 USA

A study has been made of large strain deformation characteristics of heat-treatable aluminum alloys (6061-T6) by analyzing chips created by plane strain machining. By varying the geometry of the tool, different levels of strain were imposed in the material chip in a single pass of machining. The chip micro-hardness values were found to be somewhat greater than the hardness values reported for Al 6061-T6 in Equal Channel Angular Pressing (ECAP). Also, chips deformed to larger strains were generally harder than chips deformed to smaller values of strain. Microstructure of the chips was found to be composed of relatively equi-axed grains with mean size in the range of 80-150 nm. The microstructure and hardness were found to be relatively stable even after extended periods of annealing. Small but statistically significant differences were found between the annealing behaviors of the chips deformed to different strains, with chips deformed to larger strains being less thermally stable vis-à-vis their less deformed counterparts.

A Multi-Grain Model for Subgrain Formation and GB Sliding: *A. Simone*[1]; E. van der Giessen[1]; [1]University of Groningen, Dept. of Applied Physics, Micromech., Nyenborgh 4, Groningen 9747 AG The Netherlands

Recent investigations have reported superplastic flow at high temperature and high strain rates in various coarse-grained polycrystalline materials, including some Al-alloys. Although the precise mechanism is not yet well understood, there are many indications that it involves a combination of dislocation creep and grain boundary sliding, facilitated by plasticity-induced grain refinement and subgrain formation. To contribute to a better understanding of the main mechanisms behind superplastic flow in coarse-grained polycrystalline aggregates, we present a multi-grain finite-element model that incorporates these phenomena. The model is used here to study the effect of both grain size, shape and distribution in the initial stages of superplastic flow. Special emphasis is placed on the analysis of cell formation with subsequent cell evolution into subgrains and eventually into real grains. This leads to the breaking down of coarse grains which would allow massive superplastic flow to proceed.

Ti-6Al-4V Alloy Flange Production by Isothermal Roll Forming Process: *Youngil Son*[1]; [1]ADD, R&D Ctr., Yusung PO Box 35-5, Daejeon 305-600 S. Korea

The high cost and poor machinability of Ti alloys makes them prime candidates for net-shape manufacturing techniques. Isothermal forming of Ti alloys enables net-shape forming at comparatively lower loads. This paper will show the manufacturing process and metallurgical evaluation of thin walled Ti-6Al-4V alloy flange by isothermal roll forming process. The process of isothermal roll forming employs rollers to shape a cylindrical workpiece into a complex symmetric shape by simultaneously adjusting the roll shape and by moving the rolls radially outward on the workpiece while it is rotated about its axis of symmetry. Both the workpiece and the rolls are maintained at temperatures close to the beta transus. The evaluations of the flange included microstructure, crystallographic texture, heat treatment response, tensile strength. The flange microstructures were found to be uniform and without any strongly textured colonies. Mechanical properties of the roll formed flanges were compared with those of conventionally forged flanges.

Al-Cu-Mg-Li (Mn, Zr, Sc) Alloys for Age-Forming of Damage Tolerant Curved Structures: *Marco Jan Starink*[1]; Nong Gao[1]; Nicolas Kamp[1]; Ian Sinclair[1]; [1]University of Southampton, Matls. Rsch. Grp., Sch. of Engrg. Sci., Highfield, Southampton SO17 1BJ UK

Age forming has substantial potential cost benefits for the production of curved aluminium structures. This technique is currently applied to production of upper wing skins of commercial aircraft, but is not currently applied for lower wing skins as incumbent lower wing skin alloys lose their damage tolerant properties upon ageing. On the basis of modelling of strength and general metallurgical understanding, a series of Al-Cu-Mg-Li (Mn, Zr, Sc) alloys predicted to provide good proof strength (PS) and a good balance of damage tolerant properties are designed and manufactured through casting and rolling. Extensive experimental work has been conducted on these alloys by metallography, hardness testing, tensile testing, fatigue testing, fracture testing, DSC, SEM/EBSD, TEM and 3DAP analysis. After artificial ageing representative of age-forming several of the newly designed alloys have PS, fatigue crack growth resistance and toughness that are comparable or better than incumbent 2024-T351. Strategies for counteracting the reduction in UTS-PS are discussed.

Analysing the Shear Zone for Metals Deformed by Equal-Channel Angular Processing: *Grigoreta Mihaela Stoica*[1]; Douglas E. Fielden[1]; Robert L. McDaniels[1]; Peter K. Liaw[1]; Cheng Xu[2]; Terence G. Langdon[2]; [1]University of Tennessee, Matl. Sci. & Engrg., 323 Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The plastic deformation during the Equal-Channel-Angular Processing (ECAP) of metals concentrates in a shear zone, which is located close to the plane separating the two channels of the die. Scribed split billets were used in order to experimentally observe the flow field in the shear zone for different transient states of the billet subjected to ECAP. Spatial distributions of the strain and strain rate were evaluated using an analytical procedure developed for orthogonal cutting. The experimental results are compared with the theoretical predictions of the shear strain obtained from the slip-line models. An analysis of the shearing nonuniformity across the billet was conducted for dies having both sharp and round corners.

On the Effect of SPD on Recycled Experimental Aluminium Alloys: Nanostructures, Particle Break-Up and Properties: *Przemyslaw Szczygiel*[1]; Hans J. Roven[1]; Oddvin Reiso[2]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway; [2]Hydro Aluminium AS, R&D Matls. Tech., Sunndalsora 6600 Norway

As a part of on-going research on the effects of recycled alloy chemistries on alloy design, properties and applications, the present work focuses on SPD processing. An ECAP tool is used to process different Al alloys with “recycled alloy chemistry”. A comparison between deformation routes A and Bc is performed to evaluate the effects of deformation path on particle break-up and particle distribution. The particle break-up sequences are followed under intrinsically measured strain conditions and along different flow paths in the ECAP tool. Also, the effects of friction and deformation temperature on particle structures are studied. Phase characterization is performed by microprobe, HR-SEM, TEM and calorimetric investigations. Local deformation zones around particles are studied in order to reveal the local strain gradients in the nano-to micro scale. Finally, both dynamic presipitation during ECAP and post ECAP aging characteristics as influenced by particle structures are qualitatively described.

Superplastic Joining of Y-TZP Enhanced by Titania-Doping in the Insert Material: *Yorinobu Takigawa*[1]; Hiroaki Takadama[2]; Kenji Higashi[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Grad. Sch. of Engrg., 1-1 Gakuen-cyo, Sakai, Osaka 599-8531 Japan; [2]Japan Fine Ceramics Center, Matls. R&D Lab., 2-4-1 Mutsuno Atutaku, Nagoya, Aichi 456-8587 Japan

Superplastic joining of 3mol% Yttria stabilized tetragonal zirconia polycrystal (3Y-TZP) is examined using 5wt%TiO2-doped 3Y-TZP as an insert material, in order to decrease the joining temperature and stress and to realize the local deformation near the joining surface. The joining tests are conducted by uniaxial compression in the temperature range of 1523 to 1723K. The joined specimen is characterized by scanning electron microscopy (SEM), showing a clean interface with no cavitation. Four point bending tests are conducted to evaluate the joining strength, resulting that an average flexural strength of 960MPa is obtained at 1573K compressed by 10MPa for 30 min, which is the 80% of the strength in 3Y-TZP matrix. The temperature of 1573K is 100K lower to obtain similar strength without the insert. Fracture occurs not from joining surface but from matrix in bending teat. Residual stress must be introduced during joining process.

Distinguishing Ambient-Temperature Creep Region in Deformation Mechanism Map of Annealed CP-Ti: *Hisamune Tanaka*[1]; Tomoyasu Yamada[1]; Eiichi Sato[1]; Itaru Jimbo[2]; [1]Institute of Space and Astronautical Science/Japan Aerospace Exploration Agency, Space Struct. & Matls. , 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]Tokai University, Faculty of Engrg., 1117 Kitakaname, Hiratsuka, Kanagawa 259-1292 Japan

Ambient-temperature creep behavior of typical H.C.P., F.C.C. and B.C.C. metals and alloys of annealed state were investigated at a stress levels below their yield stresses. Metals and alloys having H.C.P. structure showed large creep deformation. Among them, CP-Ti showed significant creep deformation. Therefore, activation energy and stress exponent were measured in order to compare the deformation mechanism between ambient and high-temperature creep. The activation energy of ambient-temperature creep indicated much lower than that of high-temperature creep and the stress exponent of the former indicated much higher than that of latter. Based on those creep parameters, ambient-temperature creep region for annealed CP-Ti was distinguished in the Ashby-type deformation mechanism map.

Effects of Microstructures on the Creep Rupture Properties and Fracture Mechanisms in Austenitic Heat-Resisting Steels: *Manabu Tanaka*[1]; Ryuichi Kato[2]; Junnosuke Taguchi[2]; [1]Akita University, Faculty of Engrg & Resource Sci., Rsch. Inst. of Matls. & Resources, Dept. of Mechl. Engrg., 1-1 Tegatagakuen-cho, Akita, Akita Prefecture 010-8502 Japan; [2]Akita University, Grad. Sch., Dept. of Mechl. Engrg., 1-1 Tegatagakuen-cho, Akita, Akita Prefecture 010-8502 Japan

Effects of microstructures on the creep-rupture properties were investigated using the aged specimens of the SUS304 steel and the 21Cr-4Ni-9Mn (21-4N) steel at 973 K. Size distribution of cracks was also examined during creep. The grain size dependence of creep and creep-rupture properties (especially, creep ductility) was very different in these steels because of difference in the fracture mechanisms. Grain-boundary fracture occurred in the 21-4N steel, and a mixed mode of grain-boundary fracture and transgranular fracture was observed in the SUS304 steel, although grain-boundary sliding controlled the crack initiation at grain boundaries. Cumulative size distribution of creep cracks could be fitted to a power law at the larger crack sizes in the 21-4N steel, while the cumulative size distribution could be ap-

proximated by an exponential law in the SUS304 steel. These experimental results coincided with the results of simulation on the growth and linkage of creep cracks.

Microstructures and Mechanical Properties of Weldments from Extruded and Flowformed Ti-6Al-4V Alloy Tubes: *Ibrahim Ucok*[1]; Lawrence S. Kramer[1]; Mehmet N. Gungor[1]; Philip Wolfe[1]; Hao Dong[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation, MTEC, 100 CTC Dr., Johnstown, PA 15904 USA

The main objective of this study was to perform weldability studies on seamless Ti-6Al-4V structural tubes manufactured by deformation processes such as extrusion, rotary piercing and flowforming to compare weldment properties. The tubular materials were butt-joined by plasma arc welding. The welded tubes were then subjected to a stress relief treatment prior to extraction of specimens for microstructural examination, tensile testing and fatigue testing. The effect of tube forming methods and heat treatments on microstructure and mechanical properties of the weldments are presented, correlated and discussed. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation, under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the Navy Manufacturing Technology Program.

Nanocrystallization of a Nickel Alloy Subjected to Surface Severe Plastic Deformation: *Juan C. Villegas*[1]; Kun Dai[1]; *Leon L. Shaw*[1]; Peter K. Liaw[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., 97 N. Eagleville Rd., U-3136, Storrs, CT 06269 USA; [2]University of Tennessee, Dept. of Matls. Sci. & Engrg., Rm. 427-B Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA

Surface nanocrystallization and hardening (SNH) process is a surface severe-plastic-deformation (SPD) process that has been applied to bulk specimens of Hastelloy C2000®, a nickel-base alloy. A microstructural gradient that is a function of the processing conditions was obtained and analyzed in order to discern the mechanism responsible for the observed grain refinement. Many structural and microstructural defects, such as stacking faults, high dislocation density and deformation twinning, were found to be present in the deformed surface layer. The role of these defects in the surface nanocrystallization and its implications in strengthening are discussed.

On the Influence of Processing Temperature During Severe Plastic Deformation of Iron Based Materials: *Andreas Vorhauer*[1]; [1]Austrian Academy of Sciences, Erich Schmid Inst., Jahnstrasse 12, Leoben 8700 Austria

Armco iron, a ferritic and an austenitic steel were subjected to severe plastic deformation (SPD) in order to achieve a refinement of the initially coarse grained microstructures down to microstructural sizes less than 100nm. A specially designed High Pressure Torsion (HPT) tool provides isothermal (+/-5°C) materials processing in a range of temperature between room temperature and 450°C. The aim of this work is to analyze the influence of the processing temperature on the microstructural refinement during SPD as a function of the applied strain (maximum 32). The morphology and the microtexture of the investigated materials were analyzed in a scanning electron microscope by capturing micrographs with the detector for backscattered electrons and measuring orientation maps with the Electron Back Scattering technique, respectively. In selected cases transmission electron microscopy investigations were performed. The relation between different microstructural features (as microstructural size and microtexture) and mechanical properties obtained from micro tensile tests are discussed.

Micro-Mechanics Modelling on the Toughening of Nano-/Micro-Meter Grained Composite Microstructure: *Jing Tao Wang*[1]; Dun Yan[2]; Zhong Ze Du[2]; [1]Nanjing University of Science and Technology, Sch. of Matls. Sci. & Engrg., No.200 Xiaolingwei, Nanjing 210094 China; [2]Xi'an University of Architecture and Technology, Sch. of Metallurgl. Engrg., Xi'an 710055 China

Markedly toughening effect was observed in advanced materials with a composite microstructure of ductile phase dispersed in high strength/hardness less ductile matrix, in the macroscopically elastic stage of amorphous alloys with ductile dendritic dispersions, and in the crack propagation stage of ceramics with ductile inclusions. Similar toughening effect was observed in the plastic stage in bulk nano-crystalline materials with dispersions of micro-meter grained microstructure constituents. Modelling of this toughening effect in nano-/micrometer grained composite microstructure was carried out in this paper based on micro-mechanics. It is found through modelling that the toughening effect is dependent on microstructure parameters like ductile phase dispersion, volume fraction, dimension and aspect ratio; And the encapsulation of the ductile phase by the surrounding high strength less ductile matrix phase is found to crucial for this toughening effect.

Microstructure and Texture Evolution During ECAP of an AlMgSi Alloy: Observations, Mechanisms and Modeling: *Jens C. Werenskiold*[1]; Hans J. Roven[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway

Equal channel angular pressing has been used to create severe plastic deformation (SPD) nanostructures in a standard aluminium alloy at room temperature. Advanced characterization in a modern FEG-SEM with a state-of-the-art microdiffraction unit has been performed on samples carefully prepared from different positions in the process shear zone. Intrinsic strain measurements are done in parallel in order to describe the actual strain tensor in each position studied. This technique is used to discover actual mechanisms for grain break-up and the formation of nano-sized grains. In addition, HREM-TEM investigations reveals that deformation twins are formed and is therefore one of the operating mechanisms. The texture evolution seems to fit the believed deformation micro-to-nano mechanisms and can also be linked to specific deformation structure elements of certain crystallographic orientations. Based on the observations, a crystallographic model describing the main mechanisms for the creation of ultra-fine grains is proposed and briefly discussed.

Strain Hardening During High Pressure Torsion Deformation: *Florian Wetscher*[1]; [1]Erich Schmid Institute of Material Science, CD Lab. for Lokal Analysis of Deformation & Fracture, Jahnstraße 12, Leoben 8700 Austria

Severe Plastic Deformation (SPD) has been applied to different materials (Copper, Armco-Iron, pearlitic steels) by means of High Pressure Torsion (HPT). In this study the shear stress during deformation was measured in situ under different hydrostatic pressures. By applying a simple model for a strain hardening material, a shear stress - shear strain curve can be fitted and the influence of the hydrostatic pressure can be studied. These results are compared to microhardness measurements, tensile tests and the microstructural evolution during deformation.

Creep Deformation of Ordered Intermetallic Alloys: *K. Xia*[1]; [1]University of Melbourne, Dept. of Mechl. & Mfg. Engrg., Parkville, Victoria 3010 Australia

Many ordered intermetallic alloys possess high heat resistance and low density and are being developed for high temperature structural applications. Consequently, their creep resistance is of great significance. In this review, the general creep behaviour observed in a group of selected ordered intermetallics will be compared to that in disordered alloys. Creep deformation mechanisms will be analysed in view of the unique crystal structures, bonds, defect structures and diffusion processes. Effects of microstructure will then be discussed with a view to developing highly creep resistant intermetallic materials.

Processing of Medium Carbon Steel by Hot Pressing Prior ECAP: *Jozef Zrnik*[1]; *Jaroslav Drnek*[1]; Zbysek Novy[1]; *Libor Kraus*[2]; [1]COMTES FHT Ltd., Borska 47, Pilsen 301 00 Czech Republic; [2]COMTES FHT Ltd., Pilsen 301 00 Czech Republic

Intensive plastic deformation of a number of steel grades in conjunction with controlled thermal process yields very fine preliminary microstructure providing favourable mechanical properties. Medium carbon steel containing 0.45% carbon, prior to inserting severe plastic deformation, was repeatedly hot repeatedly press forged between flat swages. Uniform and fine dynamically recrystallized structure of ferrite-pearlite mixture with grain size of about 2 ìm resulted from this specific thermomechanical treatment. Cementite within nest-like pearlite colonies retained rod-like morphology. The total effective strain of å = 3-5 inserted to specimen was estimated by numerical simulation (FEM). In order to release accumulated stress (material strengthening) and evaluate the stability of obtained structure static annealing treatment was introduced to deformed samples. In dependence on annealing temperature and hold time the cementite colonies decomposed to form more or less spherical cementite precipitates. The further grain refinement mechanism during equal channel angular pressing following processing route B was explored at each pass of repetitive pressing. The steel was subjected to five pressings. Employment of this processing route resulted in further refinement of ferrite grains. The submicrometer order ferrite grains enclosed by serrated and low angle boundaries were formed within the former ones. Transmission electron microscopy examination revealed that these boundaries resulted from slip systems interactions. Cementite particle modified the constitution of newly born substructure.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Novel Processes I

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Wednesday AM Room: 2009
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Paul R. Dawson, Cornell University, Sibley Sch. Mech. Aeros. Engrg., Ithaca, NY 14853 USA; Paul Krajewski, General Motors, Matls. & Processes Lab., Warren, MI 48090 USA

8:30 AM Invited
Adaptation of Automotive Technologies for the Production of General Aviation Aircraft: *Mahmoud Y. Demeri*[1]; [1]FormSys Inc., 40180 Woodside Dr. S., Northville, MI 48167-3427 USA

NASA has initiated a number of substantial efforts over the last decade to impact personal transportation for the future. A number of national consortia for aviation mobility and various programs in the General Aviation (GA) arena such as the Advanced General Aviation Technologies Experiment (AGATE), the General Aviation Propulsion (GAP) and the Small Aviation Transportation System (SATS) have been developed to address the ideas of virtual highways in the skies. Success for this concept depends on the ability of the aerospace industry to produce a highly affordable personal aircraft that is comfortable, safe and easy to fly. This presentation reviews and assesses advanced automotive materials and innovative manufacturing technologies for possible adaptation to the production of affordable and lightweight fuselage for the MI-SATS program. Automotive technologies are reviewed in view of the functional requirements for autobody structures and aircraft fuselage.

8:55 AM Invited
A Methodology for Accelerating the Evaluation of the Mechanical Properties of Polyphase Alloys: *Paul R. Dawson*[1]; Matthew M. Miller[1]; [1]Cornell University, Sibley Sch. of Mechl. & Aeros. Engrg., 196 Rhodes Hall, Ithaca, NY 14853 USA

A system for more rapidly determining critical mechanical properties of polyphase engineering alloys is presented that merges simulation and selective experiments. The system centers around a statistical representation of the material structure based on observable geometric features and their attributes. An example of a feature is a grain; the orientation of the atomic lattice within a grain is an example of one of its attributes. Virtual specimens are instantiated by building polycrystals comprised of grains discretized with finite elements and then assigning attribute values from sampling the corresponding probability distributions. The specimens are tested via finite element simulations to assess their mechanical behavior. Experiments provide key information to validate the simulation tools and to quantify fundamental properties of the constituent phases. The result is a properties representation for the anisotropic strength and stiffness of a multiphase alloy as functions of the microstructural state and the mode of loading.

9:20 AM
Through Process Modelling in Manufacturing of Aluminium Structures for Automotive Applications: *Ole Runar Myhr*[1]; [1]Hydro Aluminium Structures, Product & Process Dvlp., N-2831 Raufoss Norway

Simulation of the material response throughout a multistage manufacturing route, commonly referred to as "through process modelling" (TPM), has gained considerable momentum over the past decade. This is due to an increased need for better control of the end product properties. TPM is particularly useful in fabrication of automotive parts from age-hardening aluminium alloys since the manufacturing is complex and involves several consecutive operations and heat treatments. For such alloys, the material response is intimately linked to a high number density of nano-metre size precipitates evolving in the different processing steps. A model for coupled nucleation, growth and coarsening of precipitates has been fine-tuned for Al-Mg-Si alloys and implemented into a dedicated FE-code. Examples are shown on applications of this FE-code in TPM of manufacturing of aluminium structures for the automotive industry. These examples illustrate how different end product properties predicted by the model can be manipulated and optimised.

9:40 AM
Deformation Behavior and Texture Development During the Thermomechanical Processing of Fe-15 At.% Ga Alloys Containing NbC: *Sivaraman Guruswamy*[1]; Pinai Mungsantisuk[1]; Douglas Barker[2]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [2]UEES Inc.(AFRL/MLLMP), Rms. 048/131, Area B, Bldg. 655, 2230 Tenth St., Wright Patteron AFB, OH 45433-7750 USA

Fe-Ga alloys exhibit large magnetostriction in the [001] direction, and [001] oriented single crystals or [100] textured polycrystalline forms of these alloys are therefore highly desirable for use in automotive sensors and actuators. Textured polycrystalline alloys are preferred due to lower cost and better room temperature mechanical properties. This paper examines the feasibility of an inexpensive thermomechnical processing approach involving a sequence of controlled hot rolling, two-stage warm rolling with intermediate anneal, and texture anneal to obtain [001] texture in polycrystalline (Fe-15 at.% Ga) alloys containing NbC. Roll forces during each pass of the hot and warm rolling stages were measured to examine the deformation behavior of the alloys. Textures evolution during different stages of processing was examined using orientation imaging microscopy to identify conditions that favor [001] texture development. The results indicate that an appropriate NbC content and thermomechanical process condi tions can produce FeGa alloys with strong [001] texture. Work supported by NSF-DMR Grant # 0241603.

10:05 AM
Processing and Mechanical Behavior of Lightweight Particle Reinforced Metal Matrix Composites by a Novel Sinter-Forging Technique: *Nik Chawla*[1]; Jason Williams[1]; [1]Arizona State University, Dept. of Chem. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA

In an effort to explore affordable processing for metal matrix composites, this study focuses on the characterization and mechanical behavior of an aluminum matrix composite with SiC particle reinforcement processed by a novel sinter-forging technique. In this technique, reinforcement particles and matrix alloy powders are blended, cold pressed, sintered, and hot forged. This technique has the advantage of eliminating costly extrusion and secondary finishing steps to produce a net-shaped part. The microstructure, as well as the ambient-temperature monotonic and cyclic fatigue behavior of the sinter-forged composites will be reported. The effects of SiC particle size and alloy powder size on processing and properties will be discussed. Comparisons to composites produced by conventional powder metallurgy and extrusion have also been conducted. It will be shown that, although the sinter-forged composites have a somewhat lower ductility than their extruded counterparts, the strength and fatigue resistance are comparable to those of extruded materials.

10:25 AM Break

10:40 AM
Manufacturability of TiAl Alloys for Turbocharger Applications: *Sadao Nishikiori*[1]; Satoshi Takahashi[1]; Nobuhiko Yunoki[1]; Akihiro Ohkita[1]; [1]Ishikawajima-Harima Heavy Industries, Production Engrg. Ctr., 1, Shinnakahara-cho, Isogo-ku, Yokohama 235-8501 Japan

In order for TiAl alloys to be widely employed in turbocharger, it is important to understand effects of process parameters on metallurgical features and to optimize each production process. In previous report, we introduced how TiAl alloy was applied to the product turbocharger, based on the results of material tests, FOB test and several types of reliability tests. Through our manufacturing experience of TiAl turbocharger, further alloy development will be discussed in this study. Then, we focus on various TiAl alloys. Especially, castability,

phase stability, cyclic oxidation resistance and mechanical properties of these alloys have been evaluated. In addition, with an industrial view to application, turbo charger rotor of TiAl-Mo-V-Si alloy (IR-24T) developed by IHI were cast and joined to a shaft by friction welding under the production scale. Hot spin test was also carried out. Potential of this alloy for application will be discussed.

11:00 AM
Processing Ti-Al-Nb Multi-Layered Composites from Elemental Foils Using Accumulative Roll Bonding: *Rengang Zhang*[1]; Viola L. Acoff[1]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., Tuscaloosa, AL 35487 USA

Intermetallic compounds and alloys in the form of sheet materials are ideal candidates for the manufacturing of supersonic spacecrafts. One particular intermetallic that has been receiving considerable attention for this application is the alloy Ti-46Al-9Nb (at%). In this study, accumulative roll bonding (ARB) is used to process multi-layered composites with a nominal composition of Ti-46Al-9Nb (at%) from elemental foils. The microstructures of the multi-layered composites that were subjected to different levels of rolling reduction were characterized by scanning electron microscopy (SEM). The hard Ti and Nb layers were observed to neck and break down due to the repeated mechanical deformation, and the Ti and Nb particles embedded in the soft Al matrix. X-ray diffraction patterns showed only reflection of the elements, which indicates that no detectable solid-state reactions occurred. Differential thermal analysis (DTA) was also used to characterize the effect of rolling strain on the solid-state reaction and phase formation in the composites during annealing. Tensile testing and fractography of the tensile test specimens were utilized to evaluate the mechanical properties of the processed multi-layered sheet materials.

11:20 AM
Processing of Gamma Titanium Aluminide Sheets Using Cold Rolling and Reactive Synthesis: Gajanan P. Chaudhari[1]; *Viola L. Acoff*[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., 126 7th Ave., Box 870202, Tuscaloosa, AL 35487 USA

Gamma titanium aluminide sheet is a candidate material for aerospace and automotive fields. The importance is due to its low density and superior high temperature mechanical properties. A simple process using a combination of commonly available processing techniques of rolling and heat treatment is presented. Aluminum and titanium foils are cold roll bonded and subjected to a two-stage heat treatment, resulting in gamma-titanium aluminide sheets. Hot rolling of the sheet resulted in a denser product with better mechanical properties. The process parameters and resulting microstructures are presented. Mechanical properties of the sheets are evaluated.

11:40 AM
Directional Recrystallization of High Purity Ni and Ni-V Alloys: Hui Chang[1]; *Ian Baker*[1]; [1]Dartmouth College, Thayer Sch. of Engrg., 8000 Cummings Hall, Hanover, NH 03755 USA

Directional recrystallization process has been performed on both high purity (99.995%) polycrystalline nickel and nickel containing 7 wt. % vanadium. The nickel sheets were cold rolled to thickness reductions of 90%, 95% or 98%, isothermally annealed at their primary recrystallization temperatures for 30 mins and then directionally annealed in an image furnace at 1000°C. A large temperature gradient of 100°C /mm ahead of the hot zone was used during directional annealing and a wide range of hot zone velocities were examined. The as-received, as-rolled, isothermally annealed and directionally recrystallized microstructures were characterized using both optical microscopy and electron back-scattered patterns from a FEI XL30 scanning electron microscope. The results of the different processing and the of the vanadium additions will be presented and contrasted with prior studies on the directional recrystallization of nickel with purity of 99.5% cold rolled to 90% thickness reduction. Research supported by NSF grant DMI 0217565.

Alumina and Bauxite: Bauxite and Bayer Process Red Side

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday AM Room: 2005
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Monique Authier-Martin, Alcan, Arvida R&D Ctr., Jonquière, Québec G7S 4K8 Canada

8:30 AM
Applied Mineralogy Studies: An Important Tool to Understand the Red Mud Sedimentation Process: *Aurea Gomes*[1]; Beatriz Vieira[1]; Nilce Alves Santos[1]; Carlos Alberto Alves[2]; [1]CVRD, Project Dvlp./Tech., BR 262, km 296, Santa Luzia, MG 33030-970 Brazil; [2]CVRD, Project Dvlp./Exploration, Av Governador Jose Malcher, 815/516, Belem, Para 66055-260 Brazil

In the Bayer plant, bauxite mineralogy affects efficiency by driving the chemical reactions that occur in the process. The composition and morphology of the alumina-bearing minerals, as well as those of other impurities with varying solubility in caustic soda, are critical for determining alumina extraction, product purity, caustic soda losses, and energy consumption. In order to understand the factors influencing settling rate, overflow clarity, and mud compaction, the red mud settling performance of several bauxites samples was studied under different digestion conditions. The bauxite samples and their respective red muds were submitted to chemical and mineralogical analysis.

8:55 AM
Effect of Bauxite Microstructure on Beneficiation and Processing: *Károly Solymár*[1]; Ferenc Mádai[2]; Dimitris Papanastassiou[3]; [1]EPU 3000, Engineer's Consulting Ltd., Béla király út 7/A, Budapest H-1125 Hungary; [2]University of Miskolc, Dept. of Mineralogy & Petrology, Miskolc-Egyetemváros H-3515 Hungary; [3]S&B Industrial Minerals S.A., Bauxite Div., 21 A, Amerikis Str., Athens GR-106 72 Greece

The microstructure of bauxite determines to a significant extent the opportunities for its beneficiation and optimum processing downstream. Adequate fine grinding commensurate its microstructure may result in proper mineral liberation and grain size distribution required for effective ore dressing (i.e. H/M or magnetic separation)and digestion respectively. Particle size distribution, mean diameter and amount of ooidal grains as well as degree of dissemination of the impurities in polished sections of raw bauxite, ground bauxite and red mud samples were determined by means of scanning electron-microscope, electron probe micro-analyser and digital image analysis. The results of beneficiation tests (effective removal of liberated limestone but insufficient reduction of finely disseminated reactive silica) and the required digestion parameters of the mainly oolitic Greek diasporic and the Hungarian boehmitic (partly goethitic) bauxite are discussed. Based on the microstructure the effectiveness of beneficiation, the degree of grinding (required particle size) and also the necessary digestion parameters of any bauxite can be predicted.

9:20 AM
Optimation of Bauxite Grinding by Means of Empiric Model: Barnabás Csõke[1]; *Gábor Mucsi*[1]; Károly Solymár[2]; [1]University of Miskolc, Dept. of Process Engrg., Miskolc-Egyetemváros H-3515 Hungary; [2]EPU 3000 Engineer's Consulting Ltd., Béla király út 7/A, Budapest H-1125 Hungary

The grain size distribution of the ground bauxite plays an important role in the extraction yield of alumina and digestion kinetics. The ground bauxite product of ball mills can be characterised by a function of relative size distribution, F (• =X/X50), even at different grinding capacities. The median of the ground product at a given mill depends on the grinding capacity which can be determined by direct measurements (that is X50 = f (Q) function) or can be estimated by equations of Bond-Rowland-Kjos. The expected grain size distribution under modified conditions can also be calculated by means of the above equations, so the grinding process can be optimised. The results of the calculations have been confirmed by plant-scale bauxite grinding tests. A new universal Hardgrove mill was used for fast laboratory determination of grindability of different kinds of bauxite.

9:45 AM
Industrial Test With Mono and Multifilament Cloths in Kelly Filters of CVG-Bauxilum: *Ricardo Alfredo Galarraga*[1]; Rodolfo Díaz[1]; Gisela Quintero[1]; [1]CVG-Bauxilum, Lado Rojo II, Zona Industrial Matanzas, Puerto Ordaz 8015 Venezuela

CVG Bauxilum, who is always open to new technologies, recently evaluated a new filtration fabric with a base specification different any cloth that has previously been tried. The differences are: the fabric construction, which is monofilament polypropylene in the warp direction and multifilament polypropylene in the weft direction and the extraordinarily high number of yarns in the warp direction (250+). For 115 days, the performance of this material was monitored as it was installed on several Kelly filters in the Security Filtration area. The new cloths faced all of the conditions in the plant and the following determinations were made: 1.The cloths withstood the stresses of plant operation for 150 cycles (1500 hours) with no difficulty whatsoever. 2.The number of times caustic cleaning and acid cleaning were required were greatly reduced when compared to previously tried materials. 3.Due to the physical characteristics of the material, cloth manipulation was much easier thereby improving the ease of installation. 4.The quality of the filtered liquor was acceptable. 5.The physical characteristics of the material made it much easier to manually clean than any other previously tried materials.

10:10 AM Break

10:20 AM
Rod Mill Replacement at Aluminium Oxid Stade GmbH: *Hartmut Borchers*[1]; [1]Aluminium Oxid Stade GmbH, Stade 21683 Germany

After 30 years of operation Aluminium Oxid Stade GmbH (AOS) had to replace one out of their two rod mills.The mill showed a lot of cracks at the flange section caused by repair welding and cracks at the foundation. This paper will describe basic tests before replacement, bauxite treatment before installation in order to increase milling capacity of the second old rod mill, modification of the DSM screen, mill installation, main technical differences between old and new rod mill and final grinding test.

10:45 AM
Mechanical Activation of Bauxite-Potential and Prospects in the Bayer Process: *Rakesh Kumar*[1]; T. C. Alex[1]; Z. H. Khan[1]; S. P. Mahapatra[2]; [1]National Metallurgical Laboratory, Jameshedpur, Jharkhand Pin. 831 007 India; [2]National Aluminium Company, P/1, Nayapali, Bhubaneswar, Orissa Pin. 751 013 India

Mechanical activation of bauxite is known to have a beneficial effect on the alkali leaching of gibbsite. Mechanical activation is found to be more effective, in terms of improvement in alumina recovery at lower temperature and soda concentration and minimization of soda loss in leach residue. Coupling of mechanical activation with the leaching process is an important issue in the exploitation of the beneficial effect of the activation. Two separate schemes, namely, "simultaneous milling and leaching" and " separate milling and leaching" have been explored. Both the approaches result in comparable alumina recovery. However, greater soda loss was observed in the case of latter. This is explained in terms of greater duration of leaching and its effect on the dissolution of different Al-bearing phases. The results show that the concept of simultaneous milling and leaching is superior in terms of leaching time, energy utilization and simplicity of operation.

11:10 AM
Prospects of Processing Guyana Bauxite at Sweetening Stage: *Alexander G. Suss*[1]; Anatoly A. Lapin[1]; Andrey V. Panov[1]; Brindley H. Robeson Benn[2]; [1]Russian National Aluminium & Magnesium Institute, 86, Sredny Pr., 199106, St. Petersburg Russia; [2]Guyana Geology & Mines Commission, Upper Brickdam, Georgetown Guyana

Guyana has significant reserves of bauxite (~760 million tonnes) and about 100-years history of its mining and processing – ranging from refractory to metallurgical grade bauxites and alumina. Although Guyana has lost its previous dominance on the world market, due to huge integrated industries created in, for example, Australia, Guinea, Brazil and alumina processing ended in 1983, the country is still in the world's top 10 ranking, in terms of bauxite production and reserves, and has almost no rivals in bauxite quality. This paper investigates the possibility of processing Guyana bauxite at the "sweetening" stage, allowing a refinery to efficiently process medium quality bauxites thereby increasing substantially, digestion capacity at operating alumina plants. Chemical, mineral and process features of these bauxites in comparison with similar "sweetening" bauxites were investigated. It was revealed that Guyana bauxite shows excellent alumina extraction, however, due to specific composition – shell like coarse crystals of kaolinite, secondary gibbsite, low iron-bearing minerals and high kaolinite content – under-recovery of alumina, disilication and thickening problems, and high soda consumption are possible. Additional research is being pursued to reduce these negative impacts.

Aluminum Reduction Technology: Pot Control

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday AM Room: 2003
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Thomas Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA

8:30 AM
Challenges in Mass Balance Control of Aluminum Reduction Cells: *Martin Iffert*[1]; Maria Skyllas-Kazacos[2]; Barry Welch[2]; [1]Trimet Aluminium AG, Reduction, Aluminiumallee 1, Essen 45356 Germany; [2]University of New South Wales, Ctr. for Electrochmst. & Minl. Procg., Sydney, NSW 2052 Australia

Today high performance smelters operate with current efficiencies over 94% and energy consumption below 14 kWh per kg of aluminum. The increasing economical as well as ecological pressure makes it necessary to drive the process continuously at the limit. Revamping pots and increasing current intensity are part of the daily process. While this is a challenge, it is a greater challenge for the less energy efficient technology developed before point feeders and modern magnetic compensation. Hence it is important to understand the sources of process variation and disturbances in an early phase and enable the control system to distinguish between energy versus mass balance induced variations. The introduction of superheat measurements has made it possible to identify energy imbalance quicker, but there remain spatial, temporal and material induced fluctuations. This paper, discusses the combined roles of dry scrubbing and different grades and properties of alumina. These influence HF loading of the enriched alumin a, HF emissions and change in the material balance of a pot. Thus contributing to swings in temperature, superheat and bath inventory.

8:55 AM
Diagnosing Iron Contamination in Pot Room Metal: *Stephen J. Lindsay*[1]; [1]Alcoa, Inc., Primary Metals Div., 300 N. Hall Rd., MS S-01, Alcoa, TN 37701-2516 USA

The level of iron contamination in pot room metal is often defined by the average %Fe or by the percentage of the population that is lower in iron than a certain cut-off point. In this paper the author links analysis of the shape of the Fe distribution to practical approaches to improvement and suggests benchmark levels of performance.

9:20 AM
Cell Operation Improvement Using Wireless Human-Machine Interfaces: Leonel Vicente Mota Ivo[1]; *Elias Symphronio Castro Neto*[2]; Otávio Mário Guzzon[2]; [1]Atan Automation Systems, Al. Div., Av. Afonso Pena, 4001 - 9 andar, Funcionarios, Belo Horizonte, MG 30130-008 Brazil; [2]CBA Companhia Brasileira de Alumínio, Controle de Processos, Rua Moraes do Rego, 347, Alumínio, SP 18125-000 Brazil

The improvement of cell operation is one of the most important concerns in primary aluminum production. Substantial gains in productivity, energy consumption, efficiency, working conditions and environment protection can be obtained with better operational procedures. This paper describes the use of a mobile wireless HMI (Human-Machine Interface), mounted in vehicles, cranes and other equipments, along with handhelds used by field personal, to improve the cell operation at CBA, Companhia Brasileira de Alumínio, Brazil. The operational improvements, the features implemented, the architecture of the solution and the technology used are presented. Some problems found and its solutions, along with future implementations, are discussed.

9:45 AM
Experiments on Wireless Instrumentation of Potlines: Mike Schneider[1]; Daniel Steingart[1]; James W. Evans[1]; Paul Wright[1]; *Donald Ziegler*[2]; [1]University of California, Matls. Sci. & Engrg., Berkeley, CA 94610 USA; [2]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

Hall-Héroult potlines are inefficient, consuming more than twice the electrical energy required thermodynamically. In part this may be a result of inadequate instrumentation. In a typical Hall-Héroult cell the only continuous measurements are of cell voltage and line current. The cells are only minimally outfitted with sensors, mostly due to safety concerns about wires running around potlines where voltage differences can be up to several hundred volts. There is also the difficulty of possible interference with existing hardware, and the lack of reliable, maintenance free, continuous power sources for sensors. This paper presents a tested solution to accurately measure various process parameters via wireless sensing technology, specifically Berkeley Motes running the operating system TinyOS. The investigation is a joint one between Alcoa and UC, Berkeley. Early experiments at Eastalco indicated that the motes, with a few modifications, will be able to operate reliably in industrial conditions, successfully transmitting radio packets, despite the plant's strong magnetic fields, at distances of over 100 feet. The paper describes the successful testing of wireless measurement of a cell parameter and discusses what other measurements are feasible and appropriate. Research supported by the University of California Energy Institute.

10:10 AM Break

10:25 AM
Improving Reduction Cell Metal Level Measurement and Control: *Geoffrey Paul Bearne*[1]; Daniel Whitfield[2]; [1]Comalco Research and Technical Support, PO Box 316, Thomastown, Victoria 3074 Australia; [2]New Zealand Aluminium Smelters Limited, PB 90110, Invercargill New Zealand

A project was recently undertaken at the New Zealand Aluminium Smelter to improve reduction cell metal level measurement and control. The widely used Six Sigma improvement methodology, adopted by Comalco in 2002, was applied to this problem. The capabilities of two alternative measurement techniques - direct "dipping" with a steel rod and indirect measurement from anode position, were compared. Identifying and reducing the causes of variation led to improvements in both systems. The second method, known locally as Rod Height, proved to be more suitable for control purposes. An automated control chart based strategy for determining the mass of metal to be tapped from each cell was developed and implemented in three reduction lines. Key issues and findings from this project are described and the benefits of using the Six Sigma methodology for process improvement are discussed.

10:50 AM
Bath Ratio Control Improvements at Alcoa Poços de Caldas - Brazil: Leonardo Paulino[1]; *Jean Yamamoto*[1]; Jeronimo Coelho Araujo[1]; Roberta Andreia Camilli[1]; [1]Alcoa, Smelter/Potrm., Rod. Poços de Caldas/Andradas, km 10, Poços de Caldas, Minas Gerais 37701-970 Brasil

A systemic approach to improve process control in the potrooms has been in use for several years at Alcoa Aluminio S.A. in Poços de Caldas, Brasil. One of the critical sub-processes in a smelting plant deals with chemical composition control measured as bath ratio. Bath chemistry control measured as percent of pots within a desired range improved from 78% to over 95% from 1998 to 2003. This improvement was achieved due to some key factors: 1) Implementation of a ratio control algorithm to determine fluoride and soda additions using pot age (maintenance) and ratio (corrective); 2) Streamline of the logistic of bath sampling, analyzing and corrective actions; 3) Improvement in analytical techniques from Bard to X-ray diffraction; 4) Increase sampling frequency from each 96 to each 48 hours. As a result, a current efficiency increase from 91.16% in 1999 to 91.65% in 2003 was verified. This paper will present the steps taken by the Ratio Control team to achieve 95% ratio in range.

11:15 AM
A Simple Dynamic Realtime Model for Aluminum Reduction Control System: *Vladimir Yurkov*[1]; Viktor Mann[1]; [1]RUSAL, Engrg. - Technologl. Ctr. Ltd., 37, Pogranichnikov St., Krasnoyarsk 660111 Russia

An aluminum reduction cell is a dynamic essentially nonlinear object and a space-dispersed process. To describe temperature and concentration fields by finite difference or finite element methods requires too much calculation time making its application in control controller in on-line environment conjectural. An extremely simple mathematical model of an aluminum reduction cell developed by RUSAL ETC provide real time calculations which provides sufficient adequacy to the real object. The model is not totally accurate, however the forecasts of the model yield quite reasonable results in predicting the consequences of changes in operating conditions. For example, the model predicts the changes in bath temperature with the corresponding changes in amperage and resultant change in the ledge provide. As a result, the model is sufficient for real time use in the system controlling an industrial cell line.

11:40 AM
Expert System of Electrolysis Diagnostics Using FMEA Technique: Alexander Berezin[1]; *P. V. Polyakov*[2]; O. O. Rodnov[1]; V. L. Yasinski[1]; P. D. Stont[3]; [1]RUSAL Engineering & Techonology Center, Krasnoyarsk 660011 Russia; [2]STC "Light Metals", Krasnoyarsk 660025 Russia; [3]"Mayak PKF" Ltd., Krasnoyarsk 660021 Russia

Reduction of non-productive expenditures at the cost of decreasing of total amount of sick cells is one of the actual targets. Expert diagnostics system is worked out for exposure of sick cells. Diagnostics process consists of the following procedures: -Identification of cell noises and their classification as symptoms of a sickness with the help of specially trained neuronet; -Type definition and calculation of seriousness of sickness according to expert rules with the help of fuzzy logics; -Calculation of priority number of risks PNR - according to FMEA technique (Potential Failure Mode and Effects Analysis) for each symptom, sickness, cell; -Documentation of diagnostics results using FMEA protocols. Cell parameters and SPC data (Statistic process control) are the input information for the expert system of diagnostics. Diagnostics system is able to define PNR of 20 types of potroom sickness, which allows: -to reduce the number of sick cells; -to define the maximum permissible condition of the cell for overhaul.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Nanostructured Composites

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Wednesday AM Room: 3018
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Jud Ready, Georgia Tech, GTRI-EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Allentown, PA 18109 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany

8:30 AM Opening Remarks

8:35 AM Invited
Processing of Carbon Nano-Tube and its Composites at High Magnetic Fields: *Hamid Garmestani*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr. NW, Atlanta, GA 30332-0245 USA

Processing of materials under the influence of high magnetic fields has been reported to cause significant enhancements in useful properties of many materials. The mechanisms of influence of magnetic field on texture development, grain growth, and recrystallization need to be understood in order to optimize the processes and to develop commercial applications of in-field heat treatment processes. Canbon nanotubes have been produced using a variety of processing techniques and their use as reinforcements in nano-tube composites have been hindered due to the lack of proper alignment. Using high fields both nanotubes and their composites have been processed using CVD techniques. The results show that although the nano-tubes themselves have very low magnetic susceptibility but with the proper selection of the resin matrix materials a certain level of alignment is achieved. It was also discovered that the high field can adversely affect the structure of the nano-tubes and their morphology.

9:05 AM Cancelled
Fabrication of Nylon-6/Carbon Nanotube Composites

9:35 AM Break

10:00 AM
Carbon Nanotube Nanocomposites and Hybrid Materials for Multifunctional Applications: *Fernand D.S. Marquis*[1]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA

This paper discusses the full integration of carbon nanotubes in polymeric, ceramic, and other materials such as fluids, for the applications as multifunctional components with optimal structural, electrical and thermal properties. The goals are to mimic on a larger scale the properties of carbon nanotubes so that they can be used more aggressively on the micro and macro scales. The idea of mimicking carbon nanotubes involves integration by tip attachment and/or sidewall functionalization, coincident polymerization, and high shear alignment in polymer based composites. These composites are different from the conventional ones, which consist of two distinct phases: matrix and reinforcing phase. Carbon nanotube composites are hybrid materials, which are designed and manufactured in order to expand out the properties of carbon nanotubes by translating their properties to each other. The integration of the carbon nanotubes into these matrices is accomplished through the architecture of special bonds, designed to foster enhanced mechanical, electrical and thermal properties, through dispersion and alignment of the carbon nanotubes. This multifaceted design provides for innovative approaches in order to develop carbon nanotube composites and hybrids materials withn optimized performance.

10:30 AM
Effect of Fabrication Method on the Electrical Properties of ABS/CB Composites: *Sidhartha Gupta*[1]; Runqing Ou[1]; Rosario A. Gerhardt[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Acrylonitrile Butadiene Styrene (ABS) is a polymer, which is used in structural applications due to its excellent mechanical properties. Carbon Black is often added as a filler to increase its conductivity. In this study, ABS/CB composites were prepared using two different methods. The first is based on dissolution of ABS in Butan-2-one, and the second on mechanical mixing of the precursor materials. These fabrication methods led to different microstructures, which had vastly different electrical properties. The microstructures were acquired using SEM, AFM and optical microscopy while the electrical conductivity was obtained using impedance spectroscopy. The percolation threshold of the composites fabricated using mechanical mixing was found to be much lower than that of the composites fabricated using the solution method. The carbon black used had 24 nm average particle size and DBPA branching of 63 ml/100gm. This study is critical for the development of composites with more controlled properties.

Arsenic Metallurgy: Fundamentals & Applications: Process Metallurgy

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Wednesday AM Room: 2014
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Corby G. Anderson, CAMP - Montana Tech, Butte, MT 59701 USA; Bill Drinkard, Drinkard Metalox, Charlotte, NC 28205 USA

8:30 AM
The Treatment of Arsenic Bearing Ores, Concentrates and Materials with Alkaline Sulfide Hydrometallurgy: *Corby G. Anderson*[1]; [1]CAMP - Montana Tech, 1300 W. Park St., Rm. 221 ELC Bldg., Butte, MT 59701 USA

Throughout the world, there are many orebodies or materials which have significant value but also contain arsenic. As regulations on the transport, exposure, disposition and emission of arsenic have become more stringent, it has become increasingly more difficult to derive the values from these resources. This paper will outline the fundamentals of alkaline sulfide hydrometallurgy and its successful application to arsenic bearing materials.

8:55 AM
The Simultaneous Oxidation of Sulfide Minerals and the Dissolution of Gold: *Michael J. Nicol*[1]; Suchun Zhang[1]; [1]Murdoch University, Parker Ctr., South St., Murdoch, Western Australia 6150 Australia

The alkaline oxidation of refractory gold concentrates containing arsenopyrite and pyrite at ambient temperatures and pressures has been found to be accompanied by the simultaneous dissolution of gold without the addition of cyanide. A detailed study has been made of the kinetics of the oxidation of the pure minerals and of several concentrates. The reaction products of the oxidation of arsenic and sulphur have been monitored using chromatographic techniques. In the case of arsenopyrite, thiosulfate, monothioarsenate, arsenate and sulfite are the principal products while thiosulfate has been confirmed to be the dominant product of pyrite oxidation. Gold, either as added powder or as a constituent of the concentrates has been found to dissolve simultaneously during the alkaline oxidation of both arsenopyrite and pyrite, and this appears to be associated with the formation of thiosulfate of gold.

9:20 AM
Arsenic - The Technological Motivator for the Chelopech Copper/Gold Mine: *Gavin Beer*[1]; [1]Chelopech Mining EAD, Chelopech Village, Sofia Dist. 2087 Bulgaria

The Chelopech Copper/Gold Mine, located in west-central Bulgaria, has been in operation since 1959 producing a low-grade copper/gold concentrate containing up to six percent arsenic by weight. The penalties and restrictions imposed on smelting this concentrate, have seen the past operators of the mine strive to find a balance between achieving acceptable metal returns from the smelters and installing novel or unproven technologies in the face of changing political and economic climates. This paper documents the colourful history of the concentrate processing alternatives that have been investigated and implemented over the 45 year life of the Chelopech Mine, culminating in the high pressure oxidation option that is now the focus of a Definitive Feasibility Study (DFS) presently being undertaken by GRD Minproc Limited. The fundamental chemistry of the preceding alternatives is outlined followed by a discussion as to why each was ultimately not employed. A detailed description of the technology proposed in the DFS is given including: pressure oxidation, arsenic fixation, SX/EW copper recovery, CIL gold recovery and AVR/SART cyanide/copper recovery processes.

9:45 AM Break

10:00 AM
Gold Recovery from Arsenical Ores and Wastes: *Bill Drinkard*[1]; [1]Drinkard Metalox, Inc., 2226 N. Davidson St., Charlotte, NC 28205 USA

Commercial and demonstrated arsenic separation technologies for stabilization or sale from precious metal and copper ores and some major gold bearing arsenical wastes are presented. Extracted arsenic from ores and wastes has been both stabilized and sold. To date more than 100,000 tons of arsenic (as As2O3) has been profitably converted by our processes into commercial products. Even larger quantities have been safely stabilized. In this presentation, focus will be on new technologies with high gold recoveries, even from multiple refractory ores, and on secure low-cost stabilization.

10:25 AM
Sulfidization of Arsenopyrite: *Vladimir A. Luganov*[1]; Corby G. Anderson[2]; [1]The K. Satpaev Kazak National Technical University, 22 Satpaev Str., Almaty 480013 Kazakhstan; [2]CAMP - Montana Tech, 1300 W. Park St., Rm. 221 ELC Bldg., Butte, MT 59701 USA

The possibility of sulfidization of arsenopyrite with elemental and pyrite sulfur with transfer of arsenic into sublimate forms under the temperature of sulfide roasting is established by thermodynamic analysis. Kinetic investigations allowed to establish that in the process of decomposition the specific surface area and pyrite and arsenopyrite porosity changes with maximum in dependence of process duration. The highest composition velocity of arsenopyrite in the air atmosphere is achieved at 720°C, and pyrite - at 690°C, in the argon atmosphere at 730 and 690°C correspondingly. Addition pyrite to arsenopyrite charge decreases the temperature of decomposition - the highest velocity the process achieves at 580°C, which is connected with formation easily volatile arsenic sulfides. For study arsenopyrite sulfidization process with pyrite in the continuous mode were carried out researches on arsenopyrite roasting in the inert gas media in the stable bed with the consecutive feeding of pyrite into the charge. Initial arsenopyrite contained, %: 34, 8 - iron, 20, 5 - sulfur and 44, 3 - arsenic with the size 80% - 74 μm and presented in itself dense particles of small porosity with the developed surface. Mono-mineral pyrite contained 46, 1% of iron and 50, 3% of sulfur. There was established that the content of arsenic in the cinder under roasting according to the scheme with consecutive pyrite feeding.

Automotive Alloys 2005: Session II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Wednesday AM Room: 2006
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Lexington, KY 40511 USA; Tony Zhai, University of Kentucky, Dept. of Chemls. & Matls. Engrg., Lexington, KY 40506 USA

8:30 AM
Electromagnetic Welding of 6061 Aluminum Alloy: Sergey F. Golovashchenko[1]; Vladimir V. Dmitriev[1]; *Al R. Krause*[1]; [1]Ford, Mfg. & Processes, 2101 Village Rd., Dearborn, MI 48124 USA

The process of pulsed electromagnetic welding is similar to explosive welding in terms of the mechanism of metallurgical bonding The blanks are positioned at an angle to one another, and an initial clearance. The coil is positioned near the surface of the blank being accelerated. The coil is connected to a pulsed current generator. Once the capacitors are charged to a designated voltage, the switch connects the battery of capacitors to the coil. During the discharge of the capacitors, a high intensity electromagnetic field is generated in the clearance between the coil and the blank. This electromagnetic field induces an Eddy current in the blank. The interaction between the current in the coil and the induced current generates a high-intensity repulsive force between the coil and the blank. As a result, the latter is accelerated toward the fixed blank. During their impact, a cumulative jet in the contact area is formed, cleaning the surfaces of both blanks and producing a metallurgical bond. The method of p ulsed electromagnetic welding can be employed for the joining of tubes with different thicknesses using radial compression or expansion. Experiments verified the potential of using electromagnetic welding technology to weld 6061-T6 tubes using external mandrels manufactured from similar materials.

8:55 AM
Formability Analysis of Aluminum Welded Blanks: *Susan E. Hartfield-Wunsch*[1]; Nicholas J. Christoff[1]; Sherri F. McCleary[2]; [1]General Motors, Metal Fabricating Div., 2000 Centerpoint Pkwy., MC 483-520-266, Pontiac, MI 48341 USA; [2]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 45069 USA

A study was undertaken to better understand the forming characteristics of aluminum welded blanks. Laser welding, friction stir welding, and variable polarity plasma arc welding were compared to determine the best process for aluminum welded blank fabrication. Blanks were fabricated using AA5754 and AA5182 alloys, with several thickness ratios. Limited Dome Height (LDH) testing was conducted to understand the influence of the weld on formability. Dome heights and forming limit diagrams were used as measures of formability. Weld microstuctures were assessed to determine the relationship between weld geometry and formability. Results from this study indicate that the variable polarity plasma arc welds (VPPAW) offer potential performance enhancements relative to the laser welds or friction stir welds (FSW) when welding 5XXX aluminum alloys.

9:20 AM
Multi-Scale Mechanical Behavior of Open Cell Aluminum Foams: *Jikou Zhou*[1]; Z. Gao[2]; S. Allameh[1]; Alberto M. Cutino[2]; E. Akpan[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Dept. of MAE & PRISM, E-quad, Olden St., Princeton, NJ 08540 USA; [2]Rutgers University, Dept. of Mechl. & Aeros. Engrg., Piscataway, NJ 08854 USA

We will present the results of studies on mechanical behavior of open cell aluminum foams. The multi-scale nature of compressive deformation is examined from individual struts to overall foam deformation. Stress-strain curves of individual struts are investigated using micro-tensile testing. The localization (slip bands) in individual struts is discussed along with evidence of deformation bands at the macroscale. Onset and propagation of deformation localization bands are elucidated via in situ imaging and digital image correlation (DIC) techniques that provide continuous mapping of strain fields across sample sections. A simple unit cell model is then used to estimate the dependence of foam strength and stiffness on relative density and strut properties. Moreover, foam fatigue behavior under cyclic behavior is also studied, and attributed to surface crack nucleation and propagation in individual struts at different fatigue stages.

9:45 AM
Modeling of Precipitation Hardening in Pre-Aged 6000 Series Al Alloys: *Shahrzad Esmaeili*[1]; David J. Lloyd[2]; [1]University of Waterloo, Dept. of Mechl. Engrg., 200 Univ. Ave. W., Waterloo, Ontario N2L 3G1 Canada; [2]Alcan International Limited, Kingston R&D Ctr., PO Box 8400, Kingston, Ontario K7L 5L9 Canada

The 6000 series Al alloys are increasingly used for automotive skin panels for their weight savings and enhanced properties. The paint bake response of these alloys has been significantly improved in recent years by application of pre-aging processes immediately after solution heat treatment. Further optimization of the aging processes and full utilization of the strengthening potential of the commercial sheet requires that the precipitation hardening behavior of the pre-aged alloys to be predictable. Hence, a new physically-based model has been developed to determine the kinetics of precipitation and the evolution of yield strength during artificial aging of pre-aged 6000 series aluminum alloys. The model has been validated with independent experimental results. In this work, the basics of the model development, as well as some modeling and experimental results are presented.

10:10 AM
Aluminum-Ceramic (Flyash) MMCs for Automotive Applications: Graham Withers[2]; *Derek O. Northwood*[1]; [1]University of Windsor, MAME Dept., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Cyco Tech, 40 Renowden St., Cheltenham, Victoria 3192 Australia

An aluminum-based MMC (metal matrix composite) containing flyash (Ultalite) has been developed for automotive applications which typically involve the replacement of ferrous materials to reduce weight. The flyash particles are hollow microspheres, 20-50 microns in diameter, with a density about 25% of that of aluminum. Ultalite can be processed by all common casting methods. A semi-solid thixotropic casting process (high pressure die casting) has been developed which provides greatly improved casting properties compared to conventional high pressure die casting. Apart from being 1/3 the weight of cast iron and a low cost material, Ultalite does not require special tooling for machining (as is common for MMCs) and has good wear resistance. Potential applications include brake drums and discs, pistons, cylinder heads, cylinder blocks and transmission components.

10:35 AM
A Dislocation-Model of Forming Limit Diagram of Aluminum Sheet with Two Ideal Orientations: *Xiyu Wen*[1]; *Tony Zhai*[1]; Zhong Li[2]; Subodh Das[1]; [1]CAT, College of Engrg., UK, 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]Commonwealth Aluminum Concast, 1505 Bull Lea Rd., Lexington, KY 40511 USA

In this study, the forming limit diagrams (FLD) of a rate-sensitive FCC sheet with combinations of two different ideal orientations, and orientations are predicted. Unlike the M-K model, no surface groove is assumed on the sheet. On the other hand, due to the effect of segregation in the thermal-mechanical processing of the sheet, narrow bands of grains with similar orientations are assumed to exist. The band will behave like a single orientation and will deform differently from its neighbors. The result of the simulation was compared with two typical FLDs of the O-temper and H-temper aluminum alloy sheets.

11:00 AM Cancelled
Age-Hardening and Plastic Anisotropy in Extruded AA6xxx and AA7xxx Profiles

11:25 AM
Influence of Cu on the Aging Behavior of AlSi7Mg0.5 Alloy: *Yanjun Li*[1]; Stig Brusethaug[2]; [1]University of Oslo, Ctr. of Matls. Sci. & Nanotech., Dept. of Physics, PB 1126, Blindern, Oslo 0318 Norway; [2]Hydro Aluminium, R&D Ctr., 6601 Sunndalsøra Norway

The effect of Cu addition in the range of 0~3 wt.% on the age hardening behavior of AlSi7Mg0.5 alloy during T6 heat treatment has been studied. The evolution of the tensile strength and elongation of the alloys at the peak-aged T6 condition with Cu content has been measured. The tensile strength increases while the elongation decreases with increasing Cu content in the alloy. The alloy with addition of 3.0 wt.% shows the best combination of tensile strength and elongation properties. The investigation on the precipitation behavior of dispersoids during ageing by TEM and HRTEM shows that the precipitation of ϑ-Al2Cu phase caused by the addition of Cu has a strong influence on the aging behavior of the alloy.

11:50 AM
The 4xxx Aluminium Alloys Sheets Use to Deep Drawing: *Marzena Lech-Grega*[1]; Andrzej Klyszewski[1]; Tomasz Stuczynski[1]; Janusz

Zelechowski[1]; Wojciech Szymanski[1]; [1]Institute of Non-Ferrous Metals, Light Metals Div., Pilsudskiego 17, Skawina 32-050 Poland

Recycling of aluminum alloys enforces the manufactures to unify the used alloy types. Hypo eutectic silumins, which have a broad use in automotive industry for cast elements, can be used, in a form of sheets, for deep drew elements. This paper shows mechanical properties (strength, anisotropy) and sheet structure of 4xxx series alloys with addition of transitional metals, appropriate for deep drawing. Results are compared with properties of widely-used AlMg4,5 (EN AW-5082) alloy sheets.

12:10 PM
Effects of Casting Conditions on Quality of Twin-Roll Cast Al-Mg-Si Alloy Strip: *Hiroki Esaki*[1]; Yoshio Watanabe[1]; Hideyuki Uto[1]; Kazuhisa Shibue[1]; [1]Sumitomo Light Metal Ind., Ltd., R&D Ctr., 1-12 Chitose 3, Minato-ku, Nagoya 455-8670 Japan

As for the aluminum alloy 6016, it is the typical Al-Mg-Si alloy which is used as the one for automotive body applications. The AA6016 alloy strips produced experimentally by twin-roll casting (TRC) technology were investigated concerning the surface feature and the microstructure. Both the defects such as the ripple mark and the centre line segregation were clarified to influence the surface quality of the final cold rolled sheet. For example, the ripple mark is detrimental to filform corrosion resistance. Because of that, the optimum casting conditions such as casting speed and set-back distance were clarified to lower the defects. Moreover the Al-Si-Mg alloys containing ferrous impurity from 0.15 to 1% were estimated in order to develop the material recycling technology for automotive body applications by using TRC.

Beta Titanium Alloys of the 00's: Corrosion and Biomedical

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Wednesday AM Room: Salon 10/11
February 16, 2005 Location: San Francisco Marriott

Session Chairs: Sreeramamurthy Ankem, University of Maryland, Dept. of Matls. Sci. & Engrg., College Park, MD 20742-2115 USA; Yoji Kosaka, TIMET, Henderson Techl. Lab., Henderson, NV 89009 USA

8:30 AM
Fundamental Corrosion Characterization of Several Newer Generation Beta Titanium Alloys: *James S. Grauman*[1]; [1]TIMET, Henderson Techl. Lab., PO Box 2128, Henderson, NV 89009 USA

Addition of alloying elements to titanium can serve to increase or decrease the inherent corrosion behavior of the underlying titanium base metal matrix. By comparing the corrosion behavior of titanium alloys with commercially pure titanium, interesting observations can be drawn regarding the individual and multi-element effects provided by these alloys. Established beta alloys such as Ti-13-11-3, Ti-38644, Ti-15-3-3-3, and Ti-21S have been fairly well characterized in this regard with respect to basic corrosion behavior, including general and crevice corrosion, and SCC in acid chloride environments. This paper will address a similar corrosion characterization study on several newer generation beta titanium alloys. The alloys involved include TIMETAL LCB (Ti-6.8Mo - 4.5Fe - 1.5Al), TIMETAL 555 (Ti-5.5Al - 5Mo - 5V - 3Cr) , along with other recently developed beta alloys. These alloys are finding interest and application within the automotive and aerospace industries. A direct comparison of their corrosion behavior to other alloys should be of interest to design engineers.

9:00 AM
Corrosion Resistance of Beta Titanium Alloys to Hot Skydrol: *Robert D. Briggs*[1]; Katie J. Schumacher[1]; [1]Boeing Company, Boeing Commercial Airplanes/Matls. & Process Tech., PO Box 3707, MS73-44, Seattle, WA 98027 USA

Skydrol (hydraulic fluid) can cause severe corrosion of titanium alloys at elevated temperature resulting in restrictions on the use of titanium parts in applications subject to elevated temperature and possible skydrol impingement. Several beta titanium alloys were tested for their corrosion resistance to hot skydrol by an immersion testing technique. The corrosion resistance of the beta titanium alloys to hot skydrol will be compared to that of well known alpha-beta alloys.

9:25 AM
Investigation of Ti-Base Alloys with Positron Annihilation Techniques: *Falko Baier*[1]; Wolfgang Sprengel[2]; Jürgen Eckert[1]; [1]TU Darmstadt, Physl. Metall., Petersenstrasse 23, Darmstadt 64287 Germany; [2]Stuttgart University, Inst. of Theoretical & Applied Physics, Pfaffenwaldring 57, Stuttgart 70550 Germany

The research and development of Ti-based alloys for biomedical applications is focused on the development of low rigidity beta-phase Ti alloys composed of non-toxic elements with good mechanical properties and workability. A major drawback of these alloys is their poor workability. In order to overcome these limitations lots of work has been spent in order to find new Ti-alloys by varying the compositions or modifying the existing ones for the improvement of the formability. We present measurements of the positron lifetime and coincident Doppler broadening of the positron-electron annihilation photon line in as-cast and deformed Ti-base alloys in order to show the advantages of this non-destructive method for the field of engineering applications, because the sensitivity of positrons to defects in plastically deformed metals has been well-known since the 1960s. The addition of the element-sensitive Doppler broadening technique gives access to the chemical nature of the dislocation environment.

9:50 AM Break

10:05 AM
Pseudoelasticity in Beta Titanium Alloys with Nitrogen Addition: *Tadashi Furuhara*[1]; Satoshi Annaka[1]; Tadashi Maki[1]; [1]Kyoto University, Dept. Matl. Sci. Engrg., Yoshida-honmachi, Sakyo-ku, Kyoto 606-8501 Japan

A representative metastable beta titanium alloy, Ti-10V-2Fe-3Al (Ti-10-2-3), is known to exhibit good shape memory effect due to stress-induced alpha'' martensite transformation at room temperature. In the present study, nitrogen was added to this alloy to suppress slip deformation by solid solution strengthening of beta matrix and effect on the deformation behavior of beta phase was studied. During quenching after beta solutionizing, thermally-induced martensite is formed in the base alloy (Ti-10-2-3) and the alloy containing 0.1mass%N. When a bending test (of which maximum strain given is 2.5%) is conducted at room temperature, those alloys were deformed by stress-induced martensite transformation and exhibit good shape recovery in heating after deformation. In contrast, the thermally-induced martensite transformation is completely suppressed in the alloy containing 0.2mass%N and the strain provided by the bending test at room temperature is fully recovered in unloading. The forward and reverse stress-induced martensite transformation is observed by in-situ optical microscopy, confirming the occurrence of pseudoelasticity in the 0.2mass%N alloy.

10:30 AM
Phase Stability Dependence of the Plastic Deformation Behavior in Ti-Nb-Ta-Zr-O Alloys: *Junghwan Hwang*[1]; Shigeru Kuramoto[1]; Tadahiko Furuta[1]; Kazuaki Nishino[1]; Takashi Saito[1]; [1]Toyota Central R&D Labs.,Inc., Metallic Matls. Lab., 41-1, Yokomichi, Nagakute, Aichi 480-1192 Japan

We investigated the effects of alloy contents on mechanical properties to make clear a correlation between plastic deformation behavior and β phase stability in Ti-Nb-Ta-Zr-O alloys. It was realized that there was specific compositional area in which the alloy exhibited little work hardening and minimum value of Young's modulus. The specific area was expressed by "bond order" (Bo) based on the DV-X_α method of 2.87 and averaged electron/atom ratio (e/a) of 4.24, which corresponded to those of multi-functional β titanium alloy, "Gum Metal". These electronic conditions also minimized ideal strength required for plastic deformation without any dislocation activity. Actually, the deformation behavior of alloys in the specific compositional area revealed that the unique behavior characterized by "giant fault". We also confirmed that such compositional area corresponded to the phase boundary between α" martensite and β phase at room temperature.

10:55 AM
The Effect of Second Phase on the Deformation Mechanisms of Beta Titanium Alloys: Allan Wayne Jaworski[1]; *Sreeramamurthy Ankem*[1]; [1]University of Maryland, Dept. of Matls. Sci. & Engrg., 090 Stadium Dr., Rm. 1105, College Park, MD 20742-2115 USA

Recent studies have shown that grain size and stability of beta alloys affects ambient temperature tensile and creep deformation

mechanisms. In the case of a single phase beta alloy, Ti-14.8wt%V, the tensile and creep deformation mechanisms were found to be predominantly slip or predominantly twinning, depending on the grain size. However, when the same beta phase is present in an alpha-beta alloy the deformation mechanism in the beta phase is found to be predominantly stress induced martensite. The details of the investigation and the reasons for the second-phase induced deformation mechanisms will be presented. This work is being funded by the National Science Foundation under grant number DMR-0102320.

11:20 AM
Characterization of Ti-15Mo for Orthopaedic Applications: *Brian Marquardt*[1]; [1]Zimmer, Metals Rsch., PO Box 708, Warsaw, IN 46581-0708 USA

Cold hearth melting techniques, with the capability of producing large homogeneous ingots, have generated renewed interest in the Ti-15Mo beta titanium alloy. This alloy was originally developed several decades ago for chemical industry applications but was recently standardized for surgical implant applications in ASTM F 2066. Current processing routes have limited the ASTM standard to one basic microstructural condition which can be produced by beta solution treating and rapidly quenching the material to avoid the formation of alpha and/or omega phases. The equiaxed beta microstructure produced by this process has high ductility and moderate strength. Alternative processing routes and multiphase microstructures have been the subject of recent investigations at Zimmer. A vast range of material conditions and associated mechanical properties can be produced by modifying the rolling, forging, annealing and aging parameters. The purpose of this work was to characterize the influence of some of these processing parameters on microstructure/property relationships and to evaluate Ti-15Mo for applications with high fatigue strength requirements. Tensile and rotating beam fatigue data for an array of processing conditions will be presented along with metallographic results to illustrate the breadth of potential for this beta titanium alloy.

11:45 AM
Evaluation of Ti-Cr-Cu Alloys for Dental Applications: Marie Koike[2]; Masayuki Itoh[3]; Osamu Okuno[4]; Kohei Kimura[3]; Osamu Takeda[5]; Toru H. Okabe[5]; *Toru Okabe*[1]; [1]Baylor College of Dentistry, TX A&M Univ. Sys. Health Sci. Ctr., Biomatls. Sci., 3302 Gaston Ave., Dallas, TX 75246 USA; [2]Nagasaki University Graduate School of Biomedical Sciences, Removable Prosthodontics & Mgmt. of Oral Function, 1-7-1 Sakamoto, Nagasaki 852-8588 Japan; [3]Tohoku University Graduate School of Dentistry, Prosthodontics, 4-1 Seiryo-machi, Aoba-ku, Sendai 980-8575 Japan; [4]Tohoku University Graduate School of Dentistry, Dental Biomatls., 4-1 Seiryo-machi, Aoba-ku, Sendai 980-8575 Japan; [5]University of Tokyo, Inst. of Industl. Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan

To evaluate the suitability of Ti-Cr(7-19mass%)-Cu(3-7%) for dental prostheses, this study examined the characteristics of the cast alloys, including their mechanical properties, grindability (ease of grinding), wear resistance, and corrosion behavior. The grindability of the cast alloys was examined using a SiC wheel at a fixed force and time at a rotational (circumferential) speed of 1250 m/min. Wear was evaluated in a two-body wear testing machine after 50,000 cycles of simulated chewing under water spray at a fixed load. The reduced plastic deformation of the 13% Cr beta titanium alloy resulting from the inclusion of the Ti-Cu eutectoid structure when alloying with Cu significantly improved the grindability and wear resistance. Although the corrosion resistance of the alloys in artificial saliva deteriorated somewhat over the electrode potential range tested, the alloys behaved similar to many other titanium alloys within the normal intraoral oxidation potential. Supported by NIH/NIDCR grant DE11787.

Biological Materials Science and Engineering: Biological Materials Characterization and Biomimetics I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday AM Room: 3009
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Roger Narayan, Georgia Institute of Technology, Dept. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA; Eduard Arzt, University of Stuttgart, Max Planck Inst. Fuer Metallforschung, Stuttgart 70569 Germany

8:30 AM Invited
Tapping the High-Value Secrets Revealed in Biomineralized Nanocomposites: *Daniel E. Morse*[1]; [1]University of California, Inst. for Coop. Biotech., California NanoSystems Inst. & the Matls. Rsch. Lab., Santa Barbara, CA 93106 USA

Biological systems precisely control low-temperature nanofabrication of 3-dimensional mineral-organic composites by mechanisms fundamentally different from those of current technologies. We are working to identify the underlying molecular mechanisms responsible for biological nanofabrication in calcium- and silica-based systems, with the aim of incorporating these principles in new routes to synthesis for advanced technological applications. In sponges, silica nanofabrication is catalyzed and structurally directed by a unique family of enzymes. Higher-order structural control is directed by genetically controlled branching of the catalytic templating proteins operating in conjunction with molding by the surrounding lipid membranes. Biomimetic adaptation of these mechanisms to non-biological materials is now driving industrial research. Biomimetics catalyze and template the structurally controlled synthesis of non-biological semiconductors including titanium dioxide, gallium oxide, ruthenium oxide, cobalt oxide and zinc oxide. These results suggest the feasibility of a low-temperature catalytic route to the synthesis and nanostructural control of metal oxide semiconductors in a biologically inspired process analogous to current molecular organic chemical vapor deposition (MOCVD) technology.

9:00 AM Invited
A New Class of Tough Composite Materials Based on Natural Rigid Biological Systems: *George Mayer*[1]; Bryan J. Gruner[2]; [1]University of Washington, Dept. of Matls. Sci. & Engrg., Seattle, WA USA; [2]Advanced Development Program, Ft. Worth, TX USA

We have studied the structures and properties of a new class of composite materials, containing a predominantly high (95% volume fraction) ceramic or glass phase, combined with minor protein phases, which have unusual combinations of mechanical properties, such as stiffness, strength, and toughness. These composites are based on the architecture of the nacre structure, which is found in the shell of the abalone Haliotis Rufescens. The mechanisms underlying these properties have also been described. Analogs (utilizing high-performance engineering materials), that mimic many of the mechanisms underlying those superior combinations of properties, have been built. The results of the foregoing investigations are presented.

9:30 AM Keynote
Biological and Artificial Attachment Devices: Lessons for Materials Scientists from Flies and Geckos: *Eduard Arzt*[1]; [1]University of Stuttgart, Max Planck Inst. fuer Metallforschung, Heisenbergstr. 3, Stuttgart 70569 Germany

Mechanical performance governs the usefulness of many man-made devices. In biology, mechanics is often essential for survival: this is true on the molecular level, on the cellular level and for whole organisms. This keynote talk will decribe an interdisciplinary study involving materials scientists, biologists, and physicist aimed at elucidating the correlation between structure and performance of attachment devices in insects (flies, beetles), spiders, and geckos. In all of these cases, adhesion is mediated by the interaction of finely-struc-

tured contact elements with the different substrates. We study the structure of these elements on the micro and nano level by different microscopy techniques including SEM, TEM, AFM and X-ray imaging. Local mechanical properties and adhesion forces are measured by nanomechanical test methods and compared with predictions based on theoretical contact mechanics. For example, it has been possible for the first time to measure the adhesion of single gecko spatulae, with dimensions of 200 nm, to selected substrates by atomic force microscopy. Structure, size and shape of the contact elements are found to play important roles; in particular the principle of "contact splitting" has been identified: finer contact elements (down to sub-micron level) produce larger contact forces in heavier animals. The actual dimensions of the contact elements follow exactly the theoretical predictions, a relationship that covers 6 orders of magnitude in animal mass from the fruit fly to the gecko! From our findings, important conclusions can be drawn on the optimal design of artificial contact elements. The talk will present first prototype adhesive surfaces produced with this insight and identify their technical limits by introducing "adhesion mechanism maps". These developments have led to the design of artificial micro-attachment systems ("biomimicry") which are potentially useful in micro-technology.

10:15 AM Break

10:30 AM Invited
Modeling the Role of Nanostructure and Molecular Structure on the Mechanical Response in Nacre: *Kalpana S. Katti*[1]; Dinesh R. Katti[1]; Shashindra Man Pradhan[1]; Pijush Ghosh[1]; Devendra Verma[1]; [1]North Dakota State University, Civil Engrg., CIE 201, Fargo, ND 58105 USA

Nacre, the inner layer of molluscan seashells has mechanical properties that far exceed the properties of its constituents: aragonitic calcium carbonate and organics. The high fracture toughness and strength of nacre is often attributed to the unique nanoarchitecture that consists of laminated structure with about 0.5 micron sized aragonite platelets separated by about 20 nm thick organic layers. Several details of the nanostructure have been recently revealed in literature such as presence of nanoscale mineral bridges through organic layers, nanoscale asperities at organic-inorganic interfaces and rotations between layers. We have constructed three dimensional finite element models that incorporate these details of nanostructure and quantitatively evaluate the role of such details on mechanical response of nacre. Also, we examine the molecular nature of the interfaces and role of molecular structure of the proteins on mechanical response of nacre through molecular dynamics simulations and fourier transform infrared spectroscopy experiments.

11:00 AM
Mechanical Properties and Structure of Haliotis Rufescens, Strombus Gigas, and Tridacna Gigas Sea Shells: A Comparative Study: *Albert Lin*[1]; Kenneth S. Vecchio[1]; Marc A. Meyers[1]; [1]University of California, Dept. of MAE, Matls. Sci. Grp., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

Shells are composed of aragonite/calcite crystals interleaved with layers of a viscoelastic protein, having dense, tailored structures that impart (relatively speaking) excellent mechanical properties. Shells, such as conch (Strombus Gigas), giant clams (Tridacna Gigas), and Red Abalone (Haliotis Rufescens), have hierarchical architectures that differ from one shell type to another depending on the growth requirements and shell formation used by the particular mollusk. Mechanical test have been carried out on these shells for a comparison of strength with respect to microstructural architecture, sample orientation, hydration level, etc. The mechanical response is found to vary significantly from specimen to specimen and requires the application of Weibull statistics in order to be quantitatively evaluated. The complex micro-laminate structure of these bio-composite materials is characterized and related to both their mechanical properties and growth requirements. The Red Abalone (Haliotis Rufescens) is determined to have the highest mechanical strength in comparison to conch (Strombus Gigas) and giant clam (Tridacna Gigas) shells. This is attributed to an optimization of microstructural architecture in the form of 2-D laminates. The growth mechanisms and structure of abalone is observed through close examination of laboratory-grown flat pearl samples and cross sectional slices of nacreous shell allowing a detailed description of the micro-laminate structure.

11:20 AM
Quantitative Evaluation of Proteins Adsorbed on Polysaccharide Biomimetic Coatings: *Michela Ombelli*[1]; Lauren Costello[2]; Quing Cheng Meng[1]; Russell J. Composto[2]; David M. Eckmann[1]; [1]University of Pennsylvania, Dept. of Anesthesia, 3620 Hamilton Walk, Philadelphia, PA 19104 USA; [2]University of Pennsylvania, Dept. of Matls. Sci. & Engrg., 3231 Walnut St., Philadelphia, PA 19104 USA

Polysaccharide surface coatings hold considerable promise to render implantable medical devices biocompatible. Sugar based biomimetic layers are effective at preventing biofouling, yet a systematic study of the relationship between synthetic surface coating properties and plasma protein adsorption is still lacking. We have developed methods to synthesize and analyze dextran and hyaluronic acid coatings on silicon wafers. We can predict and control thickness, wettability, brushiness and grafting density of thin dextran and hyalurnoic acid layers. We have conducted experiments to quantify competitive protein adsorption (e.g., albumin, fibrinogen) using a novel approach to remove all adsorbed proteins. The method involves use of opportune detergent solutions and subsequent analysis by high performance liquid chromatography to separate and quantify each single protein. Different coating behaviors are correlated to specific coating properties and are used to optimize the synthesis route. Supported by NIH Grants R01 HL60230 and R01 HL67986.

11:40 AM
Preparation and Characterization of Chitosan/Poly(Acrylic Acid) Nanoparticles: Jian-Wen Wang[1]; *Wei-Jen Shih*[2]; Min-Hsiung Hon[2]; [1]Chung Hwa College of Medical Technology, Environml. & Safety Engrg., No. 69, Wen-Hwa Rd., Tainan 701 Taiwan; [2]National Cheng-Kung University, Matls. Sci. & Engrg., No.1, Ta-Hsueh Rd., Tainan 701 Taiwan

Chitosan based nanoparticles, which has a small particle size below 100nm have been developed in this project. Chitosan (CS)/poly(acrylic acid) (PAA) complex nanoparticles with four kinds of solvent have been prepared by a modified dropping method of PAA in chitosan solution. The physicochemical properties of nanoparticles were investigated by using FTIR-ATR, X-ray diffraction, dynamic light scattering, transmission electron microscopy and zeta potential. Chitosan dissolved in acetic acid and lactic acid has the smallest size of CS-PAA nanoparticle about 30nm. It is found that the prepared CS-PAA nanoparticles with a volume ratio of 1 to 1 and incubated in pH 3 PBS buffer solution for 24hr carry a positive charge and show the size in the range from 10 to 30 nm. Different dropping sequence can obtain various surface structures and zeta potentials of CS-PAA nanoparticles and the key factor for controlling the size and structure of the nanoparticles is the incubation pH media.

Bulk Metallic Glasses: Corrosion, Oxidation, and Phase Transformation

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Wednesday AM Room: 3006
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Jurgen Eckert, Technische Universitat Darmstadt, Darmstadt D-64287 Germany; Ralf Busch, Oregon State University, Mechl. Engrg., Corvallis, OR 97331 USA

8:30 AM
Intermediate-Range Order of Ni-Based Ternary Amorphous Metals: *Michelle L. Tokarz*[1]; John C. Bilello[1]; [1]University of Michigan, Ctr. for Nanomatls. Sci., Matls. Sci. & Engrg. Dept., 3061 HH Dow Bldg., Ann Arbor, MI 48109-2136 USA

Traditional X-ray experiments determined Radial Distribution Functions (RDFs) of NixNbySn100-x-y bulk amorphous alloys found a divergence from a random hard-sphere model with respect to neighbor shell distances/coordination numbers. However, these functions do not provide information about the specific contributions of any individual element. Solving the Partial Pair Distribution Functions (PPDFs) by varying the composition, or by chemical substitution, assumes similar behavior in different chemical environments, which may not be the case. Anomalous x-ray scattering provides better way to probe the local interactions of specific chemical pairs. Data near and far from the absorption edges of individual elements gives Differential Distribution Functions (DDFs), revealing the atomic arrangements. High-resolution synchrotron anomalous scattering experiments have indicated Ni-based clustering effects. This non-random distribution of atomic species may partially explain the failure of the random model. An

analysis is given in terms of the short and intermediate range order of this series. Research supported by DARPA under contract number: DAAD19-01-1-0525 via a subcontract from California Institute of Technology, and by DOE for use of the synchrotron facilities the Stanford Synchrotron Radiation Laboratories.

8:50 AM

Electrochemical Studies of a $Ti_{43.3}$ $Be_{27.5}$ $Zr_{21.7}$ $Ni_{7.5}$ Bulk Metallic Glass in a Phosphate-Buffered Saline Electrolyte: *Mark L. Morrison*[1]; Raymond A. Buchanan[1]; Atakan Peker[2]; Peter K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty, Knoxville, TN 37996-2200 USA; [2]Liquidmetal Technologies, 25800 Commercentre Dr., Ste. 100, Lake Forest, CA 92630 USA

Cyclic-anodic-polarization tests were conducted on a Ti-based bulk metallic glass (BMG) with a chemical composition of $Ti_{43.3}$ $Be_{27.5}$ $Zr_{21.7}$ $Ni_{7.5}$ (at.%), commonly known as LM-010. Tests were performed at 37°C in a phosphate-buffered saline (PBS) electrolyte with a physiologically-relevant dissolved oxygen content. The alloy demonstrated passive behavior at the open-circuit potential with a low, mean corrosion penetration rate of 1 μm/year. However, a susceptibility to localized corrosion was observed in all of the tests. The mean difference between the pitting potentials and the open-circuit potentials was high [785 mV]. Furthermore, the mean difference between the protection potentials and the open-circuit corrosion potentials was determined to be high as well [528 mV]. The high, positive values for these parameters signify a high resistance to localized corrosion in the tested environment. Finally, these values are the highest reported in the literature for a BMG alloy in this particular environment. The authors are grateful to the National Science Foundations, Integrative Graduate Education and Research Training (IGERT) program under grant number DGE-9989548, with Dr. L. Clesceri, Dr. W. Jennings, Dr. L. Goldberg, and Ms. M. Poats as the contract monitors, and the Division of Materials Science and Engineering, Department of Energy under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory (ORNL) operated by UT-Battelle, LLC.

9:10 AM

Variation in Cyclic-Anodic-Polarization Behavior as a Function of Bulk-Metallic-Glass Sample Size: *Brandice A. Green*[1]; William H. Peter[1]; Raymond A. Buchanan[1]; Peter K. Liaw[1]; C. T. Liu[2]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

Cyclic-anodic-polarization experiments were performed on $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ (at. %) bulk-metallic-glass specimens, which were obtained from ingots with diameters of 2 and 4 mm. The electrolyte for these experiments was an aerated 0.6 M NaCl solution, simulated seawater, with a pH of 7.00 ± 0.1. Although samples from both the 2 and 4 mm ingots consistently demonstrated passive behavior at the open-circuit potential and susceptibility to localized corrosion at increased potentials, both exhibited considerable variations in the values of the corrosion parameters (the open-circuit potential, pitting potential, and corrosion penetration rate). The samples from the 2 mm ingot proved more resistant to localized corrosion than samples from the 4 mm ingot. Examination of the microstructure and chemical distribution was performed before and after cyclic-anodic polarization experiments to explain (1) variations in corrosion parameters within samples from ingots with the same diameter and (2) differences between samples from ingots of varying diameters. The present work is supported by a National Science Foundation (NSF) Graduate Research Fellowship (B.A. Green); an Integrative Graduate Education and Research Training (IGERT) Program (DGE-9987548) with Drs. L. Clesceri, W. Jennings, and L. Goldberg as the program managers; and the Tennessee Advanced Materials Laboratory (TAML) with Dr. W. Plummer as the director.

9:30 AM

Corrosion of Zr-Based Bulk Metallic Glasses: *Isabella Gallino*[1]; Daniela Zander[2]; Beate Heisterkamp[2]; [1]Oregon State University, Dept. of Mechl. Engrg., Corvallis, OR 97331 USA; [2]University of Dortmund, Dept. of Biocheml. & Cheml. Engrg., Dortmund D-44221 Germany

Zr-based bulk metallic glasses are of increasing interest due to their excellent properties, e.g., the high elastic limit or good corrosion resistance. In general, the amorphous structure is known to improve the corrosion resistance of an alloy. A reason for such a behaviour might be the homogeneity of the metallic glass. Recent results on corrosion of amorphous and crystalline Vit106a and Zr-Cu-Al-Y are presented in comparison to the corrosion behaviour of glassy Zr-Cu-Ni-Al investigated by Gebert et al.[1] The corrosion was studied by potentiodynamic polarization methods at room temperature in 0.001-0.1 M $NaCl_{aq}$ (about pH 8) and in 0.1 Na_2SO_4 adjusted with H_2SO_4 (pH 2-8). The oxide and passivation layers were investigated by X-ray diffraction, SEM and TEM. [1]A. Gebert et al. Material and Corrosion 48 (1997) 293-297.

9:50 AM

Oxidation of $Ni_{60}Nb_{35}Sn_5$ Bulk Metallic Glasses: *Isabella Gallino*[1]; Ralf Busch[1]; Lioba Jastrow[2]; Uwe Köster[2]; [1]Oregon State University, Dept. Mechl. Engrg., 204 Rogers Hall, Corvallis, OR 97331 USA; [2]Dortmund University, Dept. Biocheml. & Cheml. Engrg., Emil-Figge Str. 66, Dortmund D-44221 Germany

Due to the high glass transition temperature (Tg > 881K) and large undercooled region, Ni-Nb-Sn bulk metallic glasses are considered a promising refractory alloy system. In addition, however, adequate oxidation resistance is necessary. The oxidation of $Ni_{60}Nb_{35}Sn_5$ bulk metallic glasses was analyzed in dry air in the temperature range close to the glass transition in order to avoid any crystallization. Thermogravimetric analyses reveal information about the oxidation kinetics. X-ray diffraction and cross-sectional electron microscopy allows phase identification in the scales as well as to reveal the oxidation mechanism. Results will be compared with the oxidation reaction of other metallic glasses as well as crystalline alloys of similar composition.

10:10 AM Break

10:30 AM

Oxidation Reactions of Zr-Based Metallic Glasses: *Uwe Koster*[1]; Lioba Jastrow[1]; [1]University of Dortmund, Dept. Biochem. & Chem. Eng., Dortmund D-44221 Germany

Zr-based metallic glassses are of increasing interest due to their excellent properties, e.g. high elastic limit or catalytic activity, appropriate oxidation behaviour provided. The aim of this paper is a detailed investigation by thermogravimetry and cross sectional microscopy on the oxidation reactions of different Zr-based metallic glasses. Oxidation kinetics seems to be controlled by oxygen diffusion through the scale towards the ZrO_2/glass interface. Two modes of oxidation were observed: depending on the alloy either (1) relative slow growth of continuous scales consisting of nodules of tetragonal ZrO_2 embedding nanocrystals of the late transition metals or (2) formation of fast growing oxide cones exhibiting a lamellar structure of monoclinic and tetragonal ZrO_2 thus allowing very fast diffusion along the interfaces between the lamellae. In order to understand the influence of structure, the oxidation behavior of these metallic glasses was compared with that of nanocrystalline or coarse crystalline alloys of similar composition.

10:50 AM

Air Oxidation and Phase Transformation of Ternary Cu-Zr-Ti Metallic Glasses: *Hsin-Hsin Diane Hsien*[1]; Wu Kai[1]; Yu-Lung Lin[2]; Rong-Tan Huang[3]; [1]National Taiwan Ocean University, Inst. of Matls. Engrg., 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Chung-Shan Institute of Science and Technology, Matls. & Electro-Optics Rsch. Div., Taoyan Taiwan; [3]National Tsing Hua University, Dept. of Engrg. & Sys. Sci., 101, Sect. 2, Kuang Fu Rd., Hsinchu 300 Taiwan

Oxidation and phase transformation of the $Cu_{60}Zr_{25}Ti_{15}$ metallic glass was studied over the temperature range 350-450°C in dry air. The oxidation kinetics, as measured by thermogravimetry, generally followed the parabolic rate law, and the oxidation rates increased with increasing temperature. The scales formed on the alloy consisted of mainly Cu_4O_3 and minor amounts of CuO and Cu_2O. The substrate remained an amorphous structure below 400°C, while slightly crystalline characteristic was detected by XRD at 425°C after long time oxidation, indicating that phase transformation has taken place at higher temperatures. The crystalline phases of $Cu_{51}Zr_{14}$ and Cu_4Ti were observed at 450°C.

11:10 AM

Resonant X-Ray Scattering Studies of Structural Order in Pd-Ni-P: *Stephan O. Hruszkewycz*[1]; Todd C. Hufnagel[1]; Sean Brennan[2]; [1]Johns Hopkins University, Matls. Sci. & Engrg. Dept., 3400 N. Charles St., 102 Maryland Hall, Baltimore, MD 21218 USA; [2]Stanford Linear Accelerator Center, 2575 Sand Hill Rd., MS 69, Menlo Park, CA 94025 USA

Recent advances in x-ray scattering instrumentation make possible highly detailed studies of short- and medium-range order in bulk metallic glasses. We have measured the resonant x-ray scattering at the Pd K absorption edge in Pd-Ni-P to determine the local atomic environment around Pd atoms in both as-cast specimens, and specimens relaxed by annealing below the glass transition temperature. In the nearest-neighbor atomic shell, structural relaxation occurs by an increase in the average coordination number (consistent with increased den-

sity), and, interestingly, an increase in the average atomic separation. There is also evidence of enhanced structural order in the second-nearest neighbor shell. We interpret these results in light of recent cluster-based structural models of metallic glasses and a relaxation of free volume regions between clusters.

11:30 AM
Consolidation of Hafnium-Based Amorphous Metal and Composites by ECAE: *Suveen N. Mathaudhu*[1]; K. Ted Hartwig[1]; Ibrahim Karaman[1]; Lazslo J. Kecskes[2]; [1]Texas A&M University, Mechl. Engrg., 3123 TAMU, Coll. Sta., TX 77843-3123 USA; [2]U.S. Army Research Laboratory, Aberdeen Proving Ground, Aberdeen, MD 21005-5069 USA

Warm equal channel angular extrusion (ECAE) in 90° tooling is used to consolidate Hf-based amorphous metal powder (Hf71.3Cu16.2Ni7.6Ti2.2Al2.6 -wt%) and to fabricate an amorphous metal matrix composite (AMMC) when blended and consolidated with pure Cu, Ni and W at temperatures between Tg and Tx for the glassy metal phase. A fully dense and uniformly consolidated product is expected after only one extrusion with good infiltration of the amorphous Hf-based alloy in between the crystalline particles. Hardness, DSC and other experimental results are reported.

11:50 AM
Structural Evolution and Stability of Bulk Zr-Based In-Situ Composites: *Ki Buem Kim*[1]; Jayanta Das[1]; Jürgen Eckert[1]; Sanat K. Roy[2]; Wolfgang Löser[3]; [1]Technische Universität Darmstadt, FB 11 Matl. und Geowissenschaften, FG Physikalische Metallkunde, Petersenstraße 23, Darmstadt D-64287 Germany; [2]Indian Institute of Technology, Dept. of Metallurgl. & Matls. Engrg., Kharagpur 721302 India; [3]Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden, Postfach 270016, D-01171 Dresden Germany

Bulk scale Zr-based in-situ nanostructured/metallic glass matrix composites have been developed with an addition of Nb as a primary â-Zr stabilizer. According to variation of experimental conditions and alloy compositions, the corresponding microstructures of the Zr-based in-situ composites are changed significantly with different mechanical properties suggesting that the optimum microstructures of these alloys are able to help improve the mechanical properties. In this study in order to optimize the microstructure of the Zr-based in-situ composites the microstructural evolution of the alloys during heat treatment were investigated by X-ray diffraction and transmission electron microscopy. Moreover, the stability of the microstructures also was investigated by differential scanning calorimetry.

Carbon Technology: Anode Baking

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland

Wednesday AM Room: 2007
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Theo Mueller, Innovatherm Schweiz AG, CH- 5400 Baden Switzerland

8:30 AM
Enhancement in Baking Furnace Operation: *Masood Talib Alali*[1]; Javed Akhtar Raja[1]; Saleh Ahmed Rabba[1]; [1]Dubai Aluminium Company, Reduction Matl./Anode Plant, PO Box 3627, Dubai 3627 UAE

Dubai Aluminum Company (DUBAL), since its inception in 1979 with production capacity of 135,000 MT per annum has grown to one of the largest single site smelters in the world with production capacity of 683,000 MT by the end of year 2004. The latest expansion which is currently underway will further enhance the capacity to 759,000MT. In order to meet continual growing anode demand, anode baking furnace operations was upgraded and optimized in majority of the operational stages to coincide with the increased aluminum smelting capacity. This article explains the methodology adopted to increase the production by 10% along with improvement in product quality. It also explains the innovative work done to improve the 7% of the product produced during furnace crossover. While enhancing productivity, refractory condition which has a significant impact on unit cost was maintained at a benchmark level reaching 170 fire cycle in one of the furnaces. This article also covers the techniques/approaches adopted to maintain the baked rejection level which is one of the key performance indicators below 0.5% level, along with some other practices to increase the kiln life.

8:55 AM
A New Method to Start Up Fires at Baking Furnaces: *Paulo Miotto*[1]; [1]Consorcio Aluminio do Maranhão, Smelter, Rod. BR 135 Km 18 - Pedrinhas, Electrode Dept., São Luis, MA 65095-604 Brazil

This paper presents a new practice of restarting the fires at pre-baked anodes baking furnaces developed by Consorcio de Aluminio do Maranhão (ALUMAR). This method reduces safety risks and environmental impacts significantly with a lower operational cost in comparison to the conventional approach.The conventional method is based on the use of special burners to provide the needed energy to start the fire. In this case, the control of the process parameters is manual and demand additional care. The cost associated with the conventional method is very high. The new technique consists in using the energy lost in the cooling zone of another fire to restart the process. Basically, the method divides the primary fire in two small parts and then, step by step, creates two new complete fires. The cost associated reduces by around 30% when compared to the conventional method. The results were measured and reported in terms of environmental impact, oil consumption and safety risk analysis.

9:20 AM
Effect of Baking Furnace Fire Pre-Heating Configuration on Environmental Characteristics: *Paulo Miotto*[1]; [1]Consorcio de Alumínio do Maranhão, Smelter, BR 135, km 18, Electrode Dept., São Luís, MA 65095-604 Brazil

This paper shows the experience of Consórcio Alumínio do Maranhão (ALUMAR) in the accomplishment of trials to define the best fire configuration and their impacts on the baked anode properties and environmental results. Pre-heating time effect on the opacity results was studied as a function of stack base temperature and the complete burnt of volatiles. Determination of an adequate configuration of pre-heating zone at baking furnaces was extremely important to guarantee anode quality and minimum environmental impact. Results from this trial show a good correlation between averaged opacity and pre-heating time. Root cause found for this correlation is the incapacity of burning the volatiles completely and the fume profile at the stack plum in specific conditions.

9:45 AM
The Application of Optical Pyrometers in Open Ring-Type Anode Baking Furnaces: *Reinhard Max Heilgendorff*[1]; Cleber Miralha Carneiro[1]; Augusto Giovanni Trindade[1]; [1]ALBRAS Aluminio Brasileiro SA, Carbon Plant Automation, Estrada Pa 483, Km21, Vila Murucupi, Barcarena, Para 68447-000 Brazil

Albras has five open ring-type furnaces with a total of eleven fires, with three burner bridges each. Since startup in 1985, type "S" thermocouples with silicon carbide protection tubes were used for flue temperature measurement and control. With the objective for reducing maintenance time and costs, an extensive test of various combinations of thermocouple and protection tubes types and optical pyrometers was made. The optical pyrometers were found to be a viable alternative. At present two fires are controlled with two color pyrometers withought protection tubes. This paper compares the use of different types of thermocouples, single color pyrometers, with and with ought protection tubes and two color pyrometers. It also shows the advantages of measuring flue wall temperatures as to flue gas temperatures.

10:10 AM Break

10:25 AM
Advanced 3D Modeling for Anode Baking Furnaces: *Dagoberto S. Severo*[1]; Vanderlei Gusberti[1]; Elton C.V. Pinto[1]; [1]PCE Engenharia S/S Ltda, Rua Felix da Cunha, 322, Porto Alegre, RS 90570.000 Brazil

The quality of the anode used in the aluminum industry depends strongly on the baking process. In general, it is desirable to achieve a more uniform temperature inside the anode during the heating process, combined with lower soaking time and energy consumption. The main objective of this study is to present a 3D fully coupled computational model able to take into account a large number of phenomena and parameters that play a role in the baking process, such as coupled fluid flow and transient heat transfer, burning of volatiles, fuel combustion using the Eddy Dissipation model, radiation and control system representation. The simulations were done using the commercial CFD code CFX 5.6. This model can be used as a powerful tool in the development of new furnaces and retrofit of the existent ones.

10:50 AM
Components Analysis of Volatile Released During Carbonization of Coal Tar Binder Pitch: *Kwangeui Yoon*[1]; Dong Jun Lee[1]; Jae Young Jo[1]; Se In Yang[1]; [1]DC Chemical Ltd., R&D Ctr., Carbon Team, 587-102, Hakik-Dong, Nam-Gu, Incheon 402-772 Korea

Coal tar binder pitch is a complex mixture of aromatic compounds. Components having low boiling point in pitch or decomposed compounds at high temperature are released out during the carbonization of binder pitch. It is the source of environmental problem related to binder pitch. We identified the volatile components released at 350C and at 650C carbonization of commercial pitch. Pitch itself and green anode were carbonized and the condensed material from them was analyzed by gas chromatography. We quantitatively analyzed the amount of released components and that of decomposition from various pitches. Volatile controlled pitch was made at laboratory scale, and volatile components and components released during carbonization were compared with those of commercial pitch. From these results, we discussed the possibility of making a pitch with reduced volatile components. Also, we discussed PAH emission from the pitches with different volatile components.

11:15 AM
LP Bricks: A New Generation of Refractories That Meet Higher Demands in Anode Baking Furnaces: *Marcel C. Franken*[1]; [1]Gouda Vuurvast NV, PO Box 56, Gouda 2800 AA Netherlands

There is a tendency in anode baking furnaces to reduce the thickness of the flue walls; the main purpose of this reduction is to increase the size of anodes (spacing). If the thickness of the flue wall is reduced with 10% the life time of the flue wall will be reduced up to 20%. To overcome this problem Gouda Vuurvast NV developed a new generation of bricks with a reduced permeability and a lower alkali content. The lower permeability prevents alkali- and fluoridecontaining gasses to penetrate in the brick. The lower alkali content gives a better starting point of the new flue walls and more "penetrated" alkali can be absorbed by these bricks before the flue walls will collapse. The lower permeability and the lower alkali content will result in a longer life time of the refractories. A number of field tests with this new generation of LP-bricks are running.

11:40 AM
New Developments About Safety and Process in Heating Equipment for the Anode Baking Furnace: *Jean Bigot*[1]; Jean Paul Kreuwen[1]; Jean-Christophe Rotger[2]; Yann El Ghaoui[2]; Hubert Gay[3]; [1]Aluminium Pechiney, Centr' Alp, BP 7, Voreppe Cedex 38341 France; [2]Aluminium Pechiney, LRF, BP114, 73303, Saint Jean de Maurienne Cedex France; [3]Setaram Engineering, 7, rue de l'Oratoire, 69300, Caluire France

Following the issuance of a European safety instruction aiming at improving the safety of firing process and protection of operating personal, all the heating equipment operated in European countries as of 2000 must be designed with regards to the new EN 746-2 safety standard. As this instruction applies also to Anode Baking Furnace, Aluminium Pechiney and Setaram Engineering have worked in cooperation to integrate the recommendation of the new standard in the heating equipment used to bake anode in open type baking furnace. This work of improving safety has led to develop new means, of which the use improves the heating process and reduces fuel consumption and tars emissions. This paper presents the result of this work, which has been implemented in the new heating equipment started last year in Alcan Vlissingen, Aluminium Dunkerque, and Tomago Aluminium.

12:05 PM
Experience with Regenerative Thermal Oxidation as a Fume Treatment Technology for an Open Ring Type Anode Baking Furnace: *Aad Kooijman*[1]; Henry Visser[1]; Karel Verheesen[1]; H. Thalhammer[2]; [1]Aluminum & Chemie Rotterdam B.V, Oude Maasweg 80, 3197KJ Botlek-Rotterdam Netherlands; [2]CTP Chemisch Thermische Prozesstechnik GmbH, Schmiedlstrasse 10, A-8052 Graz Austria

In 2003 Aluchemie has increased capacity with the construction of bake furnace #7 by approximately 25%. The emissions of volatile hydrocarbons from the baking furnaces is a major concern for the environmental authorities. In order to meet the more stringent environmental requirements for the new furnace a technique based on regenerative thermal oxidation for the fume treatment plant was introduced. This paper presents some specific considerations for the configuration of the fume treatment plant together with the experiences gathered in the first year of operation.

Cast Shop Technology: DC Casting: Melt Flow and Cooling

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday AM Room: 2001
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Rene Kieft, CORUS, Rsch. Dvlp. & Tech., IJmuiden 1970 CA The Netherlands

8:30 AM
An Experimental Heat Flux Measurement of the Wagstaff Water Hole Mold: *Ho Yu*[1]; [1]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA

In the high temperature quenching process of aluminum ingot casting, heat transfer from the ingot mold cooling water is one of the key mechanisms that affect casting performance such as ingot cracking and breakout. There are many commercial ingot mold types used within the aluminum industry. Their cooling water heat transfer characteristics, however, are unknown. The scope of this paper includes the construction of the mold secondary cooling water simulator in the Alcoa Technical Center and the experimental heat flux measurement of the Wagstaff water hole mold. The Wagstaff water hole mold was chosen because it is widely used in the aluminum cast shops. In the experiments, heat fluxes were measured as functions of ingot surface temperature at the water impingement point and as well downstream locations. Three water hole mold designs were used in the experiments:15 degree mold, 15 degree mold with inserts and 30 degree mold. Cooling water used in the experiments was de-ionized water in order to eliminate water chemistry effect. The experimental measurements provided the data base for generating empirical correlations of cooling water heat fluxes as functions of ingot surface temperature, ingot location and cooling water flow rate. The correlations are in forms that are suitable to be used as boundary conditions in the in-house Alcoa ingot computer model.

8:55 AM
Calculation of Heat Transfer Coefficients at the Ingot Surface During DC Casting: *Kazunori Kuwana*[1]; Srinath Viswanathan[2]; John A. Clark[3]; Adrian S. Sabau[4]; Mohamed Hassan[1]; Kozo Saito[1]; Subodh K. Das[5]; [1]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0503 USA; [2]Sandia National Laboratories, Matls. & Process Scis. Ctr., MS 0889, Bldg. 701, PO Box 5800, Albuquerque, NM 87185-0889 USA; [3]Albany Research Center, 1450 Queen Ave., SW, Albany, OR 97321 USA; [4]Oak Ridge National Laboratory, Metals & Ceram. Div., Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [5]Secat, Inc., 1505 Bull Lea Rd., Lexington, KY 40511 USA

Surface heat transfer coefficients representing the various regimes of water cooling during the direct chill (DC) casting of aluminum 3004 alloy ingots have been calculated using the inverse heat transfer technique. ProCAST, a commercial casting simulation package, which includes heat transfer, fluid flow, solidification, and inverse heat transfer, was used for this effort. Thermocouple data from an experimental casting run, and temperature-dependent thermophysical properties of the alloy were used in the calculation. Several variables, such as the use of a structured vs. unstructured mesh, and the presence or absence of fluid flow, were evaluated. The calculated effective heat transfer coefficient, which is a function of temperature and time, covers three water cooling regimes, i.e., convection, nucleate boiling, and film boiling, and the change of water flow rate with time.

9:20 AM
Innovative Technology Uniting New Chemicals with Advanced Monitoring and Control Optimizes the Performance of Cooling Water Systems in Metal Production Processes: *Eugene B. Smyk*[1]; Joseph J. Mazur[1]; [1]Nalco Company, 1601 W. Diehl Rd., Naperville, IL 60563 USA

The consistent performance of a cooling water system is critical to the success of many operations in aluminum, copper and steel production. This paper discusses new technology used to monitor the dynamic stresses of industrial cooling systems that results in fully automating the treatment and operating practice of these critical systems. New molecular chemistry and performance-based sensors that enable

this automation are introduced and discussed. The impact of on-line monitoring and control of the chemical actives on the major stresses experienced in these systems, including corrosion and microbial activity will be presented. Performance results including economic payout to the user, reliability and accuracy of the instrumentation, response to system upsets and the minimal operator involvement are discussed in several case studies.

9:45 AM Cancelled
Molten Aluminum Temperature Measurement with the Use of a Novel Thermocouple Assembly

10:10 AM Break

10:20 AM
Development of a Sensor to Measure Magnitude and Direction of Velocity in Liquid Aluminium: Blas Melissari Cassanello[1]; *Stavros A. Argyropoulos*[1]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., 184 College St., Toronto, Ontario M5S 3E4 Canada

This paper deals with the development of a sensor to measure magnitude and direction of velocity in Liquid Aluminium. The sphere melting technique was employed to measure magnitude of velocity. In addition using a modification of this technique the direction of velocity was detected. In this development, both aspects, experimental as well as computational work was used in tandem and they will be described in detail. The potential usage of this sensor in other high temperature liquid metals will be also discussed.

10:45 AM
Development and Use of a New Composite Material for Aluminum Contact Applications: *Sylvain P. Tremblay*[1]; Mark Vincent[2]; [1]Pyrotek Inc., 1623 Manic St., Chicoutimi, Quebec G7K 1G8 Canada; [2]Pyrotek Engineering Materials Ltd., Garamonde Dr., Wymbush, Milton Keynes, Buks MK8 8LN UK

A new composite material consisting of fiberglass fabric infiltrated with a CaSiO3 slurry will be described. Its properties as well as its behavior in contact with molten aluminum will be presented. Several plant case studies using this composite material also called "RFM" will be detailed. Utilizing its excellent properties, RFM has improved molten metal quality and brought a new problem-solving dimension to many applications. Applications such as the control pin, auto-pour ladle, continuous rod caster launder and skim dam will be detailed. Actual plant results in terms of improved metal quality and lower cost per ton usage will be shown in these applications.

11:10 AM
New Development of Calcium Silicates Especially for Flow Control and Distribution of Liquid Aluminium and Practical Results: *Volker Krasselt*[1]; Octavian Anton[2]; Mario Peter Görner[1]; [1]Promat GmbH, High Temp. Insulation, Scheifenkamp 16, Ratingen 40878 Germany; [2]Promat International NV, PRTC, Bormstraat 24, Tisselt 2830 Belgium

Within the Promat organization there are 4 different plants for the production of calcium silicate working with 6 different production technologies. This means a wide flexibility for Promat regarding R&D approach and for transfer to the production. Flow control and distribution of liquid aluminium with calcium silicates is an important market which demands precise shaped parts with high resistance to Al and with extended lifetimes. The most important process in calcium silicate technology is the autoclaving where morphology, crystal structures and pore sizes are designed. Promat developed a new method for the production of calcium silicate products: Mineral engineering and engineered matrices. Controlled crystal growth by using advanced technology allows the creation of crystal assemblages with entangled crystals of different dimensions, apparent densities and aggregate shape. Application results of the new development will be illustrated.

11:35 AM
The Effect of Process Parameters on the Metal Distribution for DC Sheet Ingot Casting: *Martin Fortier*[1]; A. Larouche[1]; X.-G. Chen[1]; Y. Caron[1]; [1]Alcan International Limited, Arvida R&D Ctr., 1955, Mellon Blvd., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

The liquid metal flow inside an ingot sump is a key issue in the casting of sound DC sheet ingots. The flow patterns are dependant on the design characteristics of the metal distributor and process parameters used (casting speed, skim dam, dip tube, etc.). To fully understand the effects of process parameter changes on metal distribution, studies were performed using water modelling, measurements during DC casting and mathematical modelling. The parameters studied were: time for transfer and distribution, casting speed, casting temperature, presence of a skim dam, distributor bag deformation and presence of a spout sock. The results show two distinct modes of metal distribution. These modes are respectively caused by the buoyancy driven flow and by the presence of the bag outflow. The interactions between the studied process parameters and the two metal distribution modes are described, giving a better understanding of the resulting variations inside the ingot sump.

12:00 PM
Interaction Between Structure Formation and Melt Flow During Solidification: First Results on Experiments With Electromagnetic Pump: *Andrey Turchin*[1]; Dmitry Eskin[1]; Laurens Katgerman[2]; [1]Netherlands Institute for Metals Research, Al Production, Rotterdamseweg 137, Delft, Zuid Holland 2628 AL Netherlands; [2]Delft University of Technology, Matls. Sci. & Engrg., Rotterdamseweg 137, Delft, Zuid Holland 2628 AL Netherlands

Melt flow is intrinsic in casting processes and influences strongly the resultant microstructure of a cast product. Only few experimental data are available on effects of melt flow on structure formation during solidification. The paper describes a new experimental setup and first results on the interaction between melt flow and structure formation during solidification of an Al-4.5% Cu alloy. An electromagnetic pump is used as a base for the experimental installation that includes also a control system, a metal guiding circuit, and a water-cooled chill for solidification of an alloy under controlled melt flow conditions. Such parameters as melt temperature, flow rate (2 to 25 cm/s) and temperature gradient are controlled. The solidification patterns are formed at the bottom of the water-cooled copper chill placed on top of the channel with the moving melt. The investigated parameters are grain size, growth direction and morphology, dendritic arm spacing, macro- and microsegregation.

12:25 PM
New Insights into Flow Structure in DC Casting of Aluminum Alloys: *Miha Zaloznik*[1]; Bozidar Sarler[2]; [1]Impol d.d., R&D Dept., Partizanska 38, Slovenska Bistrica SI-2310 Slovenia; [2]Nova Gorica Polytechnic, Lab. for Multiphase Processes, Vipavska 13, Nova Gorica SI-5000 Slovenia

DC casting of an aluminum alloy billet is numerically simulated. Of central interest in this paper is the melt flow structure, which is known to affect the predominantly advective species transport and thus macrosegregation. Therefore a more accurate finite-volume method (ULTRA-QUICK) is implemented for the solution of the velocity field, in contrast to popular but inaccurate first-order upwind methods, previously widely used for computations of melt flow. Because the focus is on the flow, a simplified physical model is employed, omitting species transport, but retaining a mushy zone. The results obtained with the more accurate method reveal a much more intricate flow structure than previously known. Several additional circulation cells are present at the bottom of the liquid pool. This result has serious implications for macrosegregation, caused by the flow inside and next to the mushy zone.

Characterization of Minerals, Metals and Materials: Materials Testing and Evaluation

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

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Session Chairs: Mitch Loan, University of Limerick, Matls. & Surface Sci. Inst., Limerick Ireland; Jeong Guk Kim, Korea Railroad Research Institute, Railroad Safety Rsch. & Testing Ctr., Kyounggi 437-050 S. Korea

8:30 AM
Utilizing the Novel Laser Spallation Technique to Measure Copper-Dielectric Adhesion Strength in Microelectronic Packages: *Arun Raman*[1]; Jun Tian[2]; [1]Intel Corporation, ATD Q&R, Mailstop CH5-263, 5000 W. Chandler Blvd., Chandler, AZ 85226 USA; [2]University of California, Dept. of Mechl. & Aeros. Engrg., 420 Westwood Plaza, Rm. 32-121, Los Angeles, CA 90095 USA

Laser spallation is a novel high strain rate ($10^6 s^{-1}$) metrology to measure thin film adhesion. A typical characteristic of slow strain rate

adhesion tests (10^{-5}-$10^{0}s^{-1}$), such as peel and 4-point bend tests, is in the contribution of plastic and viscoelastic losses to the measured adhesion strength. With laser spallation, due to the very large strain rate, the measured adhesion strength is closer to the true intrinsic value. This study reports copper-dielectric adhesion strengths of about 180MPa measured using laser spallation on test coupons of typical organic substrates used in microelectronic packages. Upon exposure to several hours of high temperature and high relative humidity, there is a change in failure locus, associated with approximately a 25% reduction in adhesion strength. An explanation of the observed phenomena is offered. In addition, laser spallation measured adhesion strengths are also compared with measurements from the more conventionally utilized slow strain rate peel tests.

8:55 AM
Automated Ball Indentation Test Methods: Progress and Accomplishments 1989-2004: *Fahmy M. Haggag*[1]; Robert L. Bridges[2]; [1]Advanced Technology Corp., 1066 Commerce Park Dr., Oak Ridge, TN 37830 USA; [2]BWXT-Y12, LLC, PO Box 2009, Oak Ridge, TN 37831 USA

The Automated Ball Indentation (ABI) test techniques were invented in 1989 to measure key mechanical properties of metallic samples and structures in a nondestructive and localized fashion. A single ABI test replaces the tension test for metallic materials and the fracture toughness test for Ferritic steels. This paper describes the ABI test methods, the progress of their capabilities, and example laboratory and field/in-situ applications in numerous industries. The laboratory version of the patented Stress-Strain Microprobe (SSM) technology has been in commercial use since 1991, and the portable SSM version received a 1996 R&D 100 Award. In 1999, a new miniature SSM system was introduced to provide easier in-situ applicability. The accuracy, reliability, and easy field applicability of the SSM system to test materials with unknown properties have been demonstrated on samples and in-service/operating pipelines as well as on components from other industries.

9:20 AM
A Fully Automated Technique for Twin Identification Through Electron Backscatter Diffraction: *Benjamin L. Henrie*[1]; Thomas A. Mason[1]; [1]Los Alamos National Laboratory, Matl. Sci. & Tech., G755, Los Alamos, NM 87545 USA

A fully automated framework has been developed for extracting twin statistics from deformed microstructure using crystallographic twin identification techniques with spatially correlated electron backscatter diffraction (EBSD) data. The key features of this analysis are the use of the mathematical definition of twin relationships, the inclination of the common K1 plane at a twin boundary, and a voting scheme for determining the parent orientation in a parent/twin pair. Twin area fractions are categorized by operative twin systems, number of active twin variants in each system, and corrected twin widths for α-zirconium and Stainless Steel 316L.

9:45 AM
Recent Advances in Concentration of Fine Particles by Hydroseparation: Vladimir N. Rudashevsky[1]; Nikolay S. Rudashevsky[1]; *Louis J. Cabri*[2]; [1]Center for New Technologies, Roentgena St. 1, St. Petersburg Russia; [2]Cabri Consulting Inc., 99 Fifth Ave., Ste. 122, Ottawa, Ontario K1S 5P5 Canada

Study of rare and accessory minerals in rocks, ores, industrial products, and potentially polluting materials is usually limited by the scarcity and small grain size of the target minerals. Recent advances in laboratory concentration methods have applied the new technology of hydroseparation (HS-01, Rudashevsky et al., 2002; HS-02 Cabri, 2004). This water-based environmentally friendly technology is very efficient in separating trace quantities of dense minerals from fine fractions of powdered, sieved samples, but is also very sensitive to manual adjustments of the water-flow regulator (WFR) and contamination from older plumbing systems. A new model hydroseparator (HS-11) has been specially designed for automatic control of concentration of heavy fine particles,including filtering of pollutants and better control (automated) of the WFR. Tests using the HS-11 will be described, which in addition, also shows promise for scale-up of the methodology for treating coarser particles on a pilot-plant scale.

10:10 AM Break

10:20 AM
Carbothermal Reduction - Nitridation Study of Aluminum-Containing Raw Materials: *F. S. Cinar*[2]; O. Yucel[1]; B. Derin[1]; S. Ercayhan[1]; V. Kizilirmak[1]; [1]Istanbul Technical University, Faculty of Chmst. & Metall., Dept. of Metallurgl. & Matl. Engrg., 34469, Maslak, Istanbul Turkey; [2]Prof. Adnan Tekin High Technological Ceramics and Composites Research Center, 34469, Maslak, Istanbul Turkey

In this study, two different aluminum containing materials (Seydisehir Al(OH)3 and Al2O3) have been used as starting materials to produce AlN powders. For both raw materials, the carbothermal reduction and nitridation process has been carried out in a horizontal tube furnace under flow of nitrogen gas. Starting alumina and aluminum hydroxide powders were wet mixed with carbon black separately in weight ratio of 1:1 (Al2O3:C) and (Al(OH)3:C) in a ball mill for 6 hours. The effects of reaction temperature (1400-1600°C) and time (0-240 min) on the reaction mechanism and morphologies of AlN powder have been investigated using XRD, SEM, BET and sedimentation methods. Full conversion to AlN which is submicron size and spherical particles has been obtained at 1600°C for 1 hour or at 1500°C for 4 hours for experiments used Al2O3 as a starting material. However 100% AlN conversion in the samples used Al(OH)3 powder as a starting material, could not be obtained even by the reaction at 1600°C for 240 min.

10:45 AM
Investigation of Residual Stresses Superposition of D2 Dies Due to Heat Treatment and Multipass Grinding: *Olga Karabelchtchikova*[1]; Iris V. Rivero[2]; [1]Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609-2280 USA; [2]Texas Tech University, Industl. Engrg., Box 43061, Lubbock, TX 79409-3061 USA

The study investigated residual stresses behaviors and their superposition effect in D2 dies for thread-rolling applications. A nested factorial experiment was applied to test hypotheses that the residual stresses profiles change due to combination of heat treatment and multipass grinding operations, and that certain patterns can be derived due to the memory relationship between pre-existing and final residual stresses distributions. The amount of the retained austenite along with residual stresses in variation with depth was measured using x-ray diffraction technique. Statistical analyses indicate that the proposed hypotheses hold. Residual stresses distributions as well as tensile peak location were found to be significantly influenced by the treatment parameters used. Unique characteristic patterns in the residual stresses profiles were observed due to multipass grinding technique across all experimental conditions. This finding suggested a plausible memory relationship between the cutting passes, and the causes of the foregoing results are further proposed and discussed.

11:10 AM
An Improved Thermal Fatigue Resistance Characterisation Method for Refractories: *Roger Pelletier*[1]; Claude Allaire[1]; [1]École Polytechnique de Montréal, Engrg. Physics, CIREP - Campus CRIQ, 8475, Christophe-Colomb, Montréal, Québec H2M 2N9 Canada

Thermal shock and thermal fatigue resistances (TFR) are traditionally estimated by methods requiring significant human intervention and because of that, suffer from low lab to lab reproducibility. This paper describes a novel TFR characterisation method that reduces to a minimum this problem. In this method, the thermal cycles are produced by moving specimens from a furnace to a copper plate using a fully automated apparatus. The damage level is measured using a non-destructive method. This allows to obtain the damage evolution curve (DEC) with a single pair of specimens. Parameterisation of these curves is used to quantify the evolution of the DEC's as a function of the temperature drop amplitude. The capability of the method is illustrated with experiment results. The shape of the damage evolution curves and the significance of their parameters are also discussed.

11:35 AM
Effect of Microwave Irradiation on the Characteristics of Bentonite Particles: *Bowen Li*[1]; *Janny-Yang Hwang*[1]; Xiaodi Huang[1]; [1]Michigan Technological University, IMP, 1400 Townsend Dr., Houghton, MI 49931 USA

The modification of interlayer structure in montmorillonite is one of the most important fields for the utilization of clay minerals. The effect of microwave energy irradiation to the characteristics of bentonite particles was investigated with laser particle analysis, SEM and XRD. Heating of bentonite powder with microwave energy (2.45 GHz) started at room temperature. The sample was excited to jump around like in boiling state. After 5 minutes of microwave irradiation, there is not significant alternation on the distribution curve of the particle size of bentonite and the surface topography of montmorillonite particle. There is only a slight increase in quantity for particles in 40-90 micron range and a slight decrease in 20-35 micron range. The peak (001) of montmorillonite weakens after 1 minute with microwave irradiation, and disappeared after 5 minutes.

Computational Thermodynamics and Phase Transformations: Theory and Simulation of Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Wednesday AM Room: 3005
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Jeffrey J. Hoyt, Sandia National Laboratories, Albuquerque, NM, USA

8:30 AM Invited
Predicting Structures from First-Principles Density Functional Calculations: Smoothing of Total Energy Surfaces: *Vidvuds Ozolins*[1]; Blanka Magyari-Kope[1]; Babak Sadigh[2]; [1]University of California, Matls. Sci. & Engrg., PO Box 951595, Los Angeles, CA 90095-1595 USA; [2]Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA

Ab initio density-functional theory (DFT) methods have the potential to fundamentally change the way new material discoveries are made. However, a persistent difficulty in applying these methods to designing new materials is the need to know detailed structural information at the atomic level. Search for thermodynamically stable phases is complicated by the existence of many local minima in the configuration space, the number of which increases exponentially with the number of atoms. Even though the previous 10-20 years have seen remarkable advances in constructing predictive Hamiltonians (e.g., cluster expansion), in practice they are limited to simple lattice-based cases and one- or two-component systems. We suggest an alternative approach which searches for structures directly during a self-consistent DFT calculation. Using smoothing methods to eliminate local minima, we can efficiently search for the most stable structures in systems that consist of many (i.e., more than two) components, are not based on a lattice, and may exhibit broken symmetries. Our approach also permits calculations in the grand-canonical ensemble.

9:00 AM Invited
First-Principles Atomistic Modeling of Ordering Phenomena and Phase Diagrams: *Manfred Fahnle*[1]; Ralf Drautz[1]; Frank Lechermann[1]; Alessandro Diaz-Ortiz[2]; Reinhard Singer[1]; Helmut Dosch[1]; [1]Max-Planck-Institut fuer Metallforschung, Heisenbergstrasse 3, Stuttgart D-70569 Germany; [2]Instituto Potosino, San Luis Potosi 78231 Mexico

In this talk we first review the fundamentals of and the interrelations between the three main techniques used to represent the adiabatic energy hypersurface of a multi-component system, i.e., the expansion into many-body potentials, the cluster functionals and the cluster expansion method (CE). It is shown how perfectly transferable many-body potentials can be defined and determined ab initio, and how the many-body potentials (which focus on the positional degrees of freedom) and the CE (which focusses on the degrees of freedom of chemical ordering) can be merged to a CE not confined to lattices. Concerning the applications of the CE method, we discuss the dense sequence of ground states found for Co-Fe when varying the composition, the rich phase diagram of the ternary system (Fe-Ni)-Al, the unusual ordering phenomena at the (110)-surface of Ni-rich Ni-Al and the search for chainlike adatom configurations on Mo(112).

9:30 AM Invited
Ab-Initio Thermodynamics of Alloys: From the Local Atomic Structure to the Substitutional Ordering of One Million Atoms: *Stefan Müller*[1]; [1]University Erlangen-Nürnberg, Lehrstuhl für Festkörperphysik, Staudtstr.7, Erlangen 91058 Germany

Although density functional theory (DFT) based calculations allow us to study several bulk and surface properties of solids, they are restricted to a small number of atoms.It will be demonstrated how this limitation can be overcome by combining DFT calculations with so-called Cluster Expansion (CE) methods and Monte-Carlo simulations. This concept gives access to both, huge parameter-spaces (e.g. for ground-state searches) and systems containing more than a million of atoms (e.g. for microstructure studies). It permits us to treat alloy properties which possess a delicate temperature-dependence like mixing enthalpies, short-range order or precipitation {\em without} any empirical parameters as input, but with an accuracy that allows the {\em quantitative} prediction of experimental results. The presented examples reach from fcc- and bcc-based disordered alloys up to intermetallic compounds. The approach is extended to alloy surfaces for investigations of ordering phenomena at surfaces and segregation, i.e. the deviation of the alloy's composition in the near-surface region compared to the bulk.

10:00 AM
First Principles Calculations of Alloy Phase Diagrams by Statistical Moment and Cluster Variation Methods: Inclusion of Anharmonicity of Thermal Lattice Vibrations: *Kinichi Masuda-Jindo*[1]; [1]Tokyo Institute of Technology, Matls. Sci. & Engrg., Nagatsuta 4259, Midori-ku, Yokohama, Kanagawa 226-8503 Japan

The thermodynamic quantities of metals and alloys are studied using the moment method in the quantum statistical mechanics, going beyond the quasi-harmonic approximations. Including the power moments of the atomic displacements up to the fourth order, the free energies and related thermodynamic quantities of alloy systems are derived explicitly in closed analytic forms.[1] The configurational entropy term is taken into account by coupling the present moment expansion scheme with the cluster variation method (CVM). The energetics of the binary alloys are treated within the framework of the first-principles TB-LMTO method coupled to CPA and GPM (generalized perturbation method). The applications of the present scheme are given both for the phase separating CuAg and order-disordered binary alloys, NiAl and Ta-W alloys. [1]K. Masuda-Jindo, V. V. Hung and P. D. Tam, Phys. Rev., B67(2003)094301-1.

10:20 AM Break

10:30 AM
Modeling Mg Alloys with a Mixed-Basis Cluster Expansion: *Gus Hart*[1]; Chris M. Wolverton[2]; [1]Northern Arizona University, Physics & Astron., PO Box 6010, Flagstaff, AZ 86011-6010 USA; [2]Ford Research Laboratory, MD3028/SRL, Dearborn, MI 48121-2053 USA

Encouraged by the success of first-principles cluster expansion methods for large-scale modeling of cubic alloys, we are developing a mixed-space cluster expansion approach for hexagonal-close-packed alloys (as well as other symmetries). We have developed an explicit strain model, an essential component of cluster expansion models for modeling precipitate formation. Using the model we demonstrate the contribution of strain to the formation enthalpies of several ordering and phase-separating binary Mg alloys, and demonstrate cluster expansions for each.

10:50 AM
First-Principles Prediction of Metastable Precipitate Phase Boundaries in Al-Cu: *Chinnappan Ravi*[1]; Chris M. Wolverton[2]; Vidvuds Ozolins[3]; [1]Ford Motor Company, (Pennsylvania State University), Physl. & Environmental Scis., MD 3083/SRL Bldg., PO Box 2053, Dearborn, MI 48121 USA; [2]Ford Motor Company, Physl. & Environmental Scis., MD 3083/SRL Bldg., PO Box 2053, Dearborn, MI 48121 USA; [3]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095 USA

In Al-Cu alloys, precipitation strengthening via heat treatment is a common practice to enhance the mechanical properties by the introduction of metastable precipitates. Knowledge of the metastable phase boundaries is important for understanding the strengthening response of various precipitate phases, as these boundaries dictate the maximum possible phase fraction of precipitate for a given composition and temperature. However, these metastable solvus boundaries are often difficult to determine unambiguously from experiment. We present here an entirely first-principles approach to computationally predict the Al-Cu solvus boundaries of the equilibrium θ (Al_2Cu) phase, as well as the metastable θ' (Al_2Cu) and GP zone phases. The calculated boundaries are obtained from first-principles T=0K energetic calculations of precipitate and solid solution phases, as well as first-principles frozen-phonon calculations of the full dynamical matrix for the vibrational free energies. Since the problem involves the dilute limit, configurational degrees of freedom are treated in a mean-field sense.

11:10 AM
The Entropy of Alloys: *Marius Stan*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., PO Box 1663, MS-G755, Los Alamos, NM 87545 USA

A major problem in simulating thermodynamic properties of alloys is modeling the entropy. While configurational entropy is incorporated in most stability calculations, the other components, such as the

vibrational and electronic entropy are often neglected or roughly estimated. In this work we propose a method of accounting for both configurational and vibrational entropy and discuss the electronic contribution for several actinide based alloys. The meaning of entropy in non-equilibrium thermodynamic processes is also discussed and illustrated for the case of phase transformations and diffusion.

11:30 AM
Application of the Cluster/Site Approximation to the Calculation of Multicomponent Alloy Phase Diagrams: *W. Wilson Cao*[1]; Y. Austin Chang[1]; J. Zhu[1]; S.-L. Chen[2]; W. A. Oates[3]; [1]University of Wisconsin, Dept. Matl. Sci. & Engrg., Madison, WI 53706 USA; [2]CompuTherm LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [3]University of Salford, Inst. for Matls. Rsch., Salford M5 4WT UK

The Cluster/Site Approximation (CSA) offers computational advantages, without loss of accuracy, over the Cluster Variation Method (CVM) in the calculation of multicomponent phase diagrams. Its ease of use and advantages are illustrated in the calculation of a prototype Cu-Ag-Au fcc order/disorder phase diagram previously calculated using the CVM.

11:50 AM
Defect Structure and Degree of Order in Ni3Ga: Experiment and Modelling: *Olga Semenova*[1]; Hiroshi Numakura[2]; Herbert Ipser[1]; [1]University of Vienna, Inst. of Inorganic Chmst., Waehringerstr. 42, A-1090 Vienna Austria; [2]Kyoto University, Dept. of Matl. Sci. & Engrg., Yoshida Hon-machi, Sakyo-ku, Yoshida Hon-machi, Sakyo-ku, Kyoto, 606-8501, Kyoto 606-8501 Japan

Intermetallic compounds with ordered crystal structure exhibit attractive high-temperature properties because of the long-range ordered (LRO) superlattice which reduces dislocation mobility and diffusion processes at elevated temperatures. In the present investigation, the compound Ni3Ga with L12-structure was employed as a model compound to perform a calculation of LRO on the base of experimental data on measurements of order-disorder kinetics by residual electrical resistometry at respective temperatures. The ordering energies in the pair interaction model and defect concentrations as a function of temperature were estimated from experimental data. It was found that the order-disorder equilibrium in Ni3Ga is reached as a result of two processes with distinctly different rates. The ordering activation energies of both fast and slow processes in Ni3Ga were found equal 1.47 eV (fast process) and 2.66 eV (slow process).The obtained results evaluated in terms of statistical-theoretical model could be compared to the Wagner-Schottky and the Bragg-Williams models, as well as, to Monte Carlo simulations.

Converter and Fire Refining Practices: Advanced Technologies

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee
Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Wednesday AM Room: 2016
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Tim Smith, SNC-Lavalin, Copper, London, England; Phillip J. Mackey, Falconbridge Technology Centre, Falconbridge, Ontario, Canada

8:30 AM
Blister Flash Smelting - Efficient and Flexible Low-Cost Continuous Copper Process: *Jukka Tuominen*[1]; Ilkka V. Kojo[1]; [1]Outokumpu Technology Oy, Riihitontuntie 7E, PO Box 86, FIN-02201 Espoo Finland

The history of making blister copper in an Outokumpu Flash Smelting Furnace dates back to the late 1960's, when Outokumpu first piloted the Outokumpu Direct Blister (ODB) process. The first commercial scale applications have been in use at Glogow, Poland, since 1978 and at Olympic Dam, Australia, since 1988. These applications utilize special concentrates, which are low in iron, and thus the formation of slag is also low. In 1995 the same principle was applied to smelting copper matte into blister copper in industrial scale at Kennecott Utah Copper. This application, the Kennecott-Outokumpu Flash Converting process, will celebrate its 10th anniversary of commercial scale operation this year. This paper outlines the development in efficiency and flexibility of Blister Flash Smelting processes compared to other smelting and converting options. The focus is on the economical aspects i.e. on the total feasibility of both greenfield investment projects and revamping of existing smelters.

9:00 AM
An Update on Flash Converting at Kennecott Utah Copper Company: *Ryan Walton*[1]; Robert Foster[1]; David George-Kennedy[1]; [1]Kennecott Utah Copper Co - Smelter, PO Box 6001, Magna, UT 84044-6001 USA

The Flash Converting Furnace at Kennecott Utah Copper converted over 465kmt of high-grade matte in 2002 but was restricted due to low matte production to 395kmt in 2003. This paper presents updated operational performance data and describes improvements implemented since the mid 2001 rebuild. Matte burner feed distribution, silica and arsenic impacts on settler lime-based slag operability, blister laundering, uptake/boiler accretion control, and dust sulfation will be discussed, as well as current development requirements dictated by operational imperatives and furnace modification plans.

9:30 AM
Risks and Opportunities in Continuous Converting for Nickel at Inco: *Jeff Donald*[1]; Ashok Dalvi[1]; *Tony Warner*[1]; Ken Scholey[2]; *Alistair G. Ross*[2]; Cameron Harris[3]; [1]Inco Technical Services Ltd, 2060 Flavelle Blvd., Mississauga, ON L5K 1Z9 Canada; [2]Inco Ltd, Procg., 18 Rink St., Copper Cliff, ON P0M 1N0 Canada; [3]HG Engineering Ltd, 400 Carlingview Dr., Toronto, ON M9W 5X9 Canada

For processing copper, continuous converting has been successfully employed in a variety of locations, but for nickel, there has yet to be a successful commercial implementation of continuous converting technology. At Inco's Copper Cliff smelter, there are specific requirements for the production of Bessemer matte relating to downstream processing that makes continuous converting for nickel mattes even more challenging. The nickel carbonyl refineries require very low iron levels in the matte, and the sulphur deficiency must be carefully controlled such that the appropriate quantity of PGM-rich metallics are produced from matte separation. The present paper outlines continuous converter technologies that have been implemented in copper, and evaluates their potential for nickel converting. The challenges of producing a Bessemer matte to the required specifications are discussed. The economic and environmental driving forces, and the challenges of justifying a novel technology in place of productive existing equipment, are discussed.

Extractive Metallurgy: Pyrometallurgy II

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Wednesday AM Room: 2018
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Pat Taylor, Colorado School of Mines, Metallurgl. & Matls. Engrg., Golden, CO 80401 USA; David Robertson, University of Missouri, Dept. of Metallurgl. Engrg., Rolla, MO 65409-1460 USA

8:30 AM Cancelled
Some Fundamentals for Partial Oxygen Lead Softening - The Role of Local Oxygen Partial Pressure

9:00 AM
Some Insights on Low-Temperature Gas-Phase Carbidization of Iron-Bearing Constituents in Red Mud: Regina Caupain[2]; *Gerard P. Martins*[1]; [1]Colorado School of Mines, MME Dept., 920 15th. St, Golden, CO 80401-1887 USA

In an effort to contribute to the research on the minimization of red mud, a study was performed involving low-temperature gas-phase carbidization of iron-bearing constituents of red mud in order to obtain iron carbides, which might serve as a viable feedstock for Electric Arc Furnace steelmaking. A dried precursor red-mud cake was "crushed" and the fraction in the particle size-range, 75µm d_p 150µm, was subjected to gas-phase carbidization, thus: CO-H2 gas mixture, molar ratio: YH2:YCO = 3:1; temperature range: 560°C to 650°C and (batch) reaction times of 15 to 45 minutes. Sooting never occurred during the

carbidization processing. Mössbauer analyses revealed that the iron-bearing phases in the carbidized product constituted 760wt.% cementite (Fe3C), 079wt% Hägg carbide (Fe5C2), 011wt.% Fe2O3, 08wt.% Fe3O4, 012wt.% Fe and 1028wt.% of an unidentified (nano-size) non-magnetic iron oxide phase. With longer reaction-times and higher reaction-temperatures the Hägg carbide content increased – this behavior was ultimately traced to the manner in which the reaction was quenched.

9:30 AM
Carbothermic Reduction of Nickel Oxide and Iron Oxide Containing Kiln Dust in an Inert Atmosphere at Elevated Temperatures: Glenn Hoffman[1]; *Jim McClelland*[1]; [1]Midrex Technologies, Inc., 2725 Water Ridge Pkwy., Charlotte, NC 28217 USA

Carbothermic direct reduction of a saprolite origin kiln dust containing nickel oxide and iron oxide was investigated under FASTMET and ITmk3 process conditions at temperatures ranging from 1,150 to 1,500°C. Carbon containing compacts were produced from the kiln dust and pulverized coal using a hydraulic press. The compacts were heated under inert atmosphere in a tube furnace at various temperatures. Reduction kinetics and rate controlling phenomena regarding thermal processing of the saprolite origin kiln dust will be discussed.

10:00 AM
Solid Oxide Membrane (SOM) Technology for Cost Effective and Environmentally Sound Production of Metals and Alloys Directly from their Oxides and Ore Concentrates: *U. B. Pal*[1]; A. Krishnan[1]; X. Lu[1]; [1]Boston University, Dept. of Mfg. Engrg., 15 St. Marys St., Brookline, MA 02446 USA

Recent progress in the use of Zirconia-based inert anodes for extraction of metals such as Ta and Mg directly from their oxides by the SOM (Solid Oxygen Ion Conducting Membrane) electrolytic process is highlighted. In the case of Mg the oxide is dissolved in the flux, and in the case of Ta the oxide can either be dissolved in the flux or used as part of the cathode. Fundamental aspects of the current-potential behavior are analyzed. Since the Zirconia-based anode is the most expensive part of the system, its long-term stability is critical to the success and eventual commercialization of the SOM process. Different ways to increase membrane stability as well as metal production rate are discussed.

10:30 AM Break

10:45 AM
Crystal Morphologies of ZnO Obtained by Oxidizing Zinc Vapor: *Yi-feng Chen*[1]; Mo-tang Tang[1]; [1]Central-South University, Sch. of Metallurgl. Sci. & Tech., Changsha, Hunan 410083 China

Effects of conditions of oxidizing zinc vapor at high temperature on the crystal morphologies of ZnO and relations of various morphologies are investigated in this paper. Results show that there are five typical morphologies-amorphous, granular, needle, tetrapod and multipod-like ZnO, depending on the physical chemical conditions of oxidizing zinc vapor and having no direct relations with particle size and surface conditions of zinc powders, the raw materials. The behavior of oxidizing zinc vapor varies with oxygen partial pressure, which leads to produce ZnO crystals of different morphologies that can be controlled by the change of preparing conditions.

11:15 AM
Preparation of Fibrillar Nanocrystalline Nickel Powder by Precursor Thermal Decomposition: Wu Jian Hui[1]; *Zhang Chuan Fu*[1]; Zhan Jing[1]; Li Chang Jun[1]; Bai Meng[2]; [1]Central South University, Sch. of Metall. Sci. & Engrg., Changsha, Hunan China; [2]JiangXi Copper GuiXi Smelter HuaXin Metal Liabilities Co., Ltd, Guixi, Jiangxi 335424 China

The precursor thermal decomposition process of fibrillar nanometer scale nickel powder and passivation treatment of obtained powder are investigated, and the composition and morphology of the products are characterized by use of IR, TGA/DTA, XRD and SEM. The results show that the precursor morphology is a decisive factor to the product morphology of the nickel powder. The morphology of the product derived at low decomposing temperature is irregular. The nickel powders produced at temperatures higher than 480 C cohere seriously. The product particles are reunited with the decomposition time prolong. The powder prepared under the optimal condition is fibrillar like woolen yarn and the grain diameter is less than 50nm.

11:45 AM
Reduction Kinetics of Dusts and Pellets from Nickeliferous Laterite Rotary Kiln Treatment: *Emmanuel N. Zevgolis*[1]; Ismene-Polyxeni Kostika[1]; Iliana Halikia[1]; Elias Rigopoulos[2]; [1]National Technical University of Athens, Sch. of Mining & Metallurgl. Engrg., Zografou Campus, 15780, Athens Greece; [2]Silver and Baryte Mines, Metallurgl. Engrg., Milos Island Greece

In this work, the reduction kinetics of dust from nickeliferous Greek laterites is presented. The dust is a by-product from laterite treatment in rotary kilns and it comes from washing towers, electrostatic filters and polycyclones. Pellets made from this dust were also examined. Reducibility was studied in the temperature range of 700°C to 850°C in a laboratory tube furnace. From this study it comes that initially the chemical reaction mechanism prevails in the reduction process, followed by the mixed kinetic model, in the first period of reduction. After that period, reduction becomes diffusion controlled. These results fit well with the experimental data until 800°C. At higher temperatures, diffusion is the rate controlling-step. Activation energy values calculated by Arrhenius law, confirm the proposed mechanisms.

Friction Stir Welding and Processing III: Modeling

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday AM Room: Nob Hill C/D
February 16, 2005 Location: San Francisco Marriott

Session Chair: Stan A. David, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

8:30 AM Keynote
Using Process Forces as a Statistical Process Control Tool for Friction Stir Welds: *William J. Arbegast*[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. & Joining Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Many OEM equipment manufacturers now provide for direct measurement of and feedback control on the principle forces (X-, Y-, and Forge) experienced during friction stir welding. Analysis techniques are being developed to convert these force measurements into SPC and feedback control algorithms that can be used to real-time monitor and control weld quality. Descriptions, examples and limitations of the X-Force, Pseudo-Heat Index, Direction Cosine, Resultant Force, and Fourier Transform control algorithms being developed at the Advanced Materials Processing Center are given. Correlations are made between processing parameters, algorithm results, metal flow patterns and the "FSW Metalworking Model", and, their relationship to weld quality.

9:00 AM Invited
Modeling, Analysis, and Validation of Friction Stir Welding and Processing: *Abe Askari*[1]; Stewart Silling[2]; Blair London[3]; Murray Mahoney[4]; [1]Boeing Company, PO Box 3707, MC 7L-25, Seattle, WA 96124-2207 USA; [2]Sandia National Laboratories, Albuquerque, NM USA; [3]California Polytechnic State University, San Luis Obispo, CA USA; [4]Rockwell Scientific, Thousand Oaks, CA USA

We have successfully developed predictive computational tools to model friction stir welding/processing (FSW/P). The model is based on a three-dimensional Eulerian code with complete thermo-mechanical coupling. The code models all the important physical effects with few assumptions. The code includes frictional heating and energy dissipation due to plastic work, thermal convection and conduction, and all relevant geometrical tool details. The Eulerian nature of the code permits modeling very large strains and predicts materials mixing. The model also predicts detailed thermal and deformation histories that impact the final microstructure. Detailed experiments and data analysis in aluminum, bronze, and steel alloys are used to validate the computational models and improve understanding of basic aspect of FSW/P processes. Placement of both tracers and thermocouples in the tool path, metallographic examination, and computed tomography are used to investigate metal flow dynamics and the mixing of material as well as thermal profile and history.

9:20 AM
An Analytical Model for Prescribing the Flow Around the Tool Probe in Friction Stir Welding: *H. Schmidt*[1]; J. Hattel[1]; [1]Technical University of Denmark, Process Modlg. Grp., Dept. of Mfg. Engrg. & Mgmt., Denmark

The objective of this paper is to present a simple model for prescribing a 2D velocity field for the material flow in the shear layer around the probe in Friction Stir Welding (FSW). By introducing an eccentrically shaped shear layer, and assuming a linear velocity profile between the welding flow outside the shear layer and the rotating velocity flow at the probe/shear layer interface, a robust 'model' for evaluating different aspects of the flow is presented. The model takes into account that the material entering the shear layer in front of the probe is transferred around the retreating side of the probe, which in turn calls for a non-symmetrical flow pattern due to the accumulation of material in the shear layer at the leading side and deposition at the trailing side. The rotation zone and transition zone are parts of the model. The model satisfies mass conservation, however, the momentum equations are not solved for. By displaying the velocity vector components, the flow field can be visualized as well as a phenomenological interpretation of a change in welding parameters. As input to the model, at least two parameters out of three variables are necessary. The three variables are: The shear layer thickness at two positions and the contact state variable. The shear layer is characterized by at least one reference thickness, e.g. at the advancing or retreating side. A uniform contact state variable is assumed at the probe/shear layer interface, i.e. the ratio between the tangential speeds of the probe and shear layer interface is constant along the circumference. Based on this, a rough estimate of flow rates in experimental welds can be made. Secondly, by measuring some specific dimensions from welds using marker material, average speeds and shear layer properties can be estimated with the model.

9:40 AM
Three-Dimensional Finite Element Model of the Friction Stir Spot Welding Process: *Karim Heinz Muci-Kuchler*[1]; Sri Satya Teja Kakarla[1]; Casey D. Allen[2]; William J. Arbegast[2]; [1]South Dakota School of Mines and Technology, Mechl. Engrg. Dept., 501 E. St. Joseph St., Rapid City, SD 57702 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Friction Stir Spot Welding (FSSW), originally developed by GKSS (Germany), has a strong potential to find applications in both the automotive and aerospace industries. At the present time, research efforts are taking place to gain a better understanding of the process, to explore different tool configurations, and to optimize the set of process parameters. In this regard, having a finite element model capable to simulate FSSW can be very useful to reduce the number of physical experiments required in those studies. In this paper, a simplified isothermal three-dimensional model of the FSSW process is presented. The model, based on a solid mechanics approach, was developed using the commercial software ABAQUS/Explicit. The results of the simulation are compared against available experimental information corresponding to the same tool geometry, sequence of operations, and process parameters.

10:00 AM
Analysis and Numerical Modeling of FSW Spot Welding Al 5754 and AZ91 Base Materials: *Adrian P. Gerlich*[1]; Peter Su[1]; Tom H. North[1]; Gabor Bendzsak[1]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Rm. 140, Toronto, ON M5S3E4 Canada

This paper presents the results produced during friction stir spot welding of Al-alloy (Al5754) and Mg-alloy (AZ91D) base materials for automotive applications. The FSW spot welding process were investigated using a combination of high-speed data acquisition output (axial force, torque, displacement and temperature), metallographic examination of completed joints and numerical modeling. The peak temperatures during FSW spot welding of Al 5754 was 0.96 Ts, where Ts is the solidus temperature of Al 5754 base material (590ºC). The peak temperature attained during FSW spot welding of Mg-alloy base material was 435ºC, which is very close to the melting temperature of the Mg-Al solid solution/Al12Mg17 eutectic (437ºC). Numerical modeling of the spot FSW process is carried out by implementing CFD techniques to simulate flow fields and the temperature distributions during welding. Simulation results are validated using metallographic cross sections and data acquired from thermocouple outputs.

10:20 AM Break

10:40 AM Invited
Modeling the FSW Process Using an Eulerian Finite Element Formulation: *Paul R. Dawson*[1]; Jae-Hyung Cho[1]; [1]Cornell University, Sibley Sch. of Mechl. & Aeros. Engrg., 196 Rhodes Hall, Ithaca, NY 14853 USA

Friction stir welding of stainless steel is modeled using a steady-state Eulerian finite element formulation. The governing equations for the velocity field, temperature distribution, and strength evolution are coupled to determine the thermomechanical history of the workpiece material. The viscoplastic behavior of the steel is modeled using a modified Hart's model in which a staturation flow stress is incorporated into a Voce-type hardening equation. During the welding process, intense shearing near tool causes rapid heating from viscous dissipation and an increased strength from strain hardening. Texture evolution is predicted from the computed velocity gradients along streamlines of the flow field. Upstream and downstream of the tool the deformation is nearly monotonic, causing steady strengthening of the texture. Material rotation around tool pin, however, causes the texture to form, break up, and reform repeatedly and rapidly. Mechanical properties such as elastic modulus, strength and R-value are analyzed from the deformation textures.

11:00 AM
CFD Modelling of the Shear Layer Around the Tool Probe in Friction Stir Welding: *H. Schmidt*[1]; J. Hattel[1]; [1]Technical University of Denmark, Process Modlg. Grp., Dept. of Mfg. Engrg. & Mgmt. Denmark

The present paper focuses on the formation of the shear layer around the probe in Friction Stir Welding (FSW). Aluminium alloys can be characterized as shear thinning or pseudo plastic which is the main reason for the development of very thin shear layer in FSW. A 2D CFD model using the FE software FEMLAB is presented. In order to analyse the flow field, a cylindrical probe rotating co-axially with the tool is modelled. At high shear rates, aluminium is well described by the power law shear rate-shear stress relationship. By changing the power law exponent (m), the degree of non-Newtonian, shear thinning behaviour of the material is investigated, and the effect is reflected in the change in shear layer properties. By following streamlines around the tool probe, a clear definition of the rotation and transition zone is proposed. A uniform contact condition is assumed at the probe/shear layer interface, thus a constant velocity boundary condition is prescribed. The results show that the shape/extent of the shear layer is highly dependent on the power law exponent. Higher shear rates (in the range of 1000 s-1) and narrowing of the shear layer are found for lower exponents. The extent of the simulated shear layer corresponds best to that observed in experiments for m=0.1. Due to the presence of the rotation zone, material entering and exiting the transition zone obtain peak velocities of a fraction (~0.5) of the maximum velocity found at the contact interface.

11:20 AM
Investigation of the Hot Deformation Characteristics During Friction Stir Welding of High-Purity Aluminum: *Bala Radhakrishnan*[1]; Zhili Feng[1]; Gorti Sarma[1]; Oleg Barabash[1]; Stan A. David[1]; [1]Oak Ridge National Laboratory, Computer Sci. & Math., Bldg. 5600, MS 6008, Oak Ridge, TN 37831-6008 USA

The quasi steady-state three-dimensional thermal and flow fields in the vicinity of the tool during friction stir welding of high-purity aluminum are modeled using the commercial flow software, FLUENT. The viscosity of aluminum is calculated from available stress-strain data at various temperatures and strain rates and used in the flow computations. From the steady state temperature and strain-rate fields the operating Zener-Hollomon parameters are calculated at various distances from the tool, and used to predict the size and shape of the swirl zone. The model results are compared with experimentally measured profiles for the swirl zone. The deformation substructure in terms of the grain size, grain size distribution, texture and grain boundary misorientation distribution are measured using orientation imaging microscopy. The model predictions combined with experimental measurements of substructure are used to elucidate whether dynamic recovery or dynamic recrystallization was the operating mechanism during hot deformation in the swirl zone.

11:40 AM
Model for Local Proof Strength of 2xxx Welds: *Marco J. Starink*[1]; Shuncai Wang[1]; Diccon P.P. Booth[1]; Ian Sinclair[1]; [1]University of Southampton, Matls. Rsch. Grp., Sch. of Engrg. Scis., Southampton SO17 1BJ UK

The microstructure development, and resulting hardness and yield strength profiles of 2xxx welds are modelled using a model which combines primary precipitation, coarsening, resolution, partial/full melting and resolidification (Sheil type) and re-precipitation. The strength contribution is dominated by two types of precipitates: the Cu-Mg clusters which form at low temperature and dissolve at temperatures above about 230C, and the S phase which dominates the reaction at the location of peak strength as well as at the locations of minimum strength. The model is compared to microstructural and hardness data on a friction stir weld.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Advanced Technology and Applications I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Wednesday AM Room: 3020
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Carl C. Koch, North Carolina State University, Matls. Sci. & Engrg., Raleigh, NC 27695-7907 USA; Roumiana S. Petrova, New Jersey Institute of Technology, Physics, Newark, NJ 07102 USA

8:30 AM
Beyond DLC: Development of Atomically Smooth Nanometer-Thick Protective Coatings for Extremely High-Density Recording Applications: *Yip-Wah Chung*[1]; [1]National Science Foundation, Civil & Mechl. Sys., Rm. 545.25, 4201 Wilson Blvd., Arlington, VA 22230 USA

In mid-2004, hard disk drives have storage densities in excess of 50 Gbits/in^2, which will increase to about 1 Tbit/in^2 within the next five years. In order to attain this storage density, the separation between the read-write head and the magnetic layer cannot be more than 5.0 nm. This creates interesting materials and engineering challenges to the manufacturing of such systems. One key challenge is the development of overcoats that are used to protect the disk and head surfaces from wear and corrosion. The thickness of this coating must be 1.0 nm or less. In this talk, we will focus on the development, synthesis, characterization and limitations of these overcoats for future disk drive applications. In addition, we will explore the application of molecular dynamics simulations to aid the synthesis of such coatings.

9:00 AM
Formation of Hydrogen-Free Diamond-Like Carbon: Theory and Experiment: *J. D. Haverkamp*[1]; [1]North Carolina State University, Dept. of Matls. Sci. & Engrg., Raleigh, NC 27695-7916 USA

Hydrogen-free diamond-like carbon films are known to possess interesting mechanical, optical, and electronic properties. These properties are fundamentally related to the fraction of sp^3 coordinated carbon atoms and the ordering of sp^2 coordinated carbon atoms in the diamond-like carbon film. It is therefore of interest to understand the mechanisms by which diamond-like carbon films are formed. To investigate this, diamond like carbon films are grown using pulsed laser deposition over a range of laser energy densities. Film properties are determined via electron energy-loss spectroscopy and visible Raman spectroscopy. Properties of the plasma created in pulsed laser deposition are studied using quadruple Langmuir probes, mass loss measurements, and magnetic field measurements. We propose a new model for the formation of hydrogen-free diamond-like carbon films based upon an activation barrier and electronic excitation of incident atoms. The model quantitatively predicts the sp^3 fraction as a function of pulsed laser energy density. The kinetic energy of incident species is shown to be a leading factor controlling the sp^3 fraction of deposited films, as the kinetic energy controls the interaction of the incident atom with the film. It is shown that the kinetic energy of neutral atoms is an important factor in the growth of diamond-like carbon films with pulsed laser deposition.

9:30 AM
Highly Nonlinear Contact Interaction, Dynamic Energy Dissipation and Nano-Fragmentation of Carbon Nanotubes: *Chiara Daraio*[1]; Vitali F. Nesterenko[1]; Joseph F. Aubuchon[1]; Sungho Jin[1]; [1]University of California, Matls. Sci., 9500 Gilman Dr., MC 0418, EBU2 - Rm. 266, La Jolla, CA 92093 USA

Mechanical response and energy dissipation of an array of carbon nanotubes under high-strain rate deformation was studied using a simple, drop-ball test with measurement of dynamic force between the ball and forest of nanotubes. The contact force exhibits a strongly nonlinear dependence on displacement, which is fundamentally different in comparison with the Hertz law. It is demonstrated that a forest of vertically aligned nanotubes may be successfully used as a strongly nonlinear spring in discrete systems, for monitoring signal propagation speed, and as a novel microstructure for localized energy absorption. We also report a new phenomenon of dynamic nano-fragmentation of carbon nanotubes by high-strain-rate stresses. The drop ball test caused them to break up into short segments in just 15 microseconds with a relatively uniform length range of ~100 - 150 nm. The ends of the fragmented nanotubes often exhibit an irregular oval or hexagonal cross-section. The observed cutting of nanotubes may conveniently be utilized for their resizing and end-opening for a variety of applications.

9:45 AM
Gel Stability in a Liquid Crystal System and Application to a Novel Templating Liquid Crystal: *Christopher R. Lubeck*[1]; Fanny Darmawan[1]; William C. Ewing[1]; Fiona M. Doyle[1]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg. # 1760, Berkeley, CA 94720-1760 USA

Liquid crystals can template inorganic semiconductors such as cadmium sulfide and zinc sulfide. The efficacy of the liquid crystalline system depends upon two major factors, the stability of the gel and the crystal structure of the inorganic phase. This paper investigates the former. Gel stability can be assessed through differential scanning calorimetry, notably the transition temperature at which the gel goes from an ordered to a disordered state. Various factors that may be manipulated during synthesis, such as the ionic concentration and the relative proportion of solvent to liquid crystal, can affect gel stability. These were examined. In addition, novel liquid crystalline systems were synthesized, and their ability to template inorganic phases was investigated. The resulting particles were characterized via transmission electron microscopy and atomic force microscopy.

10:15 AM Break

10:30 AM
ZnO Nanowire Devices: *Steve Pearton*[1]; [1]University of Florida, Matls. Sci. & Engrg., PO Box 116400, Gainesville, FL 32611 USA

The large surface area of the nanorods makes them attractive for gas and chemical sensing, and the ability to control their nucleation sites makes them candidates for micro-lasers or memory arrays. In addition, they might be doped with transition metal ions to make spin-polarized light sources. To date, most of the work on ZnO nanostructures has focused on the synthesis methods and there have been only a few reports of the electrical characteristics Single ZnO nanowire metal-oxide semiconductor field effect transistors (MOSFETs) were fabricated using nanowires grown by site selective Molecular Beam Epitaxy. When measured in the dark at 25°C,the depletion-mode transistors exhibit good saturation behavior, a threshold voltage of ~-3V and a maximum transconductance of order 0.3 mS/mm .Under ultraviolet (366nm) illumination, the drain-source current increase by approximately a factor of 5 and the maximum transconductance is ~ 5 mS/mm. The channel mobility is estimated to be ~3 cm2 /V.s, which is comparable to that reported for thin film ZnO enhancement mode MOSFETs and the on/off ratio was ~25 in the dark and ~125 under UV illumination. Pt Schottky diodes exhibit excellent ideality factors of 1.1 at 25°C and very low(1.5x10 -10A, equivalent to 2.35A.cm-2, at -10V) reverse currents. The on-off current ratio of the diodes at 0.15/ -5V was ~6. The nanowire diodes show a strong photoresponse, with the current-voltage characteristics becoming Ohmic under ultraviolet(UV) illumination(366nm light) with nanowire conductivity under UV exposure of 0.2 Ohm.cm. The photoresponse showed only a minor component with long decay times (tens of seconds)thought to originate from surface states. The results show the high quality of material prepared by MBE and the promise of using ZnO nanowire structures for solar-blind UV detection. In the temperature range from 25-150 úC, the resistivity of nanorods treated in H2 at 400 úC prior to measurement showed an activation energy of 0.089+/- 0.02 eV and was insensitive to the ambient used (C2H4,N2O,O2 or 10%H2 in N2). By sharp contrast, the conductivity of nanorods not treated in H2 was sensitive to trace concentrations of gases in the measurement ambient even at room temperature, demonstrating their potential as gas sensors. These results show the ability to manipulate the electron transport in nanoscale ZnO devices.

11:00 AM
New Novel Cu-Doped Above Room Temperature: *K. V. Rao*[1]; [1]Royal Institute of Technology, Dept. of Matls. Sci., Stockholm Sweden

Of late, a unusual new type of carrier induced magnetic long range ordering in dilute doped semiconductors has been demonstrated in a number of systems. Among these GaMnAs system has been the most studied material with the highest reported ferromagnetic ordering temperature of around 172K. Dietl et al predicted on a theoretical basis

that ZnO doped with Mn would be an above room temperature ferromagnetic semiconductor. ZnO is a well known electronic material with many desirable features and multivalued properties, with a wide range of applications. However, most of the studies reported on Mn doped ZnO until recently have been misleading, if not unsuccessful, mainly because of the processing conditions. We have for the first time reported in Nature materials (Oct issue 2003) methods to tailor the homogeneous ferromagnetic property into Mn doped ZnO. However, the saturation magnetization Ms and the moment per Mn atom obtained in the actual measurements is much smaller than the expected theoretical value. In order to enhance the carrier concentration and obtain higher Ms values we have co-doped ZnO:Mn with Cu to find that the magnetization can be enhanced by over 150% with as low a concentration as 4 at% Cu. Electronic structures of these materials investigated by using XES, XAS and RIXS confirm that Cu doping increases the hole concentration while maintaining the basic electronic structure of the matrix unchanged. In view of these results we doped ZnO with Cu alone and found it to be ferromagnetic with Tc above 450C. Such a phenomenon observed in ZnO:Cu which contains no magnetic transition elements at all is rather unusual. Some of these studies on new novel oxides, sulphides, and phosphides will be presented. Room temperature ferromagnetism in dilute doped semiconductors containing no magnetic elements is indeed a new challenging phenomenon. * with Amita Gupta, Pamanand Sharma, Jinghua GuO, R. Ahuja and B. Johansson.

11:30 AM

Epitaxial Growth of High Performance Superconducting Wires on Rolling-Assisted Biaxially-Textured Substrates (RABiTS): *Amit Goyal*[1]; [1]Oak Ridge National Laboratory, PO 2008, MS 6116, Oak Ridge, TN 37831-6116 USA

This talk will summarize the development of long lengths of flexible, single-crystal-like wires of YBa2Cu3Ox (YBCO) superconductor via epitaxial growth of the superconductor on Rolling-assisted-biaxially-textured-substrates (RABiTS). The RABiTS technique employs simple and industrially scalable thermomechanical processing routes to obtain long lengths of near single-crystal-like, cube-textured substrates. Epitaxial buffer layers of various cubic oxides (of rock salt, fluorite, perovskite and pyrochlore crystal structures) are then deposited in a reel-to-reel configuration using web-coating employing electron-beam evaporation, sputtering or solution routes. Epitaxial YBCO is then deposited using either ex-situ or in-situ methods to form a single-crystal-like superconducting wire. Such superconducting wires have critical current densities in long lengths of 3 Million Amps/cm2 at 77K in self-field. Incorporation on self-aligned nano-particles of second phases within the YBCO layer further enhances the in-field performance of the superconducting wires. This talk will provide a status of the RABiTS method of fabricating superconducting wire and will outline the path towards eventual realization of large-scale, bulk applications of superconductors.

12:00 PM

Half-Metallic Oxides for Spintronic Applications: *Arunava Gupta*[1]; [1]University of Alabama, MINT Ctr., Dept. of Chmst., Cheml. & Biological Engrg., Tuscaloosa, AL 35487 USA

The emerging field of spintronics aims to exploit the electron spin, in addition to its charge, to create a new class of devices that scale down to much smaller dimensions with possibly added functionalities. Of particular relevance are magnetic tunnel junctions (MTJs), consisting of two ferromagnetic electrodes separated by an insulating barrier, that exhibit large tunneling magnetoresistance (TMR) at relatively low fields. The MTJs are promising for a host of applications including magnetic memory (MRAM), sensors and storage devices. Most of the studies on MTJs have thus far focused on using transition metal ferromagnets (Fe, Ni, Co) and their alloys - typically with spin polarization values less than 50% - where the maximum observed TMR is limited to about 40-50% at room temperature. There is obvious interest in further enhancing the TMR by using materials with a higher degree of spin polarization. Half-metallic systems, which contain a gap in one spin band at the Fermi level and no gap in the other spin band, are expected to have a spin polarization value approaching 100%. Band structure calculations have shown that a number of magnetic oxide materials, such as the mixed-valence manganites (La1-xAxMnO3, A=Ba, Sr, or Ca), magnetite (Fe3O4), and chromium dioxide (CrO2), are half-metallic. With appropriate choice of barrier materials, MTJs have been fabricated using these oxides that exhibit reproducible tunneling characteristics with very high TMR in some cases. However, the enhancement has thus far been limited to low temperatures. I will present an overview of the work in this field over the past few years, including the growth and properties of thin films of this unique class of materials.

General Abstract Session: Mechanical Behavior—Dynamic Loading

Sponsored by: TMS

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Wednesday AM Room: 2020
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Michael J. Mills, Ohio State University, Matls. Sci. & Engrg., Columbus, OH 43210 USA

8:30 AM

The Ultrasonic Fatigue Behavior of the Ti 6Al-2Sn-4Zr-6Mo: C. J. Szczepanski[1]; A. Shyam[1]; S. K. Jha[2]; J. M. Larsen[3]; C. J. Torbet[1]; S. Johnson[1]; *J. W. Jones*[1]; [1]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; [2]Universal Technology Corporation, Dayton, OH 45432 USA; [3]AFRL/MLLMN Wright Patterson AFB, Matls. & Mfg. Direct., Dayton, OH 45433 USA

A rapid method for characterizing the fatigue crack growth behavior in the short crack regime has been developed and applied to study the fatigue behavior alpha + beta-processed Ti-6246. The measurement of very low fatigue crack growth rates was accomplished by tracking crack initiation and growth from small surface micronotches produced by femtosecond pulsed laser machining. An ultrasonic fatigue apparatus, operating at 20 kHz, was used to grow cracks at room temperature at load ratio of R=0.05. Crack length was acquired in real time using a telescopic digital image acquisition system. The role of microstructural, including micronotch severity, on crack growth was examined and a model for short crack growth in this titanium alloy was developed. This model is used to examine the importance of crack initiation in the very long life fatigue behavior of Ti-6246.

9:00 AM

Characterization of In Vitro Fatigue Crack Growth in Nitinol for Endovascular Stent Applications: *Jessica Stankiewicz*[1]; Scott W. Robertson[1]; Xiao-Yan Gong[2]; Robert O. Ritchie[1]; [1]University of California & Lawrence Berkeley National Laboratory, Matls. Scis. Div., MS 66-202, Berkeley, CA 94709 USA; [2]Nitinol Devices & Components, 47533 Westinghouse Dr., Fremont, CA 94539 USA

The unique mechanical properties, particularly superelasticity, of Nitinol, coupled with its biocompatibility, have made it a preferred choice for many biomedical devices. Prominent among these applications are endovascular stents which have features generally on the order of millimeters or smaller. It is important that the fracture and fatigue properties of Nitinol are characterized on such a size-scale in realistic product forms in order to minimize the chance of in vivo failures. This paper documents research in the area of Nitinol fatigue using a damage-tolerant approach to characterize crack growth. Critical to the validity of testing was conducting these experiments using Nitinol samples with similar texture, heat treatment, and stress history as fully-processed stents. Using thin-walled Nitinol tubes that are the starting material for such stents, this research examines the in vitro behavior of compact-tension specimens that have been cut and flattened from tubes for crack growth studies.

9:30 AM

The Role of Microstructure in Fatigue Life of Colony Ti-6Al-4V: *Alison K. Polasik*[1]; M. J. Mills[1]; J. M. Larsen[2]; H. L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMN, Wright-Patterson AFB, OH 45433-7817 USA

A necessary step towards the accelerated insertion of titanium alloys in aircraft engine components is the development of microstructurally-based models of fatigue parameters. Within this goal, the focus of this work is to develop a more detailed understanding of the effect of microstructural features on the fatigue behavior of α/β titanium alloys. Fatigue lifetime tests were conducted for two populations of β-processed Ti-6Al-4V that were subjected to different heat treatments. High resolution SEM imaging was coupled with novel stereological procedures capable of extracting 3-dimensional microstructural information from 2-dimensional micrographs of Ti alloys in

order to quantify key microstructural features in the two sample populations. These numerical values, along with the test conditions, were then used as inputs in a fuzzy logic model to isolate the microstructural features and determine their impact on lifetime, as well as to predict fatigue lifetime based on microstructural parameters were developed. Additionally, this analysis allowed for targeted application of extensive characterization techniques, including OIM, TEM and site-specific TEM foil extraction using the Focused Ion Beam. This research has been supported in part by the US Air Force under the STW-21 Program and by NSF under the Fellowship program.

10:00 AM
High Strain Rate Loading of nc Ni: Experiments and Simulation: *Eduardo M. Bringa*[1]; Y. M. Wang[1]; J. A. Caro[1]; M. Victoria[1]; A. M. Hodge[1]; J. McNaney[1]; B. Torralva[1]; B. A. Remington[1]; R. Smith[1]; C. A. Schuh[2]; B. Wu[2]; H. Van Swygenhoven[3]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; [2]Massachusetts Institute of Technology, Cambridge, MA 02139 USA; [3]PSI, Villigen CH-5232 Switzerland

We present experiments and simulations of laser-induced deformation of eletrodeposited nanocrystalline (nc) Ni at strain rates larger than 10^6/s. These strain rates make possible a direct comparison between experiments and molecular dynamics simulations. TEM of material recovered after loading shows that nanograins survive the loading, with some grain growth and grain refinement, and some grains containing large dislocation densities. Nano-indentation of recovered samples gives changes in hardness with complex pressure dependence due to the interplay between: a) large dislocation densities generated during loading, which increase hardness and b) grain growth due to thermal processing, which decreases hardness. Atomistic simulations show high dislocation densities created during loading, even for grains smaller than 10 nm, with partial recovery of the dislocations after unloading and thermal recovery, in agreement with the experimental findings. Work was performed under the auspices of the U.S. DoE by UC, LLNL, contract No.W-7405-Eng-48, LDRD 04-ERD-021.

10:30 AM Break

10:45 AM
On the Failure of NiAl Bicrystals During Laser-Induced Shock Compression: *Eric Loomis*[1]; Pedro Peralta[2]; Damian Swift[3]; Ken McClellan[4]; [1]Arizona State University, MAE, Tempe, AZ 85281 USA; [2]Arizona State University, Dept. of Mechl. & Aeros. Engrg., Tempe, AZ 85287-6106 USA; [3]Los Alamos National Laboratory, Physics, Los Alamos, NM 87545 USA; [4]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

Thin NiAl bicrystals 5 mm in diameter and 150 to 350 microns thick were tested under laser-induced shock compression to evaluate the material behavior and the effect of localized strain at the grain boundary on the failure of these specimens. Circular NiAl bicrystal samples with random misorientation were grown and prepared for shock compression at pressures below 10 GPa. Transmission electron microscopy was performed in the bulk of one grain following laser-shock testing and showed that plastic deformation occurred in a periodic fashion through nucleation of dislocation clusters at the shock front. Cracking on the free surface of the samples revealed a clear grain boundary affected zone due to scattering of the shock wave and variations in wave speed across the inclined boundary. The damage locations in these samples correlated well to the regions in which the transmitted waves impinged on the free surface as predicted by elastic scattering models.

11:15 AM
Shock and Recovery of Polytetrafluoroethylene (Ptfe) Above and Below the Phase II to Phase III Transition: *Eric Nathaniel Brown*[1]; Philip J. Rae[2]; Dana M. Dattelbaum[3]; David L. Robbins[3]; G. Rusty Gray[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., TA-35, Bldg. 455, DCDP 01S, MS E544, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Dynamic Experimentation, MS P952, Los Alamos, NM 87545 USA

Polytetrafluoroethylene (PTFE) is semi-crystalline in nature with its linear chains forming complicated temperature and pressure dependent phases. Due to its extremely low coefficient of friction, outstanding resistance to corrosion, and range of thermal stability, applications of PTFE include surgical implants, aerospace components, chemical barriers, and structures designed for dynamic large-scale plasticity excursions. Experimental studies on pressure-induced phase transitions using shock-loading techniques and the resulting changes in crystalline structure are presented. Disks of pedigreed PTFE 7C have been shock loaded in momentum trapped assemblies using a 80 mm gas launcher, and recovered in a density graded polymer network. Experiments were performed with impact pressures from 0.4 to 0.85 GPa to investigate the material response above and below the phase II to phase III crystalline transition. Changes in crystalline structure of the recovered materials were quantified using dynamic scanning calorimetry (DSC) and density.

11:45 AM
Microstructure and Mechanical Properties of Investment Cast Ti-6Al-4V: *Kevin L. Klug*[1]; Ibrahim Ucok[1]; Lawrence S. Kramer[1]; Mehmet N. Gungor[1]; Hao Dong[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

The macrostructures, microstructures and mechanical properties of investment cast Ti-6Al-4V shaped components have been studied. The macrostructure investigations were focused on the size distribution of prior-β grains as a function of casting thickness; the microstructure studies were concentrated on α phase and β phase morphology and distribution. Similar studies were conducted on plates cast from lower-cost, alternative Ti-6Al-4V electrodes containing high levels of machining chips and forging scrap. The metallographic analyses of both the components and plates were compared with tensile, fatigue and impact test properties generated for each product form. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the U.S. Navy Manufacturing Technology Program.

12:15 PM
Mechanically-Induced Surface Deformation and Associated Stress Whitening in Clay-Containing Nanocomposites: *Rohith Hadal*[1]; Harish Nathani[1]; Shankar Shanmugam[1]; Devesh K. Misra[1]; [1]University of Louisiana, Matls. Sci. & Engrg. Grp., Cheml. Engrg. Dept., PO Box 44130, Lafayette, LA 70504-4130 USA

The lateral resolution of scanning electron microscopy and vertical resolution of atomic force microscopy is combined to examine the surface damage behavior and susceptibility to mechanical surface damage in neat and clay-containing polymer nanocomposites. The surface damage in neat polymers is characterized by "psiloma-type" morphology indicative of compressive plastic deformation and quasi-periodic cracking. While in clay-containing nanocomposites, "ironing," which is a less severe surface damage mechanism was dominant. Also, clay-reinforced nanocomposites experience significantly reduced stress whitening, and is characterized by lower gray level in the plastically deformed surface damage region. This behavior is attributed to the effective reinforcement by clay particles that act in concert increasing the tensile modulus of the composite and restricts plastic deformation of the polymer matrix.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Thermal Fatigue and Reliability of Lead-Free Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Wednesday AM Room: 3014
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: K. N. Subramanian, Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1266 USA; Srinivas Chada, Jabil Circuit Inc., FAR Lab/Advd. Mfg. Tech., St. Petersburg, FL 33716 USA

8:30 AM Invited
Supporting Studies to Understand TMF Behavior of Eutectic Sn-3.5Ag Solder Joints: *K. N. Subramanian*[1]; Hongjoo Rhee[1];

[1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., EB2527, E. Lansing, MI 48824-1226 USA

The reliability of the solder joints depends on the material response to imposed service conditions. In order to gain a better understanding of such processes under thermal excursions encountered in service, supporting studies were carried out. Such tests consisted of cyclic shear straining with associated stress relaxation at the shear strain extremes during stress relaxation of pre-strained single shear-lap solder joints at various temperatures. These tests evaluated the roles of pre-strain and pre-strain rate imposed prior to cyclic shear straining, cyclic shear strain amplitude, cyclic shear strain-rate, testing temperature, etc. Acknowledgement: Work supported by the National Science Foundation under grant NSF DMR-0081796.

9:00 AM

Effect of Thermal Cycling on the Electrical Conductivity of Sn-Based Solders: J. G. Lee[1]; N. Meyer[2]; T. Hogan[2]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]Michigan State University, Dept. of Electl. & Computer Engrg., E. Lansing, MI 48824-1226 USA

Thermal excursions experienced during service result in highly inhomogeneous damage accumulation in electronic solder joints affecting their structural and electrical properties. These properties significantly drop within the first few hundred thermal cycles without any visible surface manifestation of damage. However, surface damage becomes apparent only when the deterioration in these properties stabilized. Electrical conductivity measurements were made with very small probes to quantify the local changes in the electrical conductivity of the thermally cycled Sn-based solder joints. Residual shear strengths and residual electrical conductivities of solder joints that have undergone identical thermal cycles will be compared to gain some insight for reliability predictions. Acknowledgement: Work supported by the National Science Foundation under grant NSF DMR-0081796 and NSF DMI-0339898.

9:20 AM

Evaluation of Solder Joint Reliability in Flip Chip Packages During Accelerated Testing: *Jong-Woong Kim*[1]; Seung-Boo Jung[1]; Dae-Gon Kim[1]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Cheoncheon-dong, Jangan-gu, Suwon 440-746 S. Korea

Flip chip soldering technologies are being popular as one of the chip level interconnections to meet a demand for the higher density packages. Solder must act as both the conduits of electrical interconnections and the mechanical support to hold the chip in position on the substrate. When the flip chips are in operation, temperature cycles generate thermo-mechanical fatigue reacting with a substrate and a chip that have different coefficients of thermal expansion. Therefore, a study of thermo-mechanical fatigue behavior and the failure mechanism of the solder joints is very important for guaranteeing the reliability of the flip chip packages. In the present study, the thermal shock test of the flip chip package and a relevant three-dimensional thermo-mechanical finite element analysis using ANSYS were performed. A representative lead-free Sn-3.0Ag-0.5Cu solder was used, and the packages were tested according to JEDEC test method A106B. Finally, the experimental and computational results were related.

9:40 AM

Examination of Failure Mechanisms in Pb Free Solder Joints: *Eric J. Cotts*[1]; Lawrence P. Lehman[1]; Lubov Zavalij[1]; Yan Xing[1]; Ju Wang[1]; Peter Borgesen[2]; [1]Binghamton University SUNY, Physics & Matls. Sci., PO Box 6000, Sci. 2, Binghamton, NY 13902-6000 USA; [2]Universal Instruments Corporation, Rsch., Kirkwood, NY USA

The reliabilities of ball grid array (BGA), chip scale packaging (CSP), and flip chip components with SnAgCu solder joints were examined as a function of long term thermal cycling. All components were assembled on either OSP-coated copper, or electroless-nickel-immersion-gold (ENIG), coated pads on high-Tg FR-4 boards. The samples were subjected to air-to-air thermal cycling between 0C and 100C. The 20 minute cycle had 5 minute ramps and 5 minute holds at the two temperatures. Assemblies were removed for cross sectioning and microstructural characterization at various stages of cycling. Thus damage accumulation was examined using optical and electron microscopy techniques. Polarized light microscopy provided delineation of Sn grains, while electron microscopy with EDS provided compositional analysis. The variation in the thermomechanical loads with solder joint location across an area array allowed detailed study of a range of temperature-damage combinations.

10:00 AM

A Study on Reliability Test of WLCSP Lead-Free Solder Joints: *Huann-Wu Chiang*[1]; Jun-Yuan Chen[1]; Jeffrey C.B. Lee[2]; S. W. Li[2]; [1]I-SHOU University, Dept. of Matls. Sci. & Engrg., #1, Sec.1, Hsueh-Cheng Rd., Ta-Hsu Hsiung, Kaohsiung County, Taiwan 84008 Taiwan; [2]Advanced Semiconductor Engineering, Inc., Engrg. Ctr., 26, Chin 3rd Rd., Nantze Export Procg. Zone, Kaohsiung, Taiwan 811 Taiwan

The interfacial reactions of solder joints between Sn-Ag-Cu solder ball and Sn-Ag-Cu pre-soldered paste will investigated in wafer level CSP package. After appropriate SMT reflow process on PCB with Cu-OSP and Cu-NiAu surface finish, samples will be subjected to either 150°C HTS 1000 hours aging or -40°C -150°C TCT reliability test. Sequentially, the cross-section analysis is scrutinized by SEM/EDX and EPMA to observe metallurgical evolution in the interface and solder buck itself. Pull and shear tests will also be performed on samples. The relationship between the interfacial microstructure and the joint strength will then be analyzed and discussed.

10:20 AM Break

10:30 AM

Nucleation and Propagation of Fatigue Damage in Near-Eutectic Sn-Ag-Cu Alloy: *Tia-Marje K. Korhonen*[1]; Donald W. Henderson[2]; Matt A. Korhonen[1]; [1]Cornell University, Dept. of Matls. Sci. & Engrg., 328 Bard Hall, Ithaca, NY 14850 USA; [2]IBM Corporation, Hopewell Junction 12533 USA

It has been shown that when near-eutectic SAC alloys are used to make BGA solder joints, the grain size is very large, with typically 1 to 12 grains per BGA joint. Often the BGA joints are single crystals. During thermomechanical cycling, the solder joint fatigue process is characterized with recrystallization of the Sn grains, resulting in a smaller grain size in the deformed areas. Grain boundary sliding and increased grain boundary damage then results in intergranular crack initiation and propagation along the recrystallized Sn grain boundaries. In this work, fatigue tests were used to study the initial stages of deformation in Sn-Ag-Cu alloy samples. To separate the solder properties from the constraints introduced by the substrate, the tests were done to free-standing solder specimens, instead of solder joints. The test samples were cast dog-bone specimens that have a cross section of 1mm, which corresponds to the typical solder joint diameter in ball grid arrays. The solder was heated to 245 degrees, held there for ten minutes and then cast and cooled at 1 degree/second cooling rate. Mechanical cycling was performed isothermally at several temperatures, up to 125 C. Typical test conditions were 0.5% strain and 30 minute cycles. Optical microscopy, SEM and electron back-scatter diffraction were used to study the microstructures of the samples before and after fatigue testing.

10:50 AM

Thermal Fatigue Behavior of Sn-Bi Solder Joints: *Mark A. Palmer*[1]; Samir Nashef[1]; [1]Kettering University, IMEB, 1700 W. Third Ave., Flint, MI 48504 USA

A new thermal fatigue test apparatus has been designed to assess the thermal fatigue resistance of solder joints prepared with Sn-30w/oBi, eutectic Sn-Bi, and Sn-70w/oBi alloys. The design of this new apparatus will be discussed. The thermal fatigue behavior will be correlated with the microstructural evolution of the bulk material and the solder joint. Thermal fatigue resistance will be correlated with the temperature dependent strength, temperature dependent fatigue resistance, creep resistance of annealed solder joints and alloys. Funding from the National Science Foundation CMS-0140605 is gratefully acknowledged.

11:10 AM

The Role of Cu Content on Compound Formation Near Chip and Substrate Side for the Flip Chip Sn-3.0Ag-xCu Solder Bump After Aging and Thermal Cycling: *Guh-Yaw Jang*[1]; Li-Yin Hsiao[1]; Jenq-Gong Duh[1]; Hideyuki Takahashi[2]; Szu-Wei Lu[3]; Jen-Chuan Chen[4]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec. 2 Kuang-Fu Rd., Hsinchu 300 Taiwan; [2]JEOL Ltd., Application & Rsch. Ctr., Tokyo Japan; [3]TSMC Ltd., Flip-Chip Engrg. Dept., 121, Park Ave.3, Hsinchu Sci. Park, Hsinchu 300 Taiwan; [4]ASE, Flip-Chip Operation, 550, Chung-Hwa Rd., Sect. 1, Chung-Li 320 Taiwan

The Sn-Ag-Cu solder is one of the promising candidates to replace the conventional Sn-Pb solder. Interfacial reaction for the flip chip solder bump of Sn-2.3Ag/Sn-3.0Ag-0.5Cu and Sn-3.0Ag-xCu/Sn-3.0Ag-0.5Cu (x = 0.5 and 1.5) combination structure was investigated after aging at 150°C and thermal cycling between -55°C and 125°C. The under bump metallization for the Sn-2.3Ag and Sn-3.0Ag-xCu solders on the chip side was SiO2/Cu/Al/Ni, while the bond pad for Sn-3.0Ag-0.5Cu solder on the plastic substrate side was Cu/electroless Ni/Au. In the Sn-3.0Ag-xCu joints after thermal cycling for 1000 cycles, the Ni layer at the chip side was consumed completely and reacted with Sn and Cu atoms to form $(Cu,Ni)_6Sn_5$ intermetallic compound (IMC). At the plastic substrate side, three reaction compound, $(Cu,Ni)_6Sn_5$, $(Ni,Cu)_3Sn_4$ and Ni_3P, was observed between the solder and the EN

layer. For the Sn-2.3Ag joint, nearly half Ni layer was reacted to form $(Ni,Cu)_3Sn_4$ IMC at the chip side, and $(Ni,Cu)_3Sn_4$ and Ni_3P IMCs were revealed at the solder/EN interface at plastic substrate side. The interfacial reaction in the solder joints could be related to the Cu concentration in the solder joint. In addition, the detailed microstructure evolution of solder joints after aging and thermal cycling was also discussed in this study.

11:30 AM
The Effect of Pb Contamination on Sn-Ag-Cu Solder Joint Reliability: *Masayoshi Date*[1]; Tatsuya Shoji[1]; Masaru Fujiyoshi[1]; Koji Sato[2]; [1]Hitachi Metals, Ltd., Metallurgl. Rsch. Lab., Yasugi-cho 2107-2, Yasugi-shi, Shimane 692-8601 Japan; [2]Hitachi Metals, Ltd., Yasugi Works

The effect of Pb contamination on joint reliability of Sn-3Ag-0.5Cu Pb-free solder was evaluated. In the first reflow, the Sn-Ag-Cu solder balls were bonded to electroless Au/Ni(P) pads of a chip scale package (CSP) by using rosin activated flux. In the following reflow, the package was mounted on a substrate, whose pads were electroless Au/Ni(P) or an organic solder preservative (OSP) coated Cu pads, by using either the Sn-Ag-Cu or eutectic Sn-Pb solder paste. Subsequently, a thermal cycle test was conducted ranging from -55 to 125 °C. The microstructure of the joints and the composition of interfacial compounds were dependent deeply on solder compositions and bond-pads. The joints composed of the SnAgCu balls and the SnPb paste, whose bond-pads of the substrate were Au/Ni(P), were liable to fracture at earlier cycles than others, caused by crack propagation along the bond interface on the substrate side.

11:50 AM
Orientation Imaging Microscopy Studies on Thermomechanically Cycled Lead-Free Sn-3.5Ag Solder Joints: Adwait U. Telang[1]; K. N. Subramanian[1]; Thomas R. Bieler[1]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., 2527 Eng Bldg., E. Lansing, MI 48824 USA

Single shear lap and double shear lap lead-free solder joints were made using Sn-3.5Ag solder having a 1 mm^2 joint area and solder thickness of about 100μm. Different shear strain states were induced in these joints while undergoing TMF cycling due to the differences in the geometry of the specimens. The microstructural evolution and the damage that developed at the end of 1000 cycles was studied in all joints using Orientation Imaging Microscopy (OIM) and scanning electron microscopy (SEM). Surface topography in the form of grain boundary sliding, ledge development, and further decohesion in the tin matrix that was seen after 1000 TMF cycles was correlated with the crystal orientations and misorientations present in the joint, and the grain boundary character. Slip activity that could cause such surface topography is analyzed and discussed. The effect of different strain histories on evolution of surface damage and microstructure will be discussed and probable cause for such surface damage accumulation will be identified.

WEDNESDAY AM

Magnesium Technology 2005: Magnesium Alloy Processing

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Wednesday AM Room: 2004
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Eric Nyberg, Pacific Northwest National Laboratory, Richland, WA 99352 USA; Menachem S. Pbamberger, Israel Institute of Technology, Technion Dept. of Matls. Engrg., Haifa 32000 Israel

8:30 AM
Comparison of As-Cast and Plasma Deposited Commercial Magnesium Alloys: Dirk Martin Seeger[1]; *Carsten Blawert*[1]; Wolfgang Dietzel[1]; Karl Ulrich Kainer[1]; Yvonne Bohne[2]; Stephan Mändl[2]; Bernd Rauschenbach[2]; [1]GKSS Forschungszentrum Geesthacht GmbH, Ctr. for Mg Tech., Max-Planck-Str. 1, Geesthacht 21502 Germany; [2]Leibniz-Institut für Oberflächenmodifizierung, Permoserstr. 15, Leipzig 04318 Germany

Ion beam sputtering is used to produce magnesium alloy coatings of AM50, AZ91 and AE42 alloys on silicon and magnesium substrates. Due to the specific process conditions, a very fine microstructure will result. We will compare this microstructure with the microstructure of the original commercial AM50, AZ91 and AE42 target materials. The aim is to study the effect of the microstructure on the corrosion properties. Polarisation technique will be used to study the corrosion behaviour of the coatings and the original materials.

8:50 AM
Segregations of Aluminum After Solidification: AZ91D MG-AL-ZN Plate Produced by Die Casting and Gravity Casting: *Chi-Yuan Cho*[1]; Jun-Yen Uan[1]; Huey-Jiuan Lin[2]; Te-Chang Tsai[3]; [1]National Chung Hsing University, Dept. of Matls. Engrg., 250 Kuo Kuang Rd., Taichung 402 Taiwan; [2]National United University, Dept. of Matls. Sci. & Engrg., 1 Lien Da Rd., Kung-Ching Li, Miao Li 360 Taiwan; [3]Hsiuping Institute of Technology, Dept. of Mechl. Engrg., 11Gungye Rd., Dali City, Taichung 412 Taiwan

Segregation of aluminum element in AZ91D magnesium plates produced by die casting and gravity casting was investigated. The aluminum at cast surface have higher concentration than that examined from the interior region, either by die casting or by gravity casting process. The concentration of aluminum in the die cast plate show an increasing tendency along the melt filling direction: from the gating system to the final position the melt can reach. However, for gravity casting plate the alloy elements prefer to accumulate at the position between chill block and the gating system. Before the molten metal solidified, the positions which con-tain high fraction of solid will lead to a relative large amount of alloy element segregating there. As a result, the cast product will have some specific locations where the chemical compositions are lower than the ASTM B-94 AZ91D standard.

9:10 AM
Effects of Grain Refinement and Melt Filtration on the Mechanical Properties of Sand and Permanent Mold Cast Magnesium AZ91D Alloy: *Daryoush Emadi*[1]; Jim Thomson[1]; Kumar Sadayappan[1]; Mahi Sahoo[1]; [1]CANMET, Matls. Tech. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

Magnesium finds applications in a multitude of automotive, military and commercial applications because of its lightweight, high strength/weight ratio, high-castability and excellent damping capacity and machinability. Recognition by the auto industry of weight reduction, an increase in energy cost together with government regulations controlling vehicle design has established an environment for increased magnesium use. At present, most of the automotive components in Mg alloys are produced by high-pressure die-casting. However, to increase Mg usage in components, other casting processes such as sand and permanent mold casting should be evaluated. CANMET-Materials Technology Laboratory has taken this approach and initiated a research program to address critical issues relating to high integrity sand and permanent mold casting. The effects of mold design, grain refinement and melt filtration on mechanical properties of sand and permanent mold cast test bars were investigated. The effect of machining on propert ies was also evaluated. The experimental results are presented and discussed in this paper.

9:30 AM
Advances in Technology of Processing Semisolid Magnesium Alloys: *Frank Czerwinski*[1]; [1]Husky Injection Molding Systems Ltd, Dvlp. Engrg., 560 Queen St. S., Bolton, Ontario L7E 5S5 Canada

Since its discovery over thirty years ago, semisolid processing is mainly applied to alloys with relatively low melting temperatures, particularly aluminum. Although historically an interest in magnesium reaches as early as 1974, compared with aluminum, investigations of semisolid magnesium alloys are scarce. This paper presents the key semisolid technologies, available in today's industry, based on thixo- and rheo-routes with particular attention being paid to emerging techniques of injection molding. The major requirements imposed on potential alloys and their transformations during processing are described. For selected alloys the correlation between chemistry and microstructure is analyzed the importance of preheating temperature in semi-solid range is emphasized. Examples of products, manufactured at present worldwide are given.

9:50 AM
Influence of SiC Grain Refiner Additions on the Globular Structure of New Rheocasting Mg Alloys: *Helmut Kaufmann*[1]; Mark Easton[2]; Werner Fragner[1]; [1]ARC Leichtmetallkompetenzzentrum Ranshofen GmbH, PO Box 26, Ranshofen 5282 Austria; [2]CRC for Cast Metals Manufacturing, Australia

The shape of primary phase solid particles, their contiguity and their size are the three major microstructural parameters influencing the castability of semi-solid slurries into high quality magnesium prod-

ucts. New Rheocasting (NRC) is a slurry-on-demand semi-solid casting process where a slightly super heated melt is quenched at the wall of a steel cup and subsequently cooled slowly to achieve globular growth of the primary phase. Fine grain size is extremely important for the overall performance of magnesium alloys in light weight applications. The grain size determines the mechanical properties at room temperature and elevated temperature. In the classical NRC process the grain size is determined by the amount of solidification nuclei formed at the cup wall and their subsequent growth. Typically, particle sizes of the primary phase in the range of 80 to 120 ìm can be reached. In order to achieve even finer particle sizes in the as cast structure, heterogeneous grain refiner particles were added to the melt. Grain refining of aluminium containing magnesium alloys is still an unsolved problem. A newly developed method for preparation and addition of SiC grain refining particles is applied to conventional Mg melts and tested in New Rheocasting. This paper will show the effects of wall mechanisms and heterogeneous nucleation on SiC grain refiner particles on the evolution of the primary phase in NRC semi-solid slurries, as well as their influence on the resulting properties of Mg castings.

10:10 AM Break

10:25 AM
Effects of Processing Parameters on Microstructure of Continuous Cast Magnesium Billets: *Kwang Seon Shin*[1]; Hwa Chul Jung[1]; [1]Seoul National University, Sch. of Matls. Sci. & Engrg., San 56-1 Shinrim-dong Kwanak-gu, Seoul 151-742 Korea

With many advantages derived from the process, semi-solid processing (SSP) is an emerging technology for near net-shape production of engineering components. Although there has been a significant progress in semi-solid processing of Al alloys, very limited information is available on semi-solid processing of Mg alloys. A continuous casting process has been successfully utilized in mass production of the billets of steel, copper, aluminum, etc. In this study, a continuous casting process has been developed for the production of magnesium billets for subsequent semi-solid processing. The processing parameters including melt temperature, stirring force and the withdrawal rate of the solidified billet have been carefully controlled for the successful production of magnesium billets with non-dendritic microstructure. In the present study, the effects of these processing parameters on microstructure of the continuous cast magnesium billets were investigated.

10:45 AM
High Pressure Die Casting Process of Advanced Mg Alloys: *Nir Moscovitch*[1]; Dan Eliezer[2]; Eli Aghion[2]; [1]Dead Sea Magnesium, Rsch. Div., PO Box 1195, Beer Sheva 84111 Israel; [2]Ben-Gurion University, Matls. Engrg., PO Box 653, Beer Sheva 84105 Israel

High pressure die casting (HPDC) is the dominant process technology for the mass production of Magnesium components with complex configuration having typical thin to medium wall thickness. The growing use of die cast Magnesium alloys for the automotive industry, particularly for the production of drive train applications has lead to the development of advanced die casting alloys with improved creep resistance, namely MRI 153M and MRI 230D. MRI 153M alloy can be used in applications with service temperature up to 150°C, while MRI 230D can be used at temperatures up to 190°C. Both alloys have excellent creep resistance at their designated operation temperature. This creep performance is combined with good castability, high strength and superior corrosion behavior. The present paper aims at introducing semi-empiric correlations between HPDC parameters, solidification characteristics and the properties of the die cast component. The paper also includes a systematic evaluation of the heat transfer and energy balance during the die casting process. Adequate implementation of this data can lead to reliable part design and high quality die cast components.

11:05 AM
Numerical Simulations and Experimental Study of Hot Core Distortion Phenomenon in Magnesium Casting: *Sayavur Ispandiyar Bakhtiyarov*[1]; Johnathon Capps[1]; Ruel Overfelt[1]; David Weiss[2]; [1]Auburn University, Mechl. Engrg., 202 Ross Hall, Auburn, AL 36849-5341 USA; [2]ECK Industries, Inc., Manitowoc, WI 54221-0967 USA

This paper presents the results of experimental and numerical studies of hot distortion phenomenon in the phenolic urethane cold box systems during magnesium casting. Dual Pushrod Dilatometer has been used to measure a thermal expansion/contraction of phenolic urethane cold box sand core specimens at temperature range from 200°C to 600°C. The high temperature tensile tests showed that the tensile strength of the phenolic urethane cold box sand cores is significantly affected by the bench life, temperature and binders level. High temperature hot distortion furnace tests on cylindrical cores showed that some coatings increase the temperature limit when distortion starts, but can't prevent it. The hot distortion test during magnesium castings showed that regardless of the application of coating, the type of coating, and anti-veining additives, all cores with density less than the density of the molten metal (magnesium alloy) were significantly distorted. Numerical simulations of the liquid metal flow around the cylindrical sand core and analysis of dynamic forces acting on the core during fill process showed that a buoyancy force is the major contributor to the hot distortion. It is concluded that the one of the solutions in preventing the hot distortion of sand cores is optimizing their weigh, which will balance the buoyancy force and will bring the resultant force to the minimum. The hot distortion test castings using optimized sand cores (both coated and non-coated) with density almost equal to the density of the molten magnesium proved our predictions, and hot distortion has been prevented.

11:25 AM
The Effects of Process Parameters on the Porosity of Die-Cast AM50 Alloys: *Soon Gi Lee*[1]; Gautam R. Patel[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30332-0245 USA

Pressure die-cast magnesium alloys almost always contain both gas (air) and shrinkage porosity. Quantitative characterization of gas and shrinkage porosity is essential for understanding microstructure and properties relationships. In this contribution, the microstructures of high-pressure die cast AM 50 alloy have been quantitatively characterized to understand the effects of the process parameters such as melt temperature, in-coming liquid metal gate velocity, and intensification. A novel digital image analysis technique has been used to quantify the volume fraction and other geometric attributes of the gas (air) and shrinkage pores. Three-dimensional images of the porosity have been reconstructed using a combination of montage serial sectioning and digital image processing. The differences of the total porosity under different process conditions are mainly due to the number density of large gas pores. Therefore, the large pores, which constitute the upper 10% of the size distribution, have been investigated.

11:45 AM
Macro-Segregation in High-Pressure Die-Cast AM60 Alloy: *Soon Gi Lee*[1]; *Gautam R. Patel*[1]; *Arun M. Gokhale*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr. NW, Atlanta, GA 30332-0245 USA

Macro-segregation is known to occur in the vicinity of cast surfaces in Al and other nonferrous alloy castings under certain conditions and has been well documented, but it has not been investigated in high-pressure die-cast Mg-alloys. Solidification gives rise to transport of interdendritic liquid in order to feed shrinkage, which leads to a solute-rich region close to the cast surface commonly referred to as inverse segregation. Another macro-segregation close to the surface is exudation related to air gaps between the semi-solid shell and the mould caused by solidification contraction. This contribution reports the effects of process parameters such as melt temperature, in-coming liquid metal gate velocity, and intensification on the microstructural aspects of the macro-segregation in a high-pressure die-cast AM60 alloy. These correlations have been quantitatively characterized via quantitative microstructure characterization using stereology and image analysis techniques.

Materials Issues for Advanced Nuclear Systems: Materials for Gen IV and Space Nuclear Systems

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Robert J. Hanrahan, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA

Wednesday AM Room: 3012
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Robert J. Hanrahan, Los Alamos National Laboratory, ADWP, Los Alamos, NM 87544 USA; Raul B. Rebak, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

8:30 AM
Materials for Accelerator Driven Systems: *Abderrahim Almazouzi*[1]; [1]SCK.CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

High Chromium ferritic martensitic steels and austenitic steels that are forseen as candidates to build a spallation target as well as fuel cladds, were irradiated in BR2 reactor at 200°C. The post irradiation examination of the irradiated materials, that consist of slow rate tensile testing them in a liquid metal environnment, demonstates that they conserve their performance. The liquid metal embrittlement seems to be hindered by the selfhealing mechanism.

8:55 AM
The Mechanical Properties and Fracture Mechanisms of Wrought LCAC, TZM, and ODS Molybdenum Flat Products: *Brian V. Cockeram*[1]; [1]Bechtel Bettis Laboratory, PO Box 79, ZAP 05R/MT, W. Mifflin, PA 15122-0079 USA

Molybdenum alloys possess excellent strength and creep resistance at high temperatures, with measurable tensile ductility at low temperatures. These properties have attracted interest in the use of molybdenum alloys such as Low Carbon Arc Cast (LCAC) unalloyed molybdenum, Oxide Dispersion Strengthened (ODS) molybdenum, and TZM molybdenum for structural applications at high temperatures, such as vacuum furnace components, forging dies, glass molding, and advanced power systems. Although the tensile properties of molybdenum-base alloys have been well characterized, these alloys are poorly characterized with respect to fracture toughness, and the changes in the Ductile to Brittle Transition Temperature (DBTT) that result from the presence of a notch. Rolling of molybdenum into flat products has the advantage of producing a fine grained microstructure that improves the strength and lowers the DBTT. Detailed examinations of fracture surfaces are used to show that the fracture mechanism is generally the same for these three molybdenum-base alloys. Thin sheet toughening occurs during fracture by the splitting along grain boundaries and/or oxide boundaries to leave ligaments of grains that are stretched to failure with large amounts of plastic deformation. Thin sheet toughening is shown to occur in smooth tensile specimens and pre-cracked fracture toughness specimens. A sampling of tensile properties and fracture toughness data as a function of test temperature are used to identify the DBTT. The fracture mode at the DBTT changes from transgranular cleavage to the thin sheet fracture mode.

9:20 AM
Development of Ni-W Based Alloys for Future Nuclear Reactors: *Rafael Ferreira Cury*[1]; Thierry Auger[1]; Jean-Pierre Chevalier[1]; [1]CECM-CNRS, 15, rue Georges Urbain, Vitry cedex 94407 France

Whether for molten salt or high temperature gas cooled reactor designs, alloys are required to be oxidation and corrosion resistant,to have appropriate high temperature mechanical properties (yield stress and creep resistance) as well as acceptable room temperature toughness. For instance, Hastelloy N (a Ni-Cr-Mo based alloy) was selected for the Oak Ridge experimental molten salt reactor. The related Ni-Cr-W system may offer improvements over Hastelloy N, such as a lower activation and potentially better creep resistance, due to expected lower diffusion of W with respect to Mo, whilst maintaining similar corrosion and oxidation resistance. In an initial approach, the binary Ni-W alloys have been studied, with emphasis on alloy preparation. Using electron diffraction, the structural state (in terms of long and short range order) of the alloys as a function of composition will be presented. The role of short range order on hardening will also be discussed.

9:45 AM
Interpretation of Improved Creep Properties of a 9Cr-1Mo-Nb-V (T91) Steel by Grain Boundary Engineering: *Gaurav Gupta*[1]; Gary S. Was[2]; [1]University of Michigan, Nucl. Engrg. & Radiologl. Scis., 2940 Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Nucl. Engrg. & Radiologl. Scis., 2408 LEC, 1221 Beal Ave., Ann Arbor, MI 48109-2102 USA

Ferritic-martensitic alloys are expected to play a major role as structural components in Generation IV systems that operate in the temperature range 350-700°C and to doses up to 150 dpa. They exhibit improved irradiation stability and mechanical properties such as reduced swelling, high temperature creep resistance, and thermal shock resistance over austenitic steels but may suffer from grain boundary and/or matrix creep and loss of strength at higher temperatures, and unacceptably low toughness at lower temperatures. The objective of this work is to improve the creep resistance of T91 by grain boundary engineering. Coincident site lattice (CSL) enhancement strengthens the grain boundaries against sliding and deformation, thus improving the creep resistance. High temperature creep experiments in argon are conducted to assess the effectiveness of the CSL-enhanced microstructure on the creep rate. Experimental analysis shows that the CSL-enhanced condition results in a lower creep rate by a factor of 3-4 as-compared to as-received (A/R) condition for T91 at a temperature of 500°C and in the stress range of 200-225MPa by introducing an additional term for internal stress thus reducing the effective stress. Creep experiments are conducted on both A/R and CSL-enhanced T91 to quantify the stress and temperature effect of creep over the range 500-600°C and 150-250 MPa and to determine the mechanism by which Coincident Site Lattice Enhancement affects creep.

10:10 AM Break

10:20 AM
Mechanical Behavior of Neutron Irradiated High Cr Ferritic Martensitic Steels: *Abderrahim Almazouzi*[1]; [1]SCK,CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

High Chromium Ferritic Maetensitic steels are candidates for the traget spallation source and the fuel cladding tubes in the European demonstrator of the accelerator driven system. In this work, we will present new results obtained from the irradiation compaign that has been performed at BR2 within SPIRE project to assess the mechanical behaviour of several standards Fe-9,12%Cr steels. The irradiations have been performed at the lowest temperature limite where the irradiation induced hardening would be the highest (200°C) up to 4.5 dpa. The post irradiation analysis consisted of tensile, charpy and fracture thoughness testing. Discussion on the validity of master curve analysis of this class of materials will be proposed.

10:45 AM
Mechanical Properties and Cracking of High-Temperature Heat-Exchanger Materials: Ajit K. Roy[1]; *Narendra V. Kothapalli*[1]; Raghunanadan A. Karamcheti[1]; Lalitkumar B. Savalia[1]; [1]University of Nevada, Mechl. Engrg., 4505 Maryland Pkwy., Box 454009, Las Vegas, NV 89154 USA

The structural materials selected for high-temperature heat-exchanger applications are expected to withstand very severe operating conditions including elevated temperatures and aggressive chemical species during hydrogen generation using nuclear power source. Currently, three different cycles namely sulfur-iodine, calcium-bromine and high temperature electrolysis are being considered for hydrogen generation. Temperatures ranging from ambient to 1000ºC, and a very low pH (˜ 1) can influence the performance of structure materials such as Alloys C-276, C-22 and Waspaloy. This paper will present the results of stress corrosion cracking, hydrogen embrittlement and localized corrosion studies of all three alloys in related environments. The tensile properties at different testing temperatures will also be included. Further, the results of metallographic and fractographic evaluations of the tested specimens will be presented.

11:10 AM
Effects of Processing and Prolonged High Temperature Exposure on the Microstructure of Nb-1Zr-C Sheet: *Mehmet Uz*[1]; Robert H. Titran[2]; [1]Lafayette College, Ch. E. Dept., 262 AEC, Easton, PA 18042 USA; [2]NASA-GRC (Retired), Matls. Div., 15976 Walnut Creek Dr., Strongsville, OH 44149 USA

High temperature stability of the microstructure of Nb-1Zr sheets with 0.1 and 0.06 wt.%C was investigated as affected by processing and prolonged 1350-K exposure with and without applied load. Sheets were fabricated by cold rolling bars that were single-, double- or triple-extruded at 1900 K. Creep samples were double-annealed (DA: 1 h \@ 1755 K + 2 h \@ 1475 K) prior to testing at 1350 K for 10,000 - 34,500 h. The microstructures of the as-cast, extruded, rolled, DA and crept samples were characterized using various metallographic and analytical methods. The precipitates were rather coarse Nb2C initially, but transformed to finer (•< 1 mm) carbides of (Zr,Nb)C with each subsequent high temperature process. The grain size, and the relative amount and morphology of (Zr,Nb)C were affected by processing and C-content. However, the microstructures of all the crept samples were similar with (Zr,Nb)C distributed throughout the matrix indicating that prolonged exposure to 1350 K gave rise to complete transformation of Nb2C to (Zr,Nb)C regardless of the processing history. These and other observations are presented with the emphasis on the correlation between processing, microstructure and creep properties. This work was performed for USDOE, Nuclear Energy, Reactor Sys. Development and Tech., Washington, D.C. 20545, under Interagency Agreement DE-Al03-86SF16310.

11:35 AM
Mechanical Behavior, Microstructural Evolution and Grain Morphology Studies in ZrN: *Ilsu Han*[1]; Pedro Peralta[1]; Kenneth J. McClellan[2]; Kirk Wheeler[1]; [1]Arizona State University, Dept. of Mechl. & Aeros. Engrg., PO Box 876106, Tempe, AZ 85287 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

The mechanical integrity of ZrN as an inert matrix in advanced nuclear fuels can be improved through understanding and control of texture and microstructural evolution. Results of average hardness for 80%, 86% and 90% density samples were 4 GPa, 7 GPa and 8.5 GPa, respectively. This increase in hardness is related to an increase in density and a more homogeneous distribution of porosity in the microstructure. These studies of mechanical behavior are related to texture and microstructural evolution in sintered ZrN. Orientation Imaging Microscopy was performed on a monolithic specimen processed using hot isostatic pressing and a sintered specimen with 80% density. Relationships between grain size and crystallographic orientation were studied to obtain information about microstructural evolution during sintering. A correlation between larger grains and orientations near to <111> is present for both samples. This suggests that <111> is the preferential direction for grain growth during sintering.

Materials Processing Fundamentals: Powders, Composites & Coatings

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Wednesday AM Room: 3001
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

8:30 AM
Processing of Vanadiferous Residues to Ferrovanadium: Robert Ressel[1]; *Markus Hochenhofer*[1]; Helmut Antrekowitsch[1]; [1]Christian-Doppler-Laboratory for Secondary Metallurgy of the Non-Ferrous Metals, Franz-Josef-Strasse 18, Leoben 8700 Austria

Recyclable material is rapidly gaining in importance as a source of raw materials in industrialized countries. This is also the case in the production of ferrovanadium, where, increasingly, materials such as discarded capacitors, spent catalysts, slag, dust and sludge are used. As the V-content in the raw material varies greatly, the demands on process technology are very high. The share of unwanted accompanyoning elements depends very much on the source of the raw materials, which, in turn, makes a corresponding optimization of the various refining steps necessary. The raw materials are mostly available as oxides, which means that carbon, silicon or aluminium are used as reducing agents. Thermodynamic calculations were used to show especially the behavior of vanadium and phosphorus at different process conditions.

8:50 AM
Oxidation Behavior of Multi-Phase Mo-Si-B Alloys: *Zhihong Tang*[1]; *Andrew J. Thom*[1]; John Kacuba[1]; Mufit Akinc[1]; Matthew Kramer[1]; [1]Iowa State University, Ames Lab., Dept. of Matls. Sci. & Engrg., Ames, IA 50011 USA

Multi-phase Mo-Si-B alloys, containing intermetallic phases for high temperature properties and a ductile phase for the fracture toughness, are being studied due to their potential for high temperature application. Three alloys have been investigated: alloy 1: Mo5Si3Bx (T1), MoB and MoSi2; alloy 2: T1, Mo5SiB2 (T2) and Mo3Si; alloy 3: Mo, T2 and Mo3Si. In the present study, the effect of water vapor and nitrogen on the oxidation behavior of multiphase Mo-Si-B alloys was examined at 1000°C. The combinational role of nitrogen and water vapor will be examined and compared with both dry and wet air. Oxidation kinetics, scale microstructural analysis and phase chemistry determination will be discussed to give a phenomenological explanation of these effects. Implication of these results for understanding high temperature oxidation behavior of Mo-based alloy as well as their possible application as oxidation-resistant coatings will be discussed.

9:10 AM
The Influence of Mechanical Processing on the Process of Thermal Reduction of SiO_2 by Al and Characterization of the Formed Si-Al_2O_3 Powdered and Compacted Composites: *Aghasi R. Torosyan*[1]; Nshan H. Zulumyan[1]; Zaruhi H. Hovhannisyan[1]; Sona E. Ghazaryan[1]; [1]National Academy of Sciences, Inst. of Gen. & Inorganic Chmst., 2-tup., Argutyan St. 10, Yerevan 375051 Armenia

Chemical reaction between SiO_2 and Al powders induced by heat treatment up to 1000°C has been investigated, depending on SiO_2 modification and time of initial mixture mechanical processing. Differential thermal analysis (DTA) studies have shown that both amorphous and crystalline SiO_2 start interact with Al powder well above the melting point of Al powder if the reactants were not activated mechanically. The situation changed dramatically in the case when the reactants had been undergone to preliminary ball milling and activation. The temperature of Si reduction for all activated (SiO_2-Al) mixtures decreases below 500°C Compat Si-Al_2O_3 ceramic composites have been prepared by compressing and annealing at 550°C the activated Al-SiO_2 powder. The microstructure and mechanical properties of the formed powdered and compacted Si-Al_2O_3 composites have been investigated by XRD, SEM and mechanical testing methods.

9:30 AM
Role of Nitrogen on the Oxidative Stability of Ti5Si3 Based Alloys at Elevated Temperature: *Zhihong Tang*[1]; Andrew J. Thom[1]; Mufit Akinc[1]; [1]Iowa State University, Ames Lab., Dept. of Matls. Sci. & Engrg., Ames, IA 50011 USA

Nitrogen is thought to have a critical role in the oxidation of Ti5Si3. In the present study, the isothermal reaction kinetics of Ti5Si3 at pure nitrogen at 1000°C was intensively investigated. Compared to a slow parabolic oxidation rate in oxygen, a faster linear reaction rate was observed when Ti5Si3 is exposed to nitrogen. Further studies on the oxidation behavior for changing nitrogen/oxygen atmospheres showed that Ti5Si3 is stable for exposure up to 400 hours at 1000°C when nitrogen partial pressure is below 0.5 atm.. Accelerated oxidation occurs after short exposures when the nitrogen partial pressure excess 0.75 atm. And with the increasing nitrogen partial pressure time to breakaway oxidation decreases. Extensive analysis of the oxidation products using SEM and XRD revealed that the formation and fast growth of a nitride-containing subscale interferes with the establishment of the continuous protective silica scale and contributes to the breakaway oxidation.

9:50 AM Break

10:05 AM
Effects of Gravity and Electric Current on Segregation and Permeation in Combustion Synthesis: *Cosan Unuvar*[1]; Daniela M. Fredrick[1]; Jennifer E. Sween[1]; Umberto Anselmi-Tamburini[1]; Anthony Manerbino[2]; Jacques Guigne[2]; Benjamin D. Shaw[3]; Zuhair A. Munir[1]; [1]University of California, Chemical Engrg. & Matls. Sci., 3118 Bianer Hall, 1 Shields Ave., Davis, CA 95616 USA; [2]Guigne Int. Ltd., 685 St. Thomas Line, Paradise, Newfoundland A1L 1C1 Canada; [3]University of California, Mechl. & Aeronautl. Engrg., 2132 Bainer Hall, 1 Shields Ave., Davis, CA 95616 USA

Combustion synthesis involves mixing powder reactants and igniting the mixture. Typically, reactions start when one of the reactants melt and it occurs in the form of a wave through the sample. In Field Activated Combustion Synthesis (FACS), the addition of an electric field has a marked effect on the dynamics of wave propagation and on the nature, composition, and homogeneity of the product as well as capillary flow, and mass-transport in porous media, which are influenced by gravity. Increasing the amount of liquid present in the reaction attenuates the effects of gravity on segregation and permeation. In order to retain sample integrity and maximize the quantity of liquid, chemical ovens with inserts that contain large amounts of molten phase(s) were used. Inserts contained aluminum as the liquid phase and tungsten, tantalum, nickel and titanium as the solid phases. Experiments have been performed in various gravitational conditions.

10:25 AM
Effect of Mechanical Attrition on Dispersion and Cold Compaction of Nickel and Aluminum Powders: *K. Morsi*[1]; Satyajit Shinde[1]; Eugene Olevsky[1]; [1]San Diego State University, Mechl. Engrg., 5500 Campanile Dr., San Diego, CA 92182 USA

Self-Propagating High Temperature synthesis (SHS) has recently been used to produce intermetallics and intermetallics composites, in short processing times and requiring minimal energy inputs. Mixing of elemental powders prior to SHS requires careful consideration for successful processing. This is particularly true for powders with submicron particles sizes. The present paper investigates the low-energy mechanical attrition of mixtures of nickel and aluminum prior to SHS, with the intention of producing a dispersed elemental powder mixture. The process is somewhat different from Mechanically Activated Self-Propagating High-Temperature Synthesis (MASHS) which has recently been successfully employed to produce nanomaterials. The influence of mechanical attrition on dispersion and powder compaction is pre-

sented. The concepts of the theory of plasticity of porous bodies are employed to assess the influence of attrition on the yield stress of thecomposite powder.

10:45 AM
Fully Automated PVD Process for Multilayer Metallic Film Coating: *Eugene Deyneka*[1]; Sergey Yarmolenko[1]; Jag Sankar[1]; [1]North Carolina A&T University, CAMSS, 1601 E. Market St., 242 Ft. IRC Bldg., Greensboro, NC 27411 USA

Automation of the multi-chamber multi-cathode DC magnetron sputtering system for high vacuum metallic film deposition is reported. The PC-controlled system is based on the Opto-22 hardware and software modules, with the computer code written in Microsoft Visual Basic. All three automation scenarios are developed: a) manual (maintenance) mode, including cryopumps regeneration module, b) semi-automated mode with simplified substrate movement between the RIE and sputtering chambers, and c) fully automated recipe-based mode having a total of over twenty etching, general, and sputtering process parameters, where the number of deposited layers is user-defined. The film thickness can range from several nanometers to several microns. The issues of film adhesion to the substrate, sputtering time, layer thickness fine-tuning and control, film uniformity, and incorporating time-depending variables into the code will be discussed. This automation program can be easily modified for a wide range of equipment as a cost-saving alternative to commercial products.

11:05 AM
Synthesis of Al2O3-SiCw Ceramic Matrix Composite by Carbothermal Reduction of Kaolin: *Sutham Niyomwas*[1]; Lek Sikong[2]; [1]Prince of Songkla University, Dept. of Mechl. Engrg., 15 Karnchanawanich Rd., Hat Yai, Songkla 90112 Thailand; [2]Prince of Songkla University, Dept. of Mining & Matls. Engrg., 15 Karnchanawanich Rd., Hat Yai, Songkla 90112 Thailand

The formations of Al2O3-SiCw composite have been obtained in situ by carbothermal reduction of a mixture of Kaolin and activated carbon. The reaction temperature was controlled at between 1400 C to 1600 C. The synthesized products were mixtures of alumina and silicon carbide in the form of whiskers. The effects of milling duration of precursors and reaction temperature are presented. XRD and SEM analyses indicate complete reaction of precursors to yield Al2O3-SiCw as product powders, with the SiC having porous and whisker morphology.

11:25 AM
Morphology Change of AlN with Respect to Starting Materials in SHS Process: Jae R. Lee[1]; Ik K. Lee[1]; Dong J. Kim[1]; Jong G. Ahn[1]; *Hun S. Chung*[1]; [1]Korea Institute of Geoscience & Mineral Resources, Minls. & Matls. Procg., 30 Kajung-dong, Yoosung-Ku, Daejeon 305-350 S. Korea

The particle size and shape effects of starting materials on the preparation of aluminum nitride by self-propagating high temperature synthesis technique under high nitrogen pressure were investigated with various AlN diluents and Al reactants. It was found that the structure of beds of the starting raw particles largely affected the pore channels for nitrogen gas infiltration as well as the passages for combustion propagation before and during the reaction, resulting in the morphology and purity changes of the synthesized products. The AlN product of purity over 98% with size of about 30 microns were obtained.

11:45 AM
Preparation of Sm-Co Alloy Oxide Precursor by Wet-Chemical Coprecipitation: *Guo Xueyi*[1]; [1]Central South University, Sch. of Metallurg. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Sm-Co alloy is a kind of magnetic material with high power and has been applied in modern industry widely. In this study, the wet chemical coprecipitation was developed for synthesis of the Sm-Co alloy oxide precusrsor. The themodynamic analysis of the studied system was done to clarify the solution behavior theoretically based on the themodynamic equilibrium and simulataneous equilibrium. Then,the experiments were done to address the various effects on the wet chemical process, including solution pH, reactant concentration, dispersant,reaction temperature and time, etc. It is found that by precise control of the process, the Sm-Co alloy oxide precursor was synthesized with fine crystallizaion and special size and size distribution.

Mechanical Behavior of Thin Films and Small Structures: Fatigue, Fracture, and Reliability of MEMS and Thin Structures II

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Wednesday AM February 16, 2005 — Room: 2024 Location: Moscone West Convention Center

Session Chairs: Christopher L. Muhlstein, Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16802 USA; David F. Bahr, Washington State University, Mechl. & Matls. Engrg., Pullman, WA 99164-2920 USA

8:30 AM Invited
Understanding and Engineering Reliability in Microelectromechanical Systems: *Srinivas Tadigadapa*[1]; [1]Pennsylvania State University, 111J EE W. Bldg., Univ. Park, PA 16802 USA

The very large surface area to volume ratios of microelectromechanical systems (MEMS) has a very strong influence on their long term stability and performance under dynamic and static loading conditions. The large surface area of such systems offers nucleation sites for the initiation of several surface reactions and modifications to occur under appropriate environmental conditions. Until now these topics have been the subject of study for understanding microstructural reliability and for the development of appropriate packaging strategies for such systems. In this paper the reliability of single crystal silicon microstructures realized using dissolved wafer process will be presented. Micromachined pressure sensor diaphragm characteristics after subjecting to various loading conditions will be discussed. The paper will also discuss the effect of surface treatments of the micromachined structures on the resonant characteristics of these structures and their implications on the performance of MEMS devices. Finally, the paper will present a quick overview of wafer level packaging of MEMS structures and some of our current efforts through wafer level bonding techniques towards achieving such goals.

8:55 AM
Addressing Mechanical Reliability Issues in Sandia MEMS Devices: *Brad L. Boyce*[1]; Thomas E. Buchheit[1]; Steven H. Goods[2]; Danelle M. Tanner[3]; Michelle A. Duesterhaus[4]; [1]Sandia National Laboratories, Dept. 1851, MS 0889, PO Box 5800, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 8754, MS 9409, PO Box 969, Livermore, CA 94551 USA; [3]Sandia National Laboratories, Dept. 1762, MS 1310, PO Box 5800, Albuquerque, NM 87185 USA; [4]Sandia National Laboratories, Dept. 2614, MS: 1310, PO Box 5800, Albuquerque, NM 87185 USA

Sandia National Laboratories is currently developing several MEMS devices. Specific examples include: an electrical contact switch with a spring fabricated from a LIGA Ni-Mn alloy, a low-G accelerometer fabricated from bulk-micromachined single-crystal silicon-on-insulator (SOI), and non-volatile memory fabricated from surface-micromachined polycrystalline silicon. This presentation will provide an overview of mechanical reliability studies motivated by the implementation of these devices. In the case of the electrical contact switch, the LIGA Ni-Mn spring is expected to perform predictably after repeated cycles and thermal exposures. Thus, reliability studies addressed issues of microplastic yielding, subsequent cyclic ratcheting, and thermal stability of the microstructure and resulting properties. The high-cycle fatigue performance will also be discussed with emphasis on the role of oxide-induced crack-initiation sites associated with persistent slip bands. In the case of the low-G accelerometer, the single-crystal silicon is expected to survive quasistatic and shock-loading environments. For this application, device-level tests have been used to evalu-

ate mechanical performance and process-induced failure sites. In the case of the non-volatile memory, the device utilizes thermal actuators and therefore the mechanical reliability at elevated temperatures becomes a primary concern. Micro-tensile tests in ambient and inert environments at temperatures ranging from room temperature to 800C indicated a dramatic decrease in the allowable stresses. In each of these examples, we will show the connection between device-level reliability and underpinning microstructural failure mechanisms. Sandia is a multiprogram laboratory operated by Sandia Corporation, A Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

9:10 AM
Performance and Durability of Monolayer Coatings Under Monotonic and Fatigue Loading Conditions: R. Kirkpatrick[1]; C. D. McCann[1]; *C. L. Muhlstein*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & the Matls. Rsch. Inst., 310 Steidle Bldg., Univ. Park, PA 16803 USA

Organic monolayer coatings have been used by the authors to enhance the fatigue resistance of silicon microelectromechanical systems (MEMS). The hydrophobic barrier provided by octadecyltrichlorosilane (OTS) and dichlorodimethylsilane (DDMS) layers prevents the intrusion of water and temporarily halts reaction-layer fatigue of silicon films. However, organic monolayer coatings eventually degrade and fail under cyclic loading conditions. In this work the mechanical behavior of organic monolayer coatings on bulk borosilicate glass and polycrystalline silicon are compared. Experimental data suggests that the fatigue susceptibility of these monolayer coatings is limited to the large amplitude, cyclic strains generated in micromechanical systems.

9:25 AM Invited
Characterization of Strength and Fatigue Properties of Thin Polycrystalline Silicon Films: *Joerg Bagdahn*[1]; Jan Schischka[1]; Heiko Knoll[1]; Matthias Ebert[1]; Robert Boroch[2]; Roland Mueller-Fiedler[2]; [1]Fraunhofer Institute for Mechanics of Materials, Heideallee 19, 06120 Halle (Saale) Germany; [2]Robert Bosch GmbH FV/FLD, Postfach 10 60 50, 70049 Stuttgart Germany

Polycrystalline silicon (polysilicon) is a widely used material for MEMS in automotive applications. In order to warranty a reliable function during the whole life of 15-20 years and a high number of mechanical cycles (up to 10^12) a sufficient mechanical strength of the thin films is required. Tensile and bend tests were applied to measure the strength of thin polysilicon films with different sample width and stress concentrations. In addition theoretical studies of the influence of the sample size and stress concentrations on the strength will be shown. The fatigue properties of polysilicon were studied on electrostatic and on external actuated samples under various testing conditions, e.g. the frequency was varied between several and 10^5 Hertz and also the environmental conditions during testing were changed between nearly vacuum and high humidity conditions. Finally, the microstructure of the tested polysilicon film was characterized by TEM, EBSD, FIB and SEM techniques and correlated with the measured strength and fatigue properties.

9:50 AM
Mechanical Behavior of Thin Gold Films for RF MEMS Applications: *I. Chasiotis*[1]; C. Bateson[1]; K. Timpano[1]; D. Koenigkann[1]; N. S. Barker[2]; [1]University of Virginia, Mechl. & Aeros. Engrg., PO Box 400746, Charlottesville, VA 22904-4746 USA; [2]University of Virginia, Electl. & Computer Engrg., PO Box 400743, Charlottesville, VA 22904-4743 USA

The effects of fabrication, film thickness, and strain rate on the mechanical behavior of thin gold films with applications in radio-frequency (RF) MEMS switches have been investigated. Microscale gold specimens with various thicknesses (0.2-2.5 microns) were fabricated using sputtering, evaporation, and electroplating. The gage section width of these free-standing, dog-bone shaped specimens varied between 50-200 microns. The mechanical characterization was conducted via a custom-built uniaxial tensile testing system that employed a low force load cell and a piezoelectric actuator for force and displacement measurements, respectively. The stress-strain curves of the gold films demonstrated elastic-nearly perfectly plastic behavior. The value of Young's modulus was found to agree well with that of bulk gold, averaging 71 GPa. The tensile specimens demonstrated high ductility that increased with decreasing strain rate. For strain rates between 10^{-3} s^{-1} to $10^{-5}s^{-1}$, the ductility was approximately 6% for middle range strain rates and as high as 9% for specimens subjected to monotonic loading at low strain rates. Over the entire range of strain rates the ductility was found to vary by a factor of 4. For the same range of strain rates the peak stress varied between 350-450 MPa and it decreased with decreasing strain rate. The average film strength was comparable to that reported previously for thin films of similar thickness that were also tested in tension. The yield strength was found to be about 300 MPa for low and 350 MPa for high strain rates, respectively, which is also comparable to literature values. Examination of fracture cross-sections indicated a dependence on the material fabrication process and applied strain rate. Furthermore, the effects of specimen thickness and fabrication process on the mechanical properties of thin gold films will be discussed in this presentation.

10:05 AM Break

10:20 AM Invited
Through Thickness Fracture Behavior in Hard Films on Soft Substrates: *D. F. Bahr*[1]; K. R. Morasch[1]; M. S. Kennedy[1]; S. P. Anderson[1]; N. R. Moody[2]; [1]Washington State University, Mechl. & Matls. Engrg., PO Box 642920, Pullman, WA 99164-2920 USA; [2]Sandia National Laboratories, PO Box 969, Livermore, CA 94550-0969 USA

The mechanism of film fracture, particularly in thin (sub micron) films has been difficult to quantify due to the scales of measurement of load and displacements. However, using nanoindentation coupled with atomic force microscopy, it is possible to quantify differences in film fracture in a variety of systems of hard films on soft substrates. This presentation will review 4 different film systems, all of which exhibit similar trends in fracture morphology but span a range of properties. Oxide films on active metals (aluminum and stainless steel), piezoelectric oxides on metallic layers, and tungsten films on polymer substrates have been tested using nanoindentation. Discontinuities in loading behavior have been identified using atomic force microscopy as through thickness fracture events. An applied stress intensity for fracture will be developed based on bending moments on the surface induced by nanoindentation. The wide range of elastic and plastic properties will demonstrate the appropriateness of the model. The stress intensity at fracture is impacted by film growth conditions, for instance in aluminum oxide on aluminum the stress at fracture increases as film thickness increases, but the critical stress intensity for fracture is highest for films thinner than 30 nm, and decreases asymptotically towards a toughness of 1 MPa√m.

10:45 AM
Anomalous Near-Threshold Debond Growth Rate Behavior in Thin-Film Structures: Implications for Device Life: *Bree M. Sharratt*[1]; Lorraine C. Wang[2]; Reinhold H. Dauskardt[2]; [1]Stanford University, Aeronautics & Astronautics, c/o Matls. Sci. & Engrg., Peterson Bldg. 550, 416 Escondido Mall, Stanford, CA 94305-2205 USA; [2]Stanford University, Matls. Sci. & Engrg., Peterson Bldg. 550, 416 Escondido Mall, Stanford, CA 94305-2205 USA

An understanding of the mechanisms associated with interfacial debonding is crucial for designing devices containing thin-film structures where long-term reliability is of concern. We report on an anomalous near-threshold debond growth rate behavior measured at the interface between a thin epoxy layer and an adjacent passivated silicon substrate under both monotonic and cyclic loading. The near-threshold debond growth rates plateaued at ~10^{-9} m/sec and were largely insensitive to the applied loads. The surprising similarity of the plateaus measured under cyclic and monotonic loading indicated that the mechanical fatigue damage accumulation process, a significant accelerant at higher growth rates, was overshadowed in the near-threshold region. The dependence of the plateau growth rates on environmental species and temperature is presented together with a possible chemical reaction based mechanism. These results have significant implications for the operational definition of a crack growth threshold and associated operating life of such thin-film structures.

11:00 AM
Fracture Toughness of Thin Films on Compliant Substrate Using Controlled Buckling Test: *Zhong Chen*[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Nanyang Ave. 639798 Singapore

Thin films and multilayered structures are increasingly used in industry. One of the important mechanical properties these thin layers is the fracture toughness, which is not the same as the one obtained from bulk samples. Film fracture toughness is extremely important in design for coatings on plastic or other applications where flexibility is required. This work presents a scheme using controlled buckling experiment to measure the fracture toughness of brittle thin film on compliant substrate. When the film is under tension, steady-state channeling cracks form. Critical fracture strain can be obtained by the displacement in the buckle. Calculation of fracture toughness is presented allowing the substrate to experience plastic deformation. Finally examples are given to illustrate such a test scheme.

11:15 AM Invited
Cohesive and Adhesive Fracture in Complex Device Structures: *Reinhold H. Dauskardt*[1]; [1]Stanford University, Dept. of Matls. Sci. & Engrg., Stanford, CA 94305-2205 USA

Debonding of interfaces and cracking of fragile interlayer dielectrics effects the mechanical integrity of a wide range of thin-film device structures. This results in reduced yield at all levels of device processing including survival through chemical mechanical planarization (CMP) and subsequent device packaging. Two unique challenges for the next technology nodes involve the introduction of new ultra low k dielectric materials and the effect of device architecture including length-scales and aspect ratios, on mechanical and fracture behavior. Materials are nearly always optimized for other desired properties (e.g. dielectric properties or diffusion resistance) and the resulting effects on mechanical performance can be significant. In this presentation, the mechanical and fracture behavior of representative blanket and patterned thin-film structures including glass and organic dielectrics, barriers and metal layers, are examined. The acceleration of crack growth in complex chemical environments typically encountered during processing is discussed. The effects of interface parameters and thin-film composition and porosity will also be considered. Novel strategies to toughen fragile nanoporous materials using molecular remnants of the porogen molecules used to create the porosity are described. Finally, the effect of more complex patterned thin-film structures are examined where length scales are restricted in more than one dimension. Implications for device reliability, integration of new materials, and life prediction are discussed.

11:40 AM
Driving Forces of Bond Coat Surface Rumpling in Thermal Barrier Systems: *K. Jimmy Hsia*[1]; [1]University of Illinois, Theoretl. & Applied Mech., 111B Talbot Lab, MC-262, 104 S. Wright St., Urbana, IL 61801 USA

Failure of thermal barrier coatings is often caused by nucleation and growth of interfacial cracks between the ceramic coating and metallic bondcoat (BC). The underlying process causing such cracking is the progressive rumpling of the BC surface upon thermal cycling. It is believed that the mismatch/growth stresses in the thermally grown oxide (TGO) provide the driving force, and either surface diffusion or plastic ratcheting gives rise to rumpling. Another proposal is the selective diffusion of species and resulting microstructural variations. We carefully studied these mechanisms by critical experiments, focusing in particular on the role of TGO, the role of thermal cycling, the role of microstructural evolution, and the role of the BC. Our findings are: the presence of TGO is not critical for rumpling; thermal cycling is not a necessary condition; there is poor correlation between microstructural variations and peak locations of the rumples. The likely driving force is the mismatch stress in the BC.

11:55 AM
Temperature, Loading, and pH Effects on Debonding of Silica/Adhesive Layered Structures: *Louise Y. Wang*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305 USA

Epoxy resins blended with silane coupling agents are widely used adhesives between passivated device substrates and polyimide films in microelectronic packages. However, the resulting silica/adhesive interface and adhesive itself are susceptible to accelerated debonding and cracking in moist, aqueous and corrosive environments. This study focuses on the effects of temperature, pH and more complex mechanical loading on interfacial debonding. Increased debond velocities were apparent with increasing temperature which was related to enhanced reaction kinetics at the debond crack tip. In buffered basic solutions and with increasing temperature, the fracture path was found to meander within the adhesive and adjacent interfaces. In acidic solutions debonding was restricted to the adhesive layer. A plateau growth rate region was apparent at intermediate growth rates in the basic solution. Mechanisms for the accelerated cracking are described and implications for packaging reliability and life prediction considered.

12:10 PM
Tribological Property Investigation for Pulsed Laser Deposited Oxide Thin Films: *Xinyu Wang*[1]; Sudheer Neralla[1]; Sergey Yarmolenko[1]; Dhananjay Kumar[1]; Jagannathan Sankar[1]; [1]North Carolina Agricultural and Technical State University, Dept. of Mechl. & Cheml. Engrg., CAMSS, Greensboro, NC 27411 USA

Tribological properties are an important concern for researchers developing thin films. In this study, alumina (Al_2O_3) and silica (SiO_2) thin films are deposited on silicon (Si) (100) and steel substrates using the pulsed laser deposition (PLD) technique. Different substrate temperatures ranging from room temperature to 800ºC and different laser energies are used. Al_2O_3 and SiO_2 thin films with different thicknesses ranging from 500 nm to 4 µm are developed. Tribological properties such as adhesion, friction, and wear are investigated by performing the nanoscratch test using a nanoindenter and the wear test using a microtribometer. Scratching length of 500 µm and maximum loads of 200 mN are used for the nanoscratch tests. A profilometer, a scanning electron microscope (SEM), a transmission electron microscope (TEM) and an atomic force microscope (AFM) are used to analyze film properties such as surface topography, roughness (RMS) and cross-sectional microstructures. Preliminary results have shown that a strong adhesion between a film and the substrate on which it was deposited existed and different values of adhesion were also found. Direct interfacial fracture observations are made by polishing the film-substrate cross sections. The preliminary observations illustrated the half-penny cracks. Critical loads in the nanoscratch tests and critical number of cycles in the wear tests of different films are compared. The test results provide further understanding into the tribological properties of the oxide thin films.

Metallurgical Technology for Waste Minimization: Session II

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Toru Okabe, University of Tokyo, Institute of Industrial Science, Tokyo Japan; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA

Wednesday AM Room: 2012
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Yoshiaki Umetsu, Tohoku University, Japan; Pat R. Taylor, Colorado School of Mines, Golden, CO 80401 USA

8:30 AM Invited
Investigation of Ion Exchange Resins for Use in Treatment of Semiconductor Processing Waste Streams: *Jeffrey D. Winterton*[1]; Fiona M. Doyle[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., 210 Hearst Mining Bldg. #1760, Berkeley, CA 94270-1760 USA

The semiconductor industry is rapidly shifting from aluminum to copper as the interconnect material of choice. This shift is accompanied by the creation of several copper-containing aqueous waste streams from different stages of processing. The streams typically have a range of copper concentrations and a variety of additive agents. We are investigating the use of ion exchange resins as a means of recovering the copper from these streams within the processing plant, thereby allowing for the recycling of process waste water and the minimization of hazardous waste generation. Resins of different functionalities have been investigated over a range of processing conditions with the goal of developing a computer based model for the operation. Factors investigated include copper affinity, uptake and elution kinetics, and effect of various plating additives during copper adsorption.

9:00 AM Invited
Adsorption Mechanism of Palladium by Redox Within Condensed-Tannin Gel: *Yoshio Nakano*[1]; [1]Tokyo Institute Technology, Interdisciplinary Grad. Sch. of Sci. & Tech., Dept. of Environml. Chmst. & Engrg., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

Tannin gel particles with polyhydroxyphenyl groups were synthesized as the adsorbent for the new recovery system of palladium(Pd), which was simple and generated little secondary waste in comparison with the conventional recovery processes. The properties of tannin gel particles for the adsorption of Pd were examined in $PdCl_2$ aqueous solution and resulted in that Pd is adsorbed onto the tannin gel particles as a reduced metallic Pd through redox reaction mechanism: chloropalladium(II) species are reduced to Pd(0), while hydroxyl groups of tannin gel are oxidised during the adsorption. Additionally, it was obserbed that Pd species containing fewer Cl, such as $PdCl_2(H_2O)_2$ and $PdCl(H_2O)_3^+$, are more favorable for the adsorption than $PdCl_3(H_2O)^-$ and $PdCl_4^{2-}$. By utilizing such characteristics of tannin gel particles, it is expected that they can be applied to recover Pd efficiently and simply with low cost.

9:30 AM
Heavy Metal Adsorption Properties of Zeolite Synthesized from Coal Bottom Ash: *Jin-Koo Park*[1]; So-Yun Jeon[1]; Ji-Whan Ahn[1]; [1]Korea Institute of Geoscience & Mineral Resources, Minls. & Matls. Procg. Div., Taejon 305-350 Korea

At present, about 70% of the coal fly ash that discharged from domestic thermoelectric power plants is reused as raw materials in the manufacture of cement and concrete. And researches on synthesis of zeolite synthesized from coal fly ash have been performed to obtain the high-value industrial products for the last two decades. However, coal bottom ash occupying about 15% of total coal ash is totally disposed at present. Coal bottom ash can also used as raw materials for the synthesis of zeolites because it contain large amounts of Al and Si. In this study, we synthesized zeolite from coal bottom ash at various NaOH concentrations and reaction times. Then, physio-chemical properties of the synthesized zeolite such as crystal structure, surface area and cation exchange capacity were investigated. Also, characterized adsorption and elution properties of heavy metals for apply the synthesized zeolite to soil improvement agent or water treatment agent.

9:50 AM Break

10:05 AM
Development of Recovery Process of Tetra-Methyl Ammonium Hydroxide from Waste Solution: Junji Shibata[1]; Tomohiro Yanase[1]; *Norihiro Murayama*[1]; Hideki Yamamoto[1]; [1]Kansai University, Cheml. Engrg., 3-3-35, Yamate, Suita, Osaka 564-8680 Japan

Tetra-methyl ammonium hydroxide is used in various electric and electronic parts production processes such as semiconductor, liquid crystal display and printed circuit board. The discharged amount of this chemical reaches about 2,500 ton /year from one factory in Japan. The waste liquor contains a lot of organic matter, which means high BOD and COD. Fundamental study found that tetra-methyl ammonium hydroxide can be separated and recovered from the waste liquor by the ion exchange method, which is one of the hydrometallurgical purification methods. The waste liquor discharged from the liquid crystal display production contains 0.53wt% tetra-methyl ammonium hydroxide, 60mg/dm3 phenol and ppb level of metal ions. We need development of a separation and recovery process for tetra-methyl ammonium hydroxide. In the cation exchange reaction, tetra-methyl ammonium ion is captured on the cation exchange resin. Other non-ionic organic matter like phenol goes through the resin without being captured on it, the separation being attained in this step. In the elution step, tetra-methyl ammonium ion captured on the resin is released as tetra-methyl ammonium chloride into aqueous solution by the action of dilute hydrochloric acid. Tetra-methyl ammonium chloride is converted to tetra-methyl ammonium hydroxide by the reaction between tetra-methyl ammonium chloride and OH-type anion exchange resin. The process is composed of three steps; cation exchange, elution and conversion. The experiments were carried out using ion exchange resin column of 20mmö and 735mm high, and the recovery and purity of tetra-methyl ammonium hydroxide were clarified at each step. They were dependent on various factors such as space velocity in cation exchange, elution and conversion columns, and hydrochloric acid concentration in elution column. One example of experiments exhibited that the recovery of tetra-methyl ammonium hydroxide over three steps was 80% and the purity was 99%, if 10-15 space velocity in each step and 0.3-1.0 mol/dm3 hydrochloric acid in elution step were used. A successive resin column process was developed from the experimental results and then the novel recovery process for tetra-methyl ammonium hydroxide waste liquor was proposed. The assessment of yield and purity at each step was conducted to clarify how the proposed process is effective.

10:25 AM
Biological Fluegasdesulpurization: Sustainable, Effective and Cost-Efficient: *Gerard Schouten*[1]; Jacco Huisman[1]; Henk Dijkman[1]; [1]Paques bv, Business Dvlp., PO Box 52, Balk The Netherlands

Abstract TMS 2005:With the introduction of ever-stricter environmental operating guidelines, capital expenditure restrictions and operational budget cutbacks, the biological method of SO2 removal becomes more and more attractive. Biotechnological treatment of dilute flue gases is a more cost effective technology in comparison to conventional sodium hydroxide scrubbing. Although the investment costs for sodium hydroxide scrubbing are lower, the operational costs are significantly higher. In addition, the end product of the biological installation is sulfur instead of sodium sulfate; and no further wastewater treatment of the bleed from the biological installation is required since heavy metals are removed simultaneously. Another option for the treatment of SO2 containing gas at a sulfuric acid plant is the production of extra sulfuric acid of the discharged SO2 by applying an additional converter. Comparing biological treatment with this option shows much lower investment costs for the biological installation. Sulfur is produced which can be converted to sulfuric acid, so the bio-route will be more cost-effective than an additional converter. In principle, the biotechnological flue gas desulfurisation system (BioDeSOx) is an integration of an absorber with a "biological buffer regeneration facility". In the absorber, the SO2 containing gas is brought into contact with wash liquid (a sodium bicarbonate buffer). The absorber effluent contains a mixture of sulfite and sulfate that is converted into elemental sulfur whereby the wash liquid is regenerated. This conversion takes place in an integrated system; first, the sulfite/sulfate mixture is converted anaerobically into sulfide; secondly, the sulfide is converted aerobically into elemental sulfur. Subsequently the sulfur is separated and the water is returned to the absorber. Due to the buffer capacity of the washing water, removal efficiencies over 98% are possible. In addition to SO2 removal, it is possible to convert for example other bleeds streams such as scrubber acid or weak acid to sulfur in the same SO2 installation. Recent evaluations concluded that biological desulphurization of flue gases is not only economically attractive in comparison to convention sodium hydroxide scrubbing but also in comparison to gypsum producing systems and even seawater scrubbers. This paper describes the technological and economical viability of biological flue gas desulphurization.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Mechanics of Nanostructures

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Wednesday AM Room: 3000
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: C. S. Pande, Naval Research Laboratory, Washington, DC 20375 USA; M. H. Yoo, Korea Advanced Institute of Science & Technology, Dept. of Matls. Sci. & Engrg., Daejeon 305-701 Korea

8:30 AM Invited
Relaxation Effects in Nanocrystalline and Ultra-Fine Grain Metals: Kai Zhang[1]; *Julia R. Weertman*[1]; [1]Northwestern University, Matls. Sci. & Engrg. Dept., 2220 Campus Dr., Evanston, IL 60208 USA

Microhardness measurements on nanocrystalline and ultra-fine grain copper samples show a decrease in hardness over a period of hours. The relaxation rate increases with decreasing temperature, decreasing grain size, and increasing sample purity. Characterization studies examine structural changes in the indent region associated with the mechanical behavior. This research is supported by DOE grant DE-FG02-02ER 46002.

8:55 AM Invited
Effects of Irregular Distribution of Particles on Stress Concentrations in Composites: The Case of Close Approach: *Daniel N. Beshers*[1]; Richard J. Seymour[1]; [1]Columbia University, Matls. Sci. & Engrg., MC 4701, 500 W. 120th St., New York, NY 10027 USA

A common approximation with composite materials neglects the interaction of the particles. For close particles, this neglect is not justified. There are two aspects. One is the appearance of induced stress fields both inside and outside the inclusion. The induced field of one particle adds to the stress on a neighbor and vice versa, leading to a final state with enhanced stress, analogous to interactive effects in electric and magnetic polarization. The second aspect is that there are in general discontinuities of elastic fields at the interface between a

particle and the surrounding matrix. When the inclusions are close, the material between them is subject to finite deformations on each side, but with only a small distance over which to accommodate them. The strain may become very large when the separation is of the order of nanometers. We present analyses of two simple examples.

9:20 AM Invited
Effects of Stress on the Formation of Metallic or Semiconducting Nanostructures on Si and SiGe: *L. J. Chen*[1]; [1]National Tsing Hua University, Matls. Sci. & Engrg., 101, Sec.2, Kuang Fu Rd., Hsinchu 300 Taiwan

Nanostructures, including nanodots and nanowires, are attracting much interest because they are expected to play an important role in nanometer-scale electronics and optoelectronics. Stress has often been found to play an important role in influencing the formation of nanostructures. In this presentation, we provide an overview on the effects of stress on the formation of nanostructures on Si or SiGe. Several examples involving stress-induced effects such as self-assembled NiSi quantum-dot arrays on epitaxial Si0.7Ge0.3 on (001)Si, self-assembled nano-rings in Si-capped Ge quantum dots on (001)Si, growth of TiSi2 and rare-earth silicides nanowires on silicon and self-forming silicide/SiGe-based tube structure on Si will be given.

9:45 AM Invited
Nanomechanical Behaviour of Piezoelectric Nanowire: *Xinyuan (Scott) Mao*[1]; M. Zhao[1]; C. B. Jiang[2]; Suoxing Li[2]; [1]University of Pittsburgh, Mechl. Engrg., 3700 O'Hara St., Pittsburgh, PA 15261 USA; [2]Shengyang Metal Research Institute, Shengyang China

Quasi one-dimensional (1D) solid nanostructures, such as nanobelts of semiconducting oxides, have stimulated considerable interest for scientific research due to their importance in mesoscopic physics studies and their potential applications as nano-devices, nanocantilevers, nanoactuators and nanosensors. A key challenge to today's research is the experimental difficulty in fabricating, manipulating and testing the physical properties of a single nanowire/cantilever whose size is in the nano- to micron-meter range, because the small size (diameter and length) of the object prohibits the applications of the well-established testing techniques. Belt-like oxide nanostructures, so called nanobelts, were successfully synthesized by evaporating ZnO powders at high temperatures without the presence of catalyst. Morphology analysis shows the nanobelts have a rectangle-like cross section with typical widths of several hundred nanometers, width-to-thickness ratios of 5 to 10, and lengths of hundreds of micron meters. Nanoindentations were made on individual ZnO nanobelts by using AFM and Hysitron Triboscope indenters. It was shown that the indentation size effect was still obvious for the indentation depth under 50nm. It is also demonstrated that nanomaching is possible on nanobelt using AFM tip. Piezoelectric behaviour is measured by electric force microscope, which shows strong size effect.

10:10 AM Break

10:15 AM Invited
Dielectric and Mechanical Analysis of Polymer Nanocomposites: *Julie P. Harmon*[1]; Kadine Mohomed[1]; LaNetra Clayton[1]; Timofey Gerasimov[1]; [1]University of South Florida, Chmst., SCA 400, 4202 E. Fowler Ave., Tampa, FL 33620-5250 USA

A variety of polymer nanocomposites were fabricated via in situ ultrasonic polymerization and melt blending. Various techniques were used to characterize the thermal, dielectric and mechanical properties of the nanocomposites. Differential scanning calorimetry (DSC) was used to obtain the glass transition temperature (Tg) of the nanocomposites. Dielectric analysis (DEA) monitored changes in the dielectric response with respect to increasing nanofiller concentration. Dynamic mechanical analysis (DMA) was used to characterize the viscoelastic properties. Nanofiller dispersion quality was tested via scanning electron microscopy (SEM) and optical microscopy. Optimizing dispersion quality enhances the ability to obtain peak performance in key properties. As exptected, DEA results show a dramatic change in the dielectric spectra of the nanocomposites occurring at the point where the percolation threshold is reached.

10:40 AM
Creep-Rupture Behavior of Fine-Diameter Ceramic Fibers with Nano-Sized Grains: *James A. DiCarlo*[1]; [1]NASA Glenn Research Center, Matls. Div., 21000 Brookpark Rd., MS 106-5, Cleveland, OH 44135 USA

The successful application of ceramic matrix composites (CMC) in high-temperature applications depends strongly on developing fine-diameter (~10 μm) ceramic fiber reinforcement with a variety of key thermostructural properties. Foremost amongst these are high as-produced tensile strength and retention of a large fraction of this strength for long times under anticipated CMC service conditions. Using creep-rupture data measured at NASA on single and multi-fiber specimens, this presentation reviews the high-temperature intrinsic strength behavior of commercially available oxide and non-oxide fiber types. It is shown that that there are significant differences in creep-rupture behavior between the various fiber types and that these differences are influenced by such factors as base chemical composition, process-related defect sizes, grain sizes, grain boundary phases, surface chemistry, surface morphology, and test environment. On-going studies at NASA to model the effects of these factors in order to predict and optimize fiber/CMC creep and strength retention are discussed.

11:00 AM Invited
Influence of Grain Size on Deformation Mechanisms in Polycrystalline Materials: *Yuntian Ted Zhu*[1]; Terence G. Langdon[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The deformation mechanisms in coarse-grained polycrystalline materials are understood reasonably well. Generally, the primary deformation processes are associated with the movement of dislocations either through crystallographic slip at low temperatures or through a combination of dislocation climb and glide at high temperatures. Additional possible processes include stress-directed vacancy diffusion and grain boundary sliding. It has been shown using molecular dynamic simulations, and confirmed in experiments, that different processes become important when the grain size is reduced to the nanometer level. Partial dislocation emission from grain boundaries becomes a dominant process at grain sizes of 10 to 50 nm and this leads to the formation of deformation twins even in high stacking-fault materials such as aluminum. Grain boundary sliding also becomes dominant at grain sizes below 10 nm at low temperatures. This presentation gives an overview on the influence of grain size on deformation mechanisms in polycrystalline materials.

11:20 AM Invited
A Model for the Inverse Hall-Petch Relation of the Nanocrystalline Materials: *Guojiang Fan*[1]; H. Choo[1]; P. K. Liaw[1]; E. J. Lavernia[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Dougherty Engrg. Bldg., Knoxville, TN 37996 USA; [2]University of California, Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

Nanocrystalline (nc) materials have received world-wide research interests due to their superior mechanical properties. Recently, new deformation mechanisms have been identified for the nc materials with the grain sizes on the order of several tens of nanometers, where dislocation pile-ups by the grain boundaries may not be operative. We propose a composite model to explain the phenomena of strength softening with decreasing the grain size, which was reported in some nc materials. We assume that a nc material consists of a grain interior and an amorphous grain-boundary layer. The grain interior deforms elastically under external stresses, while the plastic deformation of the grain-boundary layer was governed by a Maxwell's equation. Based on this model, we will show that the strength of a nc material decreases linearly with decreasing the grain size, when the grain size is below a certain threshold. The model is compared with the experimental data from the published studies on the nc Cu and Ni. The predictions of relevant creep mechanisms for nc materials are also discussed.

11:40 AM
Plastic Flow Localization and Shear Banding in Tungsten: *Q. Wei*[1]; E. Ma[1]; K. T. Ramesh[1]; L. J. Kecskes[2]; L. Magness[2]; R. J. Dowding[2]; R. Z. Valiev[3]; [1]Johns Hopkins University, Ctr. for Advd. Metallic & Ceram. Sys., Baltimore, MD 21218 USA; [2]Army Research Laboratory, Aberdeen Proving Ground, MD 21005 USA; [3]Ufa Technical University, Russia

Over the past twenty years, shear localization in tungsten (W) has been sought after for certain critical applications. We demonstrate that by refining the microstructure of bulk, commercial purity, W into the nanostructured and ultrafine-grained regime, adiabatic shear localization can be induced under dynamic compression in standard Kolsky bar tests. This unusual property is also observed for a number of other fine-structured bcc metals. The propensity for localization is discussed in terms of the strain hardening, strain-rate hardening, and thermal/geometrical softening behavior in bcc metals as a function of microstructure refinement.

12:00 PM
Deformation Behavior and Mechanisms of an Extruded Nanocrystalline Al-Fe-Cr-Ti Alloy: *Hong Luo*[1]; Leon L. Shaw[1]; Lichun Zhang[1]; D. Miracle[2]; [1]University of Connecticut, Inst. of Matls. Sci., 97 N. Eagleville Rd., Storrs, CT 06269 USA; [2]Air Force Research

Laboratory, Matls. & Mfg. Direct., Wright-Patterson AFB, OH 45433 USA

Deformation behavior of an extruded nanocrystalline (nc) Al-Fe-Cr-Ti alloy (80 - 150 nm) has been investigated systematically using compressive tests as a function of temperature and strain rate. A variety of analytical instruments including X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy dispersive spectrometer (EDS), and electron energy-loss spectrometry are utilized to characterize the material before and after deformation. The TEM analysis reveals the evidence of dislocation activities in the compressive deformation. Furthermore, the dislocation density depends on the test temperature and strain rate. Decreasing strain rate or increasing test temperature decreases the dislocation density. An unusual phenomenon, long stacking fault (SF) which is very difficult to form in aluminum because of the high SF energy, is found in the nc-Al alloy after compressive deformation. The mechanical properties of the nc Al-Fe-Cr-Ti alloy cannot be explained by either coble or power law creep. The strain rate sensitivity of this Al-Fe-Cr-Ti alloy is about 0.02 which is comparable with that of the coarse-grained Al alloys. The deformation mechanism of the nc Al-Fe-Cr-Ti alloy has been discussed based on the microstructure observation and mechanical properties.

Microstructural Processes in Irradiated Materials: He/H Interactions and Ferritic/Martensitic Steels

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Wednesday AM Room: 3011
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Shigeharu Ukai, Japan Nuclear Cycle Development Institute, Oarai Engrg. Ctr. Sys. Engrg. Tech. Div., Higashi-Ibaraki-Gun, Ibaraki-Prefecture 311-1393 Japan; Yutai Katoh, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA

8:30 AM
Ab Initio Study of Helium-Vacancy Defects in Iron: *Chu Chun Fu*[1]; Francois Willaime[1]; [1]CEA/Saclay, SRMP, Gif sur Yvette 91191 France

Ferritic steels are proposed as first wall material in fusion reactors. When submitted to 14 MeV neutron irradiation, not only self-defects but also Helium and Hydrogen atoms are created. Quantitative studies are required to predict the effect of Helium on microstructural and mechanical properties of these materials We report ab initio results on properties of Helium insertion and of small helium and helium-vacancy complexes in bcc Iron, and possible Helium migration mechanisms. Helium atoms prefer to occupy substitutional sites, followed by tetrahedral ones with only 0.17 eV higher in energy. Interstitial Helium migrates very fast (Em = 0.06 eV) and can be easily trapped by vacancy clusters, vacancy-helium complexes and even by helium clusters. We also show here the role of Helium on void and bubble formation. Finally, we use our ab initio data to fit the Iron-Helium interatomic potential.

8:50 AM
Role of Magnetic Interactions on the Properties of He Defects in Iron: *Tatiana Seletskaia*[1]; Yuri Osetsky[2]; Roger E. Stoller[1]; G. Malcolm Stocks[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA; [2]Oak Ridge National Laboratory, Computer Sci. & Math. Div., Oak Ridge, TN 37831-6138 USA

Density functional theory calculations of He defect properties in iron have shown an unexpected influence of magnetism arising from the defect's electronic structure. In contrast with previous work that neglected such effects, the results indicate that the tetrahedral position is energetically more favorable for the He interstitial than the octahedral site. However, the He substitutional defect is found to be energetically more favorable than the interstitial position. The dissociation energy of He from the vacancy is found to be 2.29 eV which is much smaller than in the previous calculations. This may have significant implications for He diffusion, clustering and its interaction with point defects, which will impact material performance in future fusion reactors. These results provide the basis for development of improved atomistic models.

9:10 AM
Helium - Self-Interstitial Atom Interaction in Fe: *Lisa Ventelon*[1]; Brian D. Wirth[1]; Christophe Domain[2]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA; [2]EDF-R&D, Dept. MMC, Les Renardieres, Moret sur Loing 77250 France

We present the result of atomistic calculations to investigate the effect of He impurities on the properties and behavior of self-interstitial atom clusters in Fe. Ferritic alloys are currently being considered for fusion energy first wall applications, and will be exposed to high levels of radiation damage and transmutation production in a 14 MeV fusion neutron spectrum. We present a comparison of the interaction energies between interstitial He atoms and a single self-interstitial atom (SIA) obtained with ab-initio electronic structure and semi-empirical interatomic potentials using molecular dynamics and conjugate gradient molecular statics calculations. These results provide insight into the validity of using semi-empirical interatomic potentials and a basis for extrapolating ab-initio results from small to larger system sizes. We also present the results of a MD investigation into the migration behavior of SIA and SIA clusters in the presence of interstitial and substitutional He. The MD simulations reveal a strong interaction between He and SIA clusters, often resulting in SIA - vacancy reaction that spontaneously eject helium into interstitial sites and provide quantitative information on the interaction radii, trapping - binding energetics and migration behavior of mixed He-SIA clusters.

9:30 AM
Atomistic Modeling of He Binding and Migration at Interfaces in Fe: *Richard J. Kurtz*[1]; Fei Gao[1]; Howard L. Heinisch[1]; Brian D. Wirth[2]; G. Robert Odette[3]; Takuya Yamamoto[3]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., PO Box 999, Richland, WA 99352 USA; [2]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720 USA; [3]University of California, Dept. of Mechl. & Environml. Engrg., Santa Barbara, CA 93106 USA

High transmutation He concentrations will be produced in fusion neutron environments. Helium is essentially insoluble and aggregates at grain boundaries, significantly degrading various mechanical properties. So-called nanostructured ferritic alloys are being developed to both improve creep strength and to better manage He by creating a high-density of Ti-Y-O enriched nano-particles to serve as dislocation obstacles and fine-scale He bubble nucleation sites. The binding and migration energies of He atoms and defects at grain boundaries and coherent Cu precipitate interfaces is assessed with molecular dynamics simulations using Fe-Cu-He embedded atom type potentials. Small coherent Cu clusters were used to explore the efficiency of positive misfit nano-particles for trapping He. The energy of substitutional He in grain boundaries and Cu nano-particles is moderately lower than in the matrix, while the corresponding grain boundary energy of interstitial He is much lower than in the matrix. Binding energies roughly correlate with excess volume.

9:50 AM
Atomistic Simulations and Experimental Studies of the Effects of Helium and Hydrogen on Irradiation Damage in BCC Iron: *Maria A. Okuniewski*[1]; Srinivasan G. Srivilliputhur[2]; Chaitanya S. Deo[2]; Stuart A. Maloy[2]; Mike I. Baskes[2]; Mike R. James[2]; James F. Stubbins[1]; Doug P. Wells[3]; Farida A. Selim[3]; [1]University of Illinois, Nucl. Engrg., 103 S. Goodwin Ave., Urbana, IL 61801 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA; [3]Idaho State University, Idaho Accelerator Ctr., Pocatello, ID 83209 USA

High-energy spallation neutron irradiation in metals results in the production of helium and hydrogen, as well as displacement damage. The displacement damage and the introduction of helium and hydrogen, can lead to altered mechanical and physical properties. Molecular dynamics (MD) and kinetic Monte Carlo (KMC) modeling as well as experimental techniques are used to understand the atomistics and characterize the microstructural evolution during the irradiation processes in Fe, Fe-He, and Fe-H systems. Using MD simulation techniques, pure Fe, Fe-He, and Fe-H systems are subjected to irradiation damage. The effects of incident ion energies, helium and hydrogen concentrations, and temperatures on the evolution of defects, including He and H interstitials, clusters, and bubbles are investigated. The modified embedded atom method potential, which explicitly incorporates angular forces that are essential to model the Fe-He and Fe-H systems, is used. KMC simulations are performed to understand the

evolution of defects and clustering as a function of temperature and varying defect ratios. These stochastic KMC simulations are parameterized by data obtained from experiments and MD calculations. KMC can simulate the system on a timescale much larger than MD (typically picoseconds), which allows for the diffusion of defects. Positron annihilation spectroscopy (PAS) experiments are utilized to characterize irradiation damage in single crystal bcc Fe. PAS is able to measure vacancies, voids, bubbles, and dislocation loops at low concentrations. Bubbles and voids can be detected in the sub-nanometer range, which is below the resolution of transmission electron microscopy. The defect evolution will be studied as a function of implantation dose and temperature. The results obtained from modeling using MD and KMC will be compared with experimental results obtained from PAS.

10:10 AM Break

10:40 AM Invited
Microstructural Development Under Irradiation in European ODS Ferritic/Martensitic Steels: *Robin E. Schaeublin*[1]; Nadine L. Baluc[1]; [1]Swiss Federal Institute of Technology Lausanne, Ctr. of Rsch. in Plasma Physics, Fusion Tech. - Matls., ODGA/105, Villigen 5232 Switzerland

Oxide dispersion strengthened steels based on the ferritic/martensitic steel EUROFER97 are developed within the European fusion program. The reinforcing particles represent 0.3% to 0.5% weight and are composed of yttria, which is incorporated by ball milling. Compaction is made by hot isostatic pressing or hot extrusion. ODS steel samples have been irradiated with 590 MeV protons to 0.3 and 1.0 dpa at room temperature. Microstructure is investigated by transmission electron microscopy and mechanical properties are assessed by tensile and Charpy tests. The ODS material presents a martensitic-like microstructure and a uniform distribution of the yttria particles. While the fracture behavior is drastically degraded by the oxide particles, it can be partially recovered by appropriate thermo-mechanical treatments. At room temperature the yield strength at 0.2% is 1 GPa and plastic strain at failure is 8%. Results of the irradiation on the microstructure and the mechanical properties are presented.

11:20 AM
Microstructural Development in Advanced Ferritic-Martensitic Steel HCM12A: Todd R. Allen[1]; Lizhen Tan[1]; Julie D. Tucker[1]; *Jian Gan*[2]; Gaurav Gupta[3]; Gary S. Was[3]; S. Shutthanandan[4]; S. Thevuthasan[4]; [1]University of Wisconsin, Engrg. Physics, 1500 Engrg. Dr., Madison, WI 53706 USA; [2]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403-2528 USA; [3]University of Michigan, 2522 Bonisteel Blvd., Ann Arbor, MI 48109 USA; [4]Pacific Northwest National Laboratory, Richland, WA USA

HCM12A is an advanced 12 Cr ferritic-martensitic steel designed for higher temperature operation and is under consideration for application in core components in Generation IV nuclear energy systems. This work provides information on the hardening and microstructural changes in HCM12A after irradiation using 2.0 MeV protons at 400°C to 10 dpa, at 500°C to 3 dpa, and 5 MeV Ni-ions at 500°C to 50 dpa. Following irradiation, changes in hardness were measured using Vickers hardness indentation, changes in microstructure and phase stability were studied using transmission electron microscopy, and changes in microchemistry were measured using scanning Auger microscopy. The hardness at 400°C increases by roughly 70% and saturates by roughly 5 dpa. The changes to the microstructure contributing to this hardness increase are mainly the formation of precipitate phases. Chromium is enriched at grain boundaries prior to irradiation, likely due to grain boundary carbides, and increases further during irradiation.

11:40 AM
Microstructural Evolution of Proton Irradiated T91: *Alfred Neal Ham*[1]; Gary S. Was[1]; Gaurav Gupta[1]; [1]University of Michigan, Nucl. Engrg. & Radiologl. Scis., 2940 Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48105 USA

Ferritic-Martensitic alloys are proposed as candidate structural materials for Generation IV reactors, fusion reactors, and accelerator-driven transmutation systems. They are preferred over austenitic stainless steels in these applications due to improved irradiated and mechanical properties such as reduced swelling, high temperature creep resistance, and thermal shock resistance. Understanding radiation effects in these alloys is critical for their success in the advanced reactor and transmutation systems. The objective of this work is to evaluate the microstructural, microchemical, and mechanical property changes in ferritic-martensitic alloy T91 after irradiation. In previous studies, as-received T91 samples have been irradiated using 2.0 MeV protons at 400°C and 450°C to 10 dpa. The hardness at 400°C increases by roughly 78% and saturates by approximately 7 dpa. After 10 dpa at 450°C, the average loop size was 13.4 nm with a dislocation density of 4 x 1021 cm-3. No voids or precipitates are observed following irradiation. Hardness increased approximately 95 kg/mm2 after the same dose. Further irradiations will be performed using 2.0 MeV protons at 500°C to 3 dpa on A/R T91 and coincident site lattice enhanced (CSLE) T91 samples and at 450°C to 10 dpa on He pre-implantation (to 1800 appm) T91. This will provide data as a function of temperature and dose, with and without He pre-implantation and CSL enhancement. Following irradiation, changes in hardness will be measured using Vickers hardness indentation and changes in microstructure, microchemistry and phase stability will be studied using transmission electron microscopy.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Computational Tools for Understanding Diffusion Mechanisms

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Wednesday AM Room: 3007
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Ursula R. Kattner, National Institute of Standards and Technology, Metall., Gaithersburg, MD 20899-8555 USA; John A.L. Ågren, Royal Institute of Technology, Dept. of Matls. Sci. & Engrg., Stockholm 100 44 Sweden

8:30 AM Invited
Effective Diffusivity of Heterogeneous Systems: *Y. Mishin*[1]; [1]George Mason University, Sch. of Computatl. Scis., MSN 5C3, 4400 Univ. Dr., Fairfax, VA 22030 USA

We address two related problems dealing with calculations of "effective" diffusion coefficients. One is diffusion in disperse heterogeneous alloys whose phases possess different diffusivities. Assuming that the phase distribution is periodic and diffusion is locally quasi-steady, we show that the average concentration follows an effective diffusion equation. A mathematical procedure for exact and approximate calculations of the effective diffusion tensor is presented and applied to diffusion in a periodic array of cubic particles (prototype of Ni-based superalloys). This analysis can be readily extended to include segregation and driven diffusion. The second problem is diffusion in periodic atomic structures with multiple jump rates, such as grain boundaries, dislocations, or complex crystal structures. A procedure for averaging the spectrum of jump rates to obtain the effective diffusivity is presented and demonstrated by several applications. The continuum and atomistic theories have much in common and are applicable to other properties of heterogeneous systems.

9:00 AM
A Self-Consistent Mean-Field Model for Strongly Concentration Dependent Phenomenological Coefficients: Application to Interdiffusion Kinetics: *Vincent Barbe*[1]; Maylise Nastar[1]; [1]Commissariat à l'Energie Atomique, Service de Recherches en Métall. Physique, Gif-sur-Yvette 91191 France

Starting from an atomic diffusion model which depends on the local composition through the thermodynamic interactions between atoms, we derive the phenomenological coefficients in a multicomponent alloy. The model is based on the master equation and a self-consistent estimation of the non-equilibrium distribution function which is expressed in terms of an effective Hamiltonian (Nastar et al, Phil Mag A 80 (2000) 155). A new decoupling scheme of the kinetic equations is applied, which yields to the prediction of a percolation threshold for the tracer diffusion when the ratio of atom-vacancy exchange frequencies goes to infinity. The resulting transport coefficients show a good agreement with recent Monte Carlo simulations in the particular case of a random lattice gas. We present an application of this self-consistent theory to the prediction of kinetics of interdiffusion of the

ternary alloy Fe-Ni-Cr specifically with a high Ni content to investigate the effect of chromium percolation.

9:25 AM
Reversible and Irreversible Reaction Fronts in Quasichemical Theory of the Multicomponent Diffusion: Misha Sinder[1]; Joshua Pelleg[1]; Sergey Genikhov[1]; [1]Ben Gurion University of the Negev, Matls. Engrg. Dept., PO Box 653, Beer Sheva 84105 Israel

Many systems in physics, chemistry and materials science may be studied by using the concept of the reaction front formed between initially separated reactants. The numerous examples of such applications are known: diffusion flames, internal oxidation of the metals, gas absorption with chemical reactions in liquids, new phase formation in solids, multicomponenets diffusion in semiconductors etc. Recently some new aspects of the reaction front concept have been elucidated.[1-3] The main progress are a simple way to calculate the reaction rate for reversible reactions case and a possibility to analyze the relations between the reversible and irreversible reaction patterns. By this approach the arbitrary reversible reaction - diffusion systems with initially separated reactants may be investigated in detail. The single reversible reaction A + B = C was considered in works.[1-3] In this research the model of the system with initially separated components and two reversible reactions A + B = R and R + B = S is studied. The analysis is done in the framework of the formalism developed in Ref. [3]. Assuming the same diffusion constants of the components, the analytical solution is investigated. On the basis of the obtained results, the relationship between the reversible and the irreversible regime patterns has been determined. Thus, it is shown possibility to predict the long-time behavior of the irreversible or partly irreversible reactions system with two reactions on the basis of the reversible reactions analytical solution. The presented approach could be a useful tool for the study of the multicomponent diffusion accompanied by the quasichemical reactions in solids. [1]M. Sinder and J. Pelleg, Phys. Rev. E 60, R6259 (1999). [2]M. Sinder and J. Pelleg, Phys. Rev. E 61, 4935 (2000). [3]Z. Koza, Phys. Rev. E 66, 011103 (2002).

9:50 AM Break

10:05 AM Invited
Dynamics-Based Microstructural Design in Multicomponent Alloys: *Greg Olson*[1]; [1]Northwestern University/QuesTek, Matl. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60015 USA

Advances in the theory of coarsening kinetics in multicomponent systems have significantly improved the parametric computational design of materials as dynamic systems. These advances have been incorporated in the PrecipiCalc code successfully applied to accelerated thermal process optimization at the component level. Rapid simulations of multicomponent precipitation behavior employing a mean-field approximation can be subsequently refined by rigorous DICTRA simulations of detailed multicomponent diffusion. Development of multicomponent thermodynamic and mobility databases has enabled parametric design of oxidation-resistant Nb-based alloys, and efficient simulation-based process control of the commercial high-temperature carburizing of novel high-alloy steels.

10:35 AM
Analysis of Cu-Ni-Zn Ternary Diffusion Couples Using MultiDiFlux Program: *Kevin M. Day*[1]; Mysore A. Dayananda[1]; [1]Purdue University, Sch. of Matls. Engrg., 501 Northwestern Ave., W. Lafayette, IN 47907-2044 USA

Single phase as well as multiphase diffusion couples assembled with alpha (fcc) and beta (bcc) Cu-Ni-Zn alloys were investigated at 775°C for controlled interdiffusion and development of zero-flux-planes for the individual components. The couples were analyzed by using the MultiDiFlux program developed by Dayananda and Ram-Mohan. Interdiffusion fluxes and average interdiffusion coefficients were calculated over selected concentration ranges from various couples. Such calculations will be presented to demonstrate the capabilities of the MultiDiFlux program. The program will also be employed in the reproduction of concentration profiles on the basis of error functions and the calculated interdiffusion coefficients.

11:00 AM
Effects of Concentration-Dependence of the Effective Diffusivity on Diffusion Paths in Two-Phase Ternary System: *Hongwei Yang*[1]; John E. Morral[2]; [1]University of Connecticut, Dept. of Matls. & Engrg., 97 N. Eagleville Rd., Unit 3136, Storrs, CT 06269 USA; [2]Ohio State University, Dept. of Matls. Sci. & Engrg., 177 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

In current work, variation of the effective diffusivity with concentration was studied for one hypothetical and two real ternary systems. In order to determine the effects of concentration-dependence of the effective diffusivity on diffusion paths, a DICTRA finite difference simulation of the diffusion paths, which accounts for diffusivity variations was compared to an error function prediction, which assumes a constant effective diffusivity. The effects changed the diffusion path from linear "zig-zag" shape predicted by error function theory to the formation of "horns" in the vicinity of initial interface. The horns may protrude to either the same direction or the opposite direction. Comparison on the results from all three ternary systems shows that how the effective diffusivity varies with concentration is only necessary but not enough condition to led to different pointing directions of the horns.

11:25 AM
Diffusion in fcc and $L1_2$ Phases of Ni-Al-Mo: *T. Wang*[1]; S. H. Zhou[1]; J. Z. Zhu[1]; L. Q Chen[1]; Z. K Liu[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

Diffusion in disordered fcc and ordered $L1_2$ phases is a key factor for the high-temperature applications of Ni-base superalloys and is modeled in the present work for the Ni-Al-Mo ternary system. For the fcc phase, atomic mobility in the Ni-Mo and Al-Mo binaries is evaluated from the experimental data in the literature, and the previous modeling in the Ni-Al system is compared with recent experimental results in the literature, and the thermodynamic factors needed for modeling are calculated from the recent Ni-Al-Mo thermodynamic database.[1] For the $L1_2$ phase, the effect of chemical ordering on atomic mobility is described by a phenomenological model.[2] The available experimental data for Ni3Al are used to evaluate model parameters. The diffusion of Al in $L1_2$ is simulated, indicating the anti-site mechanism being dominant. The atomic mobility modeling of Al is then refined based on the anti-site mechanism. As $L1_2$ is not stable in the Ni-Mo and Al-Mo binary systems, no interaction parameter is introduced. By combining the above binary results, atomic mobility in fcc and $L1_2$ are obtained for the Ni-Al-Mo ternary system, and diffusion processing in the ternary system are simulated. [1]S. H. Zhou, Y. Wang, T. Wang, J. Z. Zhu, L. Q. Chen and Z. K. Liu, In Submit, (2004). [2]T. Helander and J. Agren, Acta Mater, 47 (1999) 1141-1152.

Neutron Diffraction Characterization of Mechanical Behavior: Residual Stress I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Wednesday AM Room: 3004
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Judy W.L. Pang, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; Sven C. Vogel, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

8:30 AM Invited
Stress Measurements in Welds: Problem Areas: *Thomas M. Holden*[1]; Hiroshi Suzuki[2]; David G. Carr[3]; Maurice I. Ripley[3]; [1]Northern Stress Technologies, 208, Pine Point Rd., Deep River, Ontario K0J 1P0 Canada; [2]Japanese Atomic Energy Research Institute, Tokai Japan; [3]Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW Australia

There have been many stress measurements on welds by neutron diffraction over the past 20 years but there are a number of serious experimental issues that are often not addressed. The primary fact is that the microstructure generally changes across the weld and accompanying this may be a change in the concentration of strengthening elements in solution. This will lead to a shift in lattice spacing which may be incorrectly interpreted as a strain. Secondly, a gradient of plastic deformation near the weld is expected. Since plastic deformation always generates intergranular (type-2) strains this leads to a range of intergranular effects superposed on the conventional weld-related strains. These effects are illustrated by neutron diffraction studies of ferritic, austenitic and Zr-alloy welds where chemistry, inter-

granular effects and often strong crystallographic texture all play a role.

8:50 AM Invited
Residual Stresses in Inertia and Linear Friction Welded Aeroengine Components: *Philip John Withers*[1]; Michael Preuss[1]; Philipp Frankel[1]; [1]Manchester University, Manchester Matls. Sci., Grosvenor St., Manchester SK6 7DT England

Inertia and linear friction welding are candidates for welding 'difficult-to-weld' aeroengine materials used for components such as disc to disc and blade to disc respectively. Furthermore, they allow a movement to new design approaches such as bladed rings and discs. Safe operation of these components requires a good knowledge of the residual stresses that develop during welding and through subsequent post weld heat treatment. In this paper we describe neutron diffraction measurements aimed at measuring the evolution of these stresses. In addition the range of these stresses are compared to the scale of the micrsotructural changes in the near weld zone. Results for both Ni base superalloys and Ti alloys will be presented.

9:10 AM
Processing, Microstructure, Hardness, and Residual Strains of 6061-T6 Aluminum Alloy Friction Stir Welds: *W. Woo*[1]; H. Choo[1]; D. W. Brown[2]; Z. Feng[3]; P. K. Liaw[1]; S. A. David[3]; C. R. Hubbard[3]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830 USA

Friction stir welding (FSW) is a solid-state joining process, which creates a strong bond through severe plastic deformation and frictional heating. The residual stresses (RS) in FSW can approach the yield point of the base material and cause a drastic increase in the crack-growth rate. In this study, we prepared three different weld specimens from 6061-T6 aluminum alloy: (Case 1) a plate processed with both the stirring pin and pressing shoulder, i.e., a regular FSW, (Case 2) a plate processed only with the pressing shoulder, and (Case 3) a plate processed only with the pin. Three residual strain components (longitudinal, transverse, and through-thickness) were measured across the weld line and through the thickness of the specimen using neutron diffraction. The comparison among the three cases shows distinctly different strain profiles revealing de-convoluted effects of the different sources of the residual stress, i.e., plastic deformation, friction heat, or their combination. The relationship among the processing parameters, microstructure, hardness, and sources/distributions of the residual strains in FSW will be discussed.

9:30 AM Cancelled
Neutron Diffraction Strain Measurement in High Strength Steel Welds - A Tool to Aid in Life Prediction

9:50 AM Break

10:10 AM Invited
High-Performance Kirkpatrick-Baez Super Mirrors for Neutron Milli- and Micro-Beams: *Gene Emery Ice*[1]; Camden R. Hubbard[1]; Bennett C. Larson[2]; Judy W.L. Pang[1]; John D. Budai[3]; Stephen Spooner[3]; Sven C. Vogel[4]; Ronald Roggie[5]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Rm. B260, Bldg. 4500S, MS 6118, Oak Ridge, TN 37831-6118 USA; [2]Oak Ridge National Laboratory, Condensed Matter Sci. Div., Rm. B200, Bldg. 3025M, MS 6030, Oak Ridge, TN 37831-6030 USA; [3]Oak Ridge National Laboratory, Oak Ridge, TN USA; [4]Los Alamos National Laboratory, LANCE, PO Box 1663, Los Alamos, NM 87545 USA; [5]Chalk River Laboratories, Bldg. 459, Sta. 18, Chalk River, Ontario K0J1J0 Canada

Kirkpatrick-Baez neutron super mirrors can efficiently focus neutron beams into small areas with a maximum divergence that is limited by the mirror critical angle. The size of the focal spot is primarily determined by geometrical demagnification of the source and by figure errors in the mirror shape. Ray tracing calculations show that high-performance Kirkpatrick-Baez supermirrors can preserve neutron-source brilliance when focusing down to tens of microns and can focus ~ two orders of magnitude greater power into a 100 micron spot than is practical without focusing. The predicted performance is near the theoretical limit set by the source brilliance. We describe the phase space arguments, ray tracing calculations and actual performance of an M3 supermirror system designed to produce a focal spot below 100 microns. Although the current design is optimized for neutron polychromatic microdiffraction, the design principles are widely applicable to a range of neutron science. Some example experiments that exploit the increased flux density of these optics are described.

10:30 AM Invited
Dynamical Diffraction Effects in Neutron Stress Analysis: Ersan Ustundag[2]; Mark R. Daymond[3]; *I. C. Noyan*[1]; [1]Columbia University, Dept. of Applied Physics & Applied Math., 500 W. 120th St., New York, NY 10027 USA; [2]Iowa State University, Dept. of Matl. Sci. & Engrg., 2220 Hoover Hall, Ames, IA 50011-2300 USA; [3]ISIS-Rutherford-Appleton Labs, ENGIN-X, Didcot England

In this presentation we will discuss effects of grain size on the accuracy and precision of stress results obtained with neutron diffraction. In particular, the effects of beam divergence and dynamical diffraction artifacts will be discussed. These effects will be compared to those in x-ray diffraction. Guidelines for avoiding these effects will also be presented.

10:50 AM Invited
Improving the Structural Integrity of Welded Structures Through Reliable Residual Stress Measurements: *Lyndon Edwards*[1]; [1]Open University, Dept. of Matls. Engrg., Walton Hall, Milton Keynes MK7 6AA UK

Many of the factors controlling the structural integrity of welded structures are reasonably well understood and the importance of residual stress is well known. However, previous access to reliable, spatially accurate residual stress field data has been limited so that standards controlling the design of welded structures are typically very conservative. Recent advances in neutron diffraction allow a far more detailed picture of weld residual stress fields to be obtained in increasingly large components and structures. This permits the development and use of predictive models that can be used for the accurate design of new structures or the life extension of legacy plant. This paper uses examples from the aeronautical and nuclear industries to illustrate how fully integrated studies of the 3D residual stress distribution accompanying fusion welds and how they affected by subsequent manufacture and/or service environments can be used to improve the structural integrity of welds structures.

11:10 AM
Residual Stress Measurements in Gas Pipeline Girth Welds: *Michael Law*[1]; [1]Australian Nuclear Science and Technology Organisation, Matls. & Engrg. Sci., PMB1, Menai, NSW 2234 Australia

Gas pipelines have an enviable safety record in transporting energy, however there is still room for improvement. Welding is used extensively in gas pipelines, the welds are made without post weld heat treatment. The welding process generates significant residual stresses along the welding direction with the maximum values occurring in the weld and heat affected zone. The stresses are lower in the axial direction (perpendicular to the weld) but these residual stresses have the most significant effect on fracture and the allowable defect size. Reliable fracture mechanics assessments can only be performed when the residual stresses are known accurately. Neutron residual stress measurements offer many advantages in determining these stresses: the great penetrating power and non-destructive nature of the measurement means that stresses can be measured at a range of depths with good spatial resolution. The residual stresses in the welds are superimposed on those generated by the considerable working during rolling and pipe forming. In the present work, the stress in un-welded pipe, and in girth welded gas pipelines were measured by neutron diffraction. The welds are manual metal arc cellulosic electrode welds made in X70 linepipe with 3 differing wall thicknesses (7.1 mm, 6.4, and 5.2 mm). The full stress tensor was determined for 2 types of sample: - 3 girth welded rings cut from linepipe, and 3 corresponding unwelded rings. The unwelded rings were measured at 5 through-thickness positions with a spatial resolution of 1 mm3. The welded rings were measured at the same 5 through-thickness positions at 19 locations from the center of the weld up to 35 mm away from the weld. The results will be presented and compared.

11:30 AM
The National Science Foundation Major Research Instrumentation [MRI] Program on "Development of an In-Situ Neutron-Scattering Facility for Research and Education in the Mechanical Behavior of Materials": *Peter K. Liaw*[1]; Hahn Choo[1]; Raymond A. Buchanan[1]; Camden R. Hubbard[2]; Xun-Li Wang[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 427-B Dougherty Engrg. Bldg., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830 USA; [3]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37830 USA

A National Science Foundation [NSF] Major Research Instrumentation [MRI] Program on "Development of an In-Situ Neutron-Scattering Facility for Research and Education in the Mechanical Behavior of

Materials" is proposed by the University of Tennessee [UT], Oak Ridge National Laboratory [ORNL], and their team members. The collaborating participants come from 16 different universities, 13 industries, and 6 national laboratories with diverse disciplines that include [1] Materials Science and Engineering; [2] Physics; [3] Chemistry; [4] Mechanical, Aerospace, and Biomedical Engineering; [5] Civil Engineering; [6] Nuclear Engineering; and [7] Biochemistry. A four-year development Program has been funded by NSF to establish a suite of instruments for studying mechanical behavior of advanced materials using in-situ neutron scattering at the impending Spallation Neutron Source [SNS, ORNL], which, when completed in 2006, will provide the most intense pulsed-neutron source in the world. The objective of this program is to develop the state-of-the-art in-situ and real-time characterization instrumentation for VULCAN at SNS. The VULCAN diffractometer is designed to conduct fundamental studies in materials science and engineering with a focus on mechanical behavior. The proposed instrumentation will enable VULCAN to fulfill its full potential. The underlying goal of this proposal is to [1] fully exploit the best US neutron-source capabilities, [2] advance neutron science and engineering by providing the most-advanced neutron-scattering instrumentation to the broadest possible materials-research community, and [3] broaden the participation at the frontiers of the scientific/engineering enterprise by educating a diverse cadre of individuals skilled in the applications of advanced, state-of-the-art neutron technologies for fundamental materials studies. We are very grateful to NSF for the support [DMR-0421219]. Drs. Charles E. Bouldin and Guebre Tessema are the Program Directors.

Neutron Scattering in Materials Research: Dynamics and Inelastic Scattering

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Wednesday AM Room: 3022
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Ken W. Herwig, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831-6474 USA; Ron R. Berliner, North Carolina State University, Nucl. Reactor Prog., Raleigh, NC 27695-7909 USA

8:30 AM
Dynamical Sources of Entropy in Phase Transformations in Materials: *Brent T. Fultz*[1]; [1]California Institute of Technology, Mail 138-78, Pasadena, CA 91125 USA

Temperature drives the excitations of phonons, spins, and electrons. In thermal equilibrium these vibrational, magnetic, and electronic contributions to the entropy can be assessed if their energy spectra are known. Each of them can make a significant contribution to the entropy of phase transformations in materials, comparable to the contribution from the configurational entropy that is well known from textbooks. This talk explains how inelastic neutron scattering experiments are used to measure the energy spectra of phonons and other dynamic excitations in materials. Some thermodynamic trends are presented, such as the effect of solute mass and atomic size mismatch on phonon entropies of compound formation. Electronic entropy plays a big role in crystal structure transformations in cerium, uranium and plutonium. Overall, however, an understanding of the entropy of solid state phase transitions requires detailed analyses. Rules of thumb are, unfortunately, inadequate. An excellent opportunity remains for further experimental work.

9:00 AM Invited
Measuring Medium and High Energy Excitations in Condensed Matter Using Spallation Neutrons: *Douglas L. Abernathy*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, PO Box 2008, Oak Ridge, TN 37831-6474 USA

The traditional instrument for measuring inelastic neutron scattering from condensed matter systems has been the reactor-based triple axis spectrometer. More recently, time-of-flight techniques have been developed at spallation neutron sources, which produce thermal and epithermal neutrons in short pulses. The Spallation Neutron Source (SNS) is an accelerator-based neutron source currently under construction at the Oak Ridge National Laboratory in Tennessee. It will provide the most intense pulsed neutron beams in the world for scientific and industrial research and development. Two instruments under development for this facility will specialize in measuring high energy excitations in materials. The operating principles of these instruments will be presented, along with the different optimizations of the two to access a broad wavevector transfer range and offer high resolution for magnetic scattering studies. An example of how single crystal measurements are made on these instruments will be given.

9:30 AM Invited
Thermodynamics of Metals with Strong Electron-Phonon Interactions: *Michael Edward Manley*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. - 6, MS G770, Los Alamos, NM 87545 USA

Contrary to prevailing wisdom, recent inelastic neutron scattering measurements of phonons indicate that electron-phonon coupling plays a major role in the high-temperature thermodynamics of several actinides and at least one rare-earth. Results are forcing us to rethink the way we approach the thermodynamic problem. For example, the heat capacities of electrons and phonons cannot be separated and the exchange between the phonon entropy and electron energy results in large enhancements of the free energy. For uranium, I will present evidence of the coupling from the perspective of both phonons observed with neutron scattering and electronic distributions observed in photoemission spectra. A simple statistical argument shows how extra entropy generated by phonon softening stabilizes electron distributions to energies higher than that predicted from normal Fermi-statistics. Possible sources of strong electron-phonon coupling will also be discussed.

10:00 AM Break

10:20 AM
Neutron Scattering and Density Functional Theory Study of Lattice Dynamics and Entropy in Dilute Vanadium Alloys: *Olivier Delaire*[1]; Tabitha L. Swan-Wood[1]; Max Kresch[1]; Brent T. Fultz[1]; [1]CALTECH, Matls. Sci., 1200 E. California, MC 138-78, Pasadena, CA 91125 USA

In this study, we investigate the lattice dynamical and entropic effects of alloying dilute impurities into pure bcc vanadium. Using inelastic neutron scattering, we have measured the changes in the phonon density of states (DOS) and entropy of vanadium associated with the alloying of a few percent impurities of the 3d, 4d and 5d series transition metals. A clear trend was observed across the d-series, Ti, Zr and Hf causing a softening of the V phonon DOS, while elements to the right of V in the periodic table induce a stiffening gradually increasing with atomic number. The DOS stiffening observed for 6% of Co or Pt impurities is very large and results in a decrease of vibrational entropy that is larger than the configurational entropy gain of alloying. These changes in the phonons were analyzed both with classical Bornvon Karman lattice dynamics models and with density functional theory computer simulations, using supercells to describe the alloys. We report our findings on the origin of the perturbation of V phonons by transition metal impurities and discuss the consequences on the thermodynamics and transport properties.

10:45 AM
Neutron Scattering Study of the Martensitic Phase Transformation in Fe71Ni29: Olivier Delaire[1]; *Max Kresch*[1]; Tabitha L. Swan-Wood[1]; Brent T. Fultz[1]; [1]California Institute of Technology, Matls. Sci., MC 138-78, Pasadena, CA 91125 USA

Martensitic transformations occur via cooperative and well organized shears of the crystal. Although atoms are displaced, there is no significant change in the disorder of atomic configurations. The entropy of these phase transitions can be entirely vibrational in origin. Inelastic neutron scattering was used to measure elementary exitations in both face centered cubic (fcc) austenite and body centered cubic (bcc) martensite phases of Fe71Ni29. Phonon densities of states (DOS) for the two phases were found by analysis of the incoherent scattering. Comparison of the two room temperature DOS showed a significant change in vibrational entropy: DS(fcc->bcc) = -0.13 kB/atom. Born von Karman (BvK) models of the lattice dynamics were used to relate thermodynamic data to interatomic force constants, which stiffen in the BCC phase. These BvK models took advantage of both coherent and incoherent scattering. We discuss the thermodynamic importance of the results.

11:10 AM
Vibrational Entropy of Mixing and the Phase Stability of Al-Ag: *Tabitha Liana Swan-Wood*[1]; Max Kresch[1]; Jiao Lin[1]; Mike McKerns[1]; Brent T. Fultz[1]; [1]California Institute of Technology, Matls. Sci., MS 138-78, Pasadena, CA 91125 USA

The Al-Ag system has an unusually strong temperature dependence of the solubility of Ag in fcc Al. This phenomena is seen in many binary alloys, particularly those where the impurity has a mass ratio of at least 3 to that of the matrix atom. It is possible this mass ratio causes resonance modes that stabilize the solid solution with a large vibrational entropy of mixing. In our experiments on Al-Ag, phonon density of states (DOS) were obtained from pure Al, and Al-Ag alloys. A distinct resonance mode was found in the 7% Ag DOS. In addition, the hcp phase of Al- 60% Ag showed surprisingly large softening of all phonon modes. An extremely large softening with temperature was seen in the 7% Ag while insignificant softening was seen in the 60% Ag, making a substantial contribution to the temperature dependence of the solvus in this system.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Interfacial Reactions and IMC Formation in Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Wednesday AM Room: 3016
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan; Kenneth L. Erickson, Sandia National Laboratories, Dept. 9112, Albuquerque, NM 87185 USA

8:30 AM
A Study on Intermetallic Growth Kinetics of Sn-Ag-Cu Lead-Free Alloy on Cu, Ni and Alloy-42 Substrates During Pb-Free Soldering: Nader Dariavach[1]; Paul Callahan[1]; *Jin Liang*[1]; *Raymond A. Fournelle*[2]; [1]EMC, 176 South St., Hopkinton, MA 01748 USA; [2]Marquette University, Milwaukee, WI 53201-1881 USA

Lead-free soldering with Sn-Ag-Cu alloys requires substantially higher temperatures (around 235ºC to 250ºC). The rates for intermetallic growth and substrate dissolution are expected to be significantly greater than those for the current Sn-Pb eutectic solder. This study systematically investigates the intermetallic growth kinetics for Sn3.8Ag0.7Cu lead-free solder with three different substrates: Cu, Ni and Alloy 42, at temperatures ranging from 225ºC to 280ºC for reaction times from 10 sec to 4 hrs. The kinetic reaction rates and temperature-dependency were measured experimentally and simulated with first principle and solid/liquid diffusion theories. A thorough understanding of lead-free solder/substrate interfacial reactions can lead to the optimum lead-free soldering processes and to the optimum lead-free coating thicknesses for component and PCB terminal finishes, as well as to the optimum barrier layers needed for interconnections inside of advanced packages.

9:00 AM
A Study of the Interfacial Microstructures of Sn-Ag-y%Cu Interconnects: *Henry Lu*[1]; Haluk Balkan[1]; Simmon Ng[2]; [1]Flip Chip International, LLC, 3701 E. Univ. Dr., Phoenix, AZ 85034 USA; [2]Wayne State University, Dept. of Cheml. Engrg., Detroit, MI 48202 USA

Sn-Ag-y%Cu interconnects (Cu wt% = 0.0, 0.5, 1.0, and 2.0) were studied after solid-liquid reaction. Combining mechanical testing, surface micro-etching and microanalysis, the 3-D interfacial microstructures were identified, and the relationship between interfacial microstructures and interconnect macro mechanical performances was correlated. The virgin groups had different CuSn IMC morphologies: for Cu 0.5% group, scissors-like; 1.0%, tweezer-like; 2.0%, round clusters. No Ag3Sn platelets were observed. There were no differences in mechanical behaviors. After solid-liquid reactions, there was no CuSn IMC spalling from the interface, except 0.0% group. Ag3Sn platelets were observed for 0.5% group or higher. Cu behaved like a catalyst: the higher Cu content, the more Ag3Sn platelets. CuSn IMC grew as big bricks for the 2.0% group. The interconnect mechanical performance was diverse. Fracture mode was ductile for low Cu content and brittle for 1.0% above. Abnormal growth of Ag3Sn platelets and CuSn bricks were responsible for the interconnects' mechanical degradation.

9:20 AM
Interfacial Reactions in the Pb-Free Composite Solders with Indium Layers: *Sinn-wen Chen*[1]; Shih-kang Lin[1]; Ching-feng Yang[1]; [1]National Tsing-Hua University, Chem. Engrg. Dept., #101, Sec. 2, Kuang-Fu Rd., Hsin-chu 300 Taiwan

A lead-free composite solder is prepared with lead-free solder substrate and a plated-indium layer. The indium containing layers melt during the soldering process, wets the substrates, and forms sound solder joints. Since the melting temperature of indium is 156.6°C, even lower than that of the eutectic Sn-Pb which is at 183°C, so the soldering process can be carried at a temperature lower than the conventional soldering process. During the soldering process, the molten indium alloys with its surrounded Sn based Pb-free solder alloys and then reacts with the nickel and copper substrates. The interfacial reactions between the indium alloys and the substrates are investigated, and binary intermetallic compounds are observed at the interfaces.

9:40 AM
Cross-Interaction Between Au and Cu in Au/Sn/Cu Diffusion Couple: *Chien Wei Chang*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan

It was well known that both Au and Cu are fast diffusers in many metals, including Sn. These two metals can diffuse through a long distance in Sn in a relatively short time. In this study, we would like to investigate the cross-interaction between Au and Cu across a thick layer of Sn. In other words, we would like to study the Au/Sn/Cu ternary diffusion couple. Using electroplating method, a 100 microns Sn layer was deposited over Cu plate, and then a 5 microns Au layer was deposited over the Sn layer. The diffusion couples were then aged at 100-200°C for upto 10 days. It was found that Cu and Au can start to cross-interact in a few hours. At the Au/Sn interface, Cu can be detected in the $AuSn_4$ phase. At the Sn/Cu interface, Au can also be detected in the Cu_6Sn_5 phase. The detailed reaction sequence and the mechanism will be presented. Implications related to electronic packaging will also be summarized.

10:00 AM
Study of Interfacial Reactions of AuSn/Au and AuSn/Ag for Flip-Chip RF Assembly: *Yuan-Tai Lai*[1]; *Cheng-Yi Liu*[1]; [1]National Central University, Cheml. & Matls. Engrg., No.300, Jungda Rd., Jhongli, Taoyuan 320 Taiwan

As the signal frequency in RF (Radio Frequency) device approaches several tens of GHz, flip-chip technology has become increasingly important for the chip-assembly of RF device. To meet the requirement of the fluxless process for the flip-chip assembly of RF device, AuSn solder would be the good choice to bond Au bumped RF chips. Hence, the reaction characteristics of eutectic AuSn solder with Au in air ambient are very important. Beside Au substrate, we also studied the wetting reaction between AuSn and Ag, since using Ag substrate could prevent the serious Kirkendall voids forming at the interface between AuSn/Au after thermal aging process. The experiments were carried at four different temperatures, which are 300°, 330°, 360°, and 390° for 30sec, 1min, 2min and 5min. The preliminary results indicated that AuSn has very poor wettability on Au and Ag substrate. The molten AuSn solder ball remained spherical shape on substrates. With increasing of reflowing temperature, the spherical shape of AuSn solder remained, yet, we found that the bottom of the molten AnSu started to spread and wet on substrate. According to the SEM X-section study, Au was found to dissolve into AuSn quickly during the reflowing process. Yet, Ag has much slow dissolution into AnSn comparing to Au. In this talk, we will report the detail kinetics of AuSn/Au and AuSn/Ag reactions and their wetting be.

10:20 AM Break

10:30 AM
Modeling the Effect of Finite Material Boundaries on Multi-Component Base Metal Dissolution and Inter-Metallic Com-

pound Growth: *Kenneth L. Erickson*[1]; Polly L. Hopkins[2]; Paul T. Vianco[3]; [1]Sandia National Laboratories, Dept. 9112, PO Box 5800, MS 0834, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 9114, PO Box 5800, MS 0834, Albuquerque, NM 87185 USA; [3]Sandia National Laboratories, Dept. 1861, PO Box 5800, MS 0889, Albuquerque, NM 87185 USA

Experiments examining multi-component base metal dissolution by molten solders were reported previously. Preferential dissolution of base metal constituents influenced short-term base metal erosion and long-term inter-metallic compound (IMC) growth. A modeling capability is needed to extend observed results to further applications. In one-dimensional, diffusion-controlled systems involving constant properties and time scales for which the solder is semi-infinite with respect to diffusion, interface displacement proceeds monotonically. Relative constituent concentrations in IMC's and solder are established quickly, determined by solubility, and remain constant. Governing equations can be solved analytically. In systems involving variable properties and time-scales for which the solder is finite, interface displacement proceeds non-monotonically. The direction of interface displacement can reverse. Relative concentrations of constituents can change significantly. Governing equations require numerical solution. This paper summarizes numerical simulation of these effects based on data from previously reported experiments involving 63Sn-37Pb solder and 76Au-21Pt-3Pd (wt%) alloy sheet and porous thick film.

10:50 AM
Phase Field Simulations of Liquid Channel Formation and its Influence on the Growth Kinetics of Intermetallic Compound Layer During Soldering Reactions: *Kyoung-Kook Hong*[1]; Joo-Youl Huh[1]; [1]Korea University, Div. of Matls. Sci. & Engrg., 5-1, Anam-Dong, Sungbuk-Ku, Seoul 136-701 Korea

With ever decreasing pitch size in ball grid array (BGA) technology, it has been a crucial issue in electronic packaging to control the intermetallic compound (IMC) layer growth kinetics for the solder joint reliability. In an attempt to understand the drastic difference in the IMC growth kinetics between during the soldering and aging processes, we investigated the influence of the liquid channel formation through the IMC grain boundaries during soldering reactions on the IMC layer growth kinetics using phase field simulations. Computations show that the deeper liquid channel formed into the IMC grain boundaries causes the faster growth of the IMC layer and the increased coarsening rate of IMC grains. This talk will also discuss how the formation of liquid channel into the IMC grain boundaries can be affected by the liquid solder diffusivity and the solder/IMC interface energy.

11:10 AM
Study of Copper Dissolution During Soldering Process: *Mohammad Faizan*[1]; [1]University of Akron, Mechl. Engrg., 302 Buchtel Mall, Akron, OH 44325 USA

During soldering process involving copper and Sn-rich solders, a Sn-Cu interaction takes place at the copper/solder interface. The copper-Sn soldering reaction is very intense at the beginning of the soldering process. Copper is dissolved into the molten solder and subsequently intermetallic compounds (IMCs) are formed at the interface. Due to miniaturization of electronic components and increasing space constraints in the electronic packaging devices, amount of copper available for soldering is very limited. Also, some of the copper must remain intact even after soldering to avoid the dewetting of the solder. Therefore, the rate of copper loss during soldering must be under control. Understanding of the dissolution rate of copper during soldering is critical in achieving reliable soldered joints. This paper presents a numerical analysis of the dissolution process of pure copper, when is comes in contact with the Sn-rich, lead-free molten solder. The results show non-equilibrium dissolution of copper at the early stage of the soldering process.

11:30 AM
Dissolution Behavior of Cu and Ag Substrates in Molten Solders: *Kwang-Lung Lin*[1]; Po-Yi Yeh[1]; Jenn-Ming Song[2]; [1]Cheng Kung University, Dept. of Matl. Sci. & Engrg., No.1, Ta-Hsueh Rd., 701, Tainan Taiwan; [2]Dong Hwa University, Dept. of Matl. Sci. & Engrg., 1, Sec. 2, Da Hsueh Rd., Shou-Feng, Hualien Taiwan

This study investigated the dissolution behavior, at 300~400°C, of Cu and Ag substrates wire in molten Sn-4.0Ag-0.5Cu, Sn-8.6Zn and Sn-8.55Zn-0.5Ag-0.1Al-0.5Ga lead-free solders as well as in Sn-37Pb solder for comparison. Results show that dissolution behavior follows linear kinetics. The dissolution rate was significantly lower for both Cu and Ag in the Sn-8.6Zn and Sn-8.55Zn-0.5Ag-0.1Al-0.5Ga solders than in the Sn-37Pb and Sn-4Ag-0.5Cu solders. This tendency can be attributed to the difference in the interfacial reactions between the substrates and the different solders. The interfacial intermetallic compounds (IMC) formed by the dissolution were investigated after solidification. The Sn-Zn solders produced compact layer IMC while the other solders gave rise to columnar IMCs.

Phase Transformations Within Small-Size Systems: Amorphous to Nanocrystal Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee

Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Wednesday AM Room: 3002
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Kazuhiro Hono, National Institute of Materials Science, Tsukuba 305-0047 Japan; Raju V. Ramanujan, Nanyang Technological University, Sch. of Matls. Engrg., Singapore 639798 Singapore

8:30 AM Invited
Nanostructure Synthesis During Devitrification: *J. H. Perepezko*[1]; J. Hamann[1]; W. S. Tong[1]; G. Wilde[2]; R. J. Hebert[2]; H. Roesner[2]; [1]University of Wisconsin, Dept. of Matl. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Forschungszentrum Karlsruhe, INT, PO Box 3640, Karlsruhe D-76021 Germany

The recent innovations in metallic glasses involve either bulk volumes slowly cooled to a glass under a nucleation-controlled synthesis or marginal glass formers that are synthesized under growth-controlled kinetics. The new metallic glasses serve as effective precursors to synthesize nanostructured solids. With marginal glass formers, heating often does not yield a clear glass transition, Tg, but instead there is an onset of a partial crystallization into a high number density of nanocrystals (1021-1022 m-3) of the major component (i.e. Al or Fe) within a residual amorphous matrix. The origin of the nucleation sites and the crystallization rate are key kinetics analysis issues. At the same time, alternate strategies involving intense deformation reveals another control over the primary crystallization. These developments represent a major level of microstructure control that have an impact of the structural performance and stability. The support of the ARO (DAAD 19-01-1-0486) is gratefully appreciated.

9:05 AM
Deformation-Induced Devitrification of Al-Based Amorphous Alloys: *Rainer J. Hebert*[1]; Harald Rösner[1]; Gerhard Wilde[1]; John H. Perepezko[2]; [1]Forschungszentrum Karlsruhe, Inst. für Nanotech., PO Box 3640, Karlsruhe 76021 Germany; [2]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison 53706 USA

Primary crystallization reactions in amorphous alloys require both, a driving force and a diffusive redistribution of solute atoms in the amorphous matrix. Annealing treatments at temperatures of more than 50 K below the glass transition temperature can induce Al nanocrystals in amorphous Al-Sm alloys. At even higher levels of undercooling, Al nanocrystals develop during intense plastic deformation at ambient temperatures in amorphous Al88Y7Fe5 alloys. Limits to the growth of deformation-induced nanocrystals are examined in this work based on measurements of the nanocrystal size distribution and microstructural investigations of the nanocrystals in the shear bands. A dislocation-based fragmentation process reduces the size of thermally-induced Al dendrites to a minimum size of approximately 18 nm. The formation of dislocations in deformation-induced as well as thermally-induced nanocrystals appears to be crucial for the size distribution evolution during deformation. The possibility of a dynamical steady state of the size distribution is discussed.

9:30 AM
Phase Transformations in Fe-Ni Based Nanomagnetic Systems: *Raju V. Ramanujan*[1]; S. W. Du[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Block N4.1, Nanyang Ave., Singapore 639798 Singapore

There is intense current interest in nanomagnetic materials, synthesis of such materials can be carried out by a variety of techniques; phase transformations in mechanically alloyed powders and crystallization of amorphous alloys has been utilized in this investigation. Fe-Ni based alloys with the composition Fe49Ni46Mo5, Fe42Ni40B18 and Fe40Ni38B18Mo4 were processed by mechanical alloying and melt spinning and studied by XRD, SEM and TEM techniques. Nanocrystalline f.c.c. Fe-Ni solid solution as well as amorphous material could be produced by mechanical alloying of the elemental powders. The presence of boron was found to make amorphization easier, phase transformation studies of the as milled powders showed that molybdenum increased the thermal stability. The magnetic properties were studied by VSM. The results obtained from the study of the powders will be compared to the counterpart crystallization studies of initially amorphous Fe40Ni38B18Mo4 ribbons produced by melt spinning.

9:55 AM Break

10:10 AM Invited
3DAP Studies of Amorphous Nanocrystallization - Elemental Distribution Within Small-Size Systems: *K. Hono*[1]; [1]National Institute for Materials Science, 1-2-1 Sengen, Tsukuba 305-0047 Japan

Nanocrystallization processes of amorphous alloys are widely used for processing nanocrystalline soft and hard magnetic materials, and nanocrystalline ultrahigh strength alloys. Recent studies also reported that many bulk-forming metallic glasses develop nano(quasi)crystalline microstructure after devitrification. In this talk, we will give an overview on our recent studies on the nanocrystallization processes of various amorphous and glassy alloys investigated by the three dimensional atom probe (3DAP) technique complemented by high resolution electron microscopy (HREM). The final nanocrystalline microstructures are very sensitive to alloy compositions, microalloyed elements and heat treatment conditions. Using 3DAP, the distributions of solute atoms in the course of the crystallization processes of Fe, Al, Cu and Zr based amorphous and glassy alloys were analyzed with an atomic resolution, and the overall microstructures were examined by TEM. The factors influencing the nanocrystalline microstructures and the structure-property relationship of nanocrystalline alloys were clarified, by which the optimizations of the microstructures and properties were successfully made in (Fe,Co)-based soft magnetic materials. The possibility and the potential applications of a glass phase separation in bulk-forming metallic glasses will also be discussed.

10:45 AM Invited
Magnetic and Structural Phase Transformations in Nanocrystalline Soft Magnetic Materials: *Matthew A. Willard*[1]; Ramasis Goswami[1]; Vincent G. Harris[2]; [1]Naval Research Laboratory, Code 6324, 4555 Overlook Ave., SW, Washington, DC 20375 USA; [2]Northeastern University, Electl. & Computer Engrg. Dept., Boston, MA 02115 USA

Alloys consisting of nanocrystallites surrounded by a residual amorphous matrix have been produced by rapid solidification processing with subsequent isothermal annealing. The resulting materials, made up of (Fe,Co,Ni)-(Zr,Nb)-B-Cu, possess both high permeability and low core losses making them excellent soft magnetic materials. Characterization of this class of alloys reveals abundant phase transformations, including, primary and secondary crystallization, ferromagnetic-to-paramagnetic phase transformations, polymorphic phase transformations, and order/disorder transformations. This study focuses on magnetic and structural phase transformations and their consequential effects on the properties of these materials. Differential thermal analysis, thermomagnetic analysis, x-ray diffraction, and transmission electron microscopy will explicate the transformations, product phases, and microstructures of these alloys.

11:20 AM
Phase Transformation in Sputter Deposited Al-Zr Thin Film: J. H. Yang[1]; *Y. Yang*[1]; C. X. Ji[1]; Y. A. Chang[1]; [1]University of Wisconsin, Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI USA

The sputter-deposited Al-Zr thin films have been reported in the literature to exhibit either a crystalline or amorphous structure at different compositions. In this study, we first present a thermodynamic argument to calculate the compositions of these binary alloys with tendencies to form amorphous phase. Thin-films of Al-Zr alloys having the compositions predicted by these calculations were prepared by co-sputtering of Al and Zr. These films were found to be amorphous as characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), and Auger analysis (AES). The thermal stability of these amorphous films has been examined by differential scanning calorimetry (DSC).

Powder Metallurgy Research and Development in the Transportation Industry: Titanium Alloys - P/M Developments

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Powder Materials Committee
Program Organizer: James W. Sears, South Dakota School of Mines & Technology, Additive Manufacturing Laboratory, Rapid City, SD 57701 USA

Wednesday AM Room: 3008
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Eugene A. Olevsky, San Diego State University, Mechl. Engrg. Dept., San Diego, CA 92182-1323 USA

8:30 AM
Influence of Si on the Microstructure of Reactive Sintered TiAl: *David E. Alman*[1]; [1]U.S. Department of Energy - Albany Research Center, 1450 Queen Ave., SW, Albany, OR 97321 USA

TiAl with between 0 and 20 volume percent (v%)Ti5Si3 was produced by reactive sintering of cold pressed compacts of elemental Ti, Al and Si powder mixtures at 700C for 15 minutes in vacuum. The results show that adding Si to Ti and Al reduces the swelling associated with reactive sintering of TiAl, as composites containing more than 5 v%Ti5Si3 densified during reactive sintering. However, composites containing more than 10v% Ti5Si3 did not retain their shape during processing, and the TiAl+20v% Ti5Si3 composite completely melted during the sintering process. The formation of pre-reaction liquid phase and the increase in adiabatic flame temperature with simultaneous compound formation resulted in the melting that occurred and the enhanced densification (minimization of swelling) during reactive sintering of the insitu composites.

8:55 AM
Low-Cost PM Titanium Materials for Automotive Applications: *Orest M. Ivasishin*[1]; Dmytro G. Savvakin[1]; Vladimir S. Moxson[2]; Vlad A. Duz[2]; F. H. Froes[3]; Richard W. Davies[4]; [1]Institute for Metal Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [2]ADMA Products, Inc., 1890 Georgetown Rd., Hudson, OH 44236 USA; [3]University of Idaho, Inst. for Matls. & Advd. Processes, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [4]Pacific Northwest National Laboratory, 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

Titanium alloys are attractive materials for a wide range of applications in automotive industry under condition of noticeable reduction of cost of components made out of them. Blended elemental powder metallurgy (BEPM) is a viable way to achieve this goal. In present study Ti-6Al-4V, Ti-5Al-2.5Fe, and some binary Ti-Mn alloys were synthesized with simplest press-and-sinter BEPM technique. The approach used was based on employment of hydrogenated titanium powder to which alloying elements were added either as elemental or master alloy powders. Minimal content of hydrogen in titanium sufficient to radically influence on the synthesis of titanium based alloys was determined. The advantage of hydrogenated titanium approach in attaining high dense (99%) condition which tensile and fatigue properties are matching the level of corresponding alloys produced with conventional ingot metallurgy will be discussed. Hydrogenated titanium based technology is planned to be tried in manufacturing of various automotive components.

9:20 AM
Fabrication of Titanium Automotive Parts by Laser Powder Deposition: Matt Heath[2]; Bryan Woods[2]; Seth Miller[2]; Aaron Costello[1]; Daniel F. Dolan[2]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technology, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines & Technology, Mechl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Fabrication of titanium components by Laser Powder Deposition (LPD) offers some unique solutions for high performance automotive applications. LPD is a CAD/CAM solid freeform fabrication technology that uses metal powder and laser fusion to produce components. This paper describes the fabrication of titanium engine valves and wheel uprights. LPD's unique capabilities allow direct fabrication of titanium components with design features that are difficult to obtain by other technologies. It has been shown that the titanium valves can be produced lighter and more wear resistant and the wheel uprights 7 times stiffer than existing aluminum designs.

9:45 AM
Processing and Consolidation of Titanium Based Metal-Ceramic Composite, Intermetallic and Alloy Powders for Applications in Transport Industry: *Deliang Zhang*[1]; Zhihong Cai[1]; Gorgees Adam[1]; Stiliana Raynova[1]; Zhiguang Liu[1]; [1]University of Waikato, Dept. of Matls. & Process Engrg., PB 3105, Hamilton, Waikato 2001 New Zealand

Light metallic materials play vital roles in improving the energy efficiency of vehicles used for transport. Titanium based materials are one type of light metallic materials which have not been utilised to their full potential for applications in transport industry because of the limitation of high materials costs and high component manufacturing costs. At Waikato University, we have been working on developing new processes which produce titanium based metal-ceramic composite (such as Ti(Al,O)/Al2O3 and TiAl/Al2O3 composites), intermetallic (such as Ti3Al/TiAl and TiAl), and alloy (such as Ti-6wt%Al-4wt%V) materials directly from low cost raw materials such as titanium oxide and aluminium powders. The new processes involve processing titanium based powders using a combination of several physical and chemical steps, and the powders are consolidated into bulk materials or near net shaped components using novel powder metallurgy processes. This paper will provide an overview of the recent findings and outcomes of the research programme, and discuss the opportunities and issues of using the new materials and the new processes for manufacturing automotive components.

10:10 AM Break

10:20 AM
Synthesis and Characterization of Nanocrystalline Ti Alloys by a Cryomilling Technique: *Fusheng Sun*[1]; P. Rojas[1]; A. Zuniga[1]; E. J. Lavernia[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

The paper reports on the synthesis and characterization of nanocrystalline Ti alloys using a cryomilling technique. The as-received Ti powders with an average powder particle size of 75 mm were cryomilled in a modified Union Process 1-S attritor under a liquid nitrogen medium. The effects of processing parameters such as milling time and ball to powder ratio on the synthesis and structure of the cryomilled Ti powders were investigated. The microstructural evolution of the cryomilled Ti powders were analyzed using XRD, DSC, SEM, and TEM. The results showed that nanocrystalline Ti powders with a grain size of about 5-20nm can be obtained using the cryomilling technique. The grain size and particle size of the cryomilled Ti powders decrease with increasing cryomilling time.

10:45 AM
A Comparison of the Sintering of Various Titanium Powders: *Stephen J. Gerdemann*[1]; David E. Alman[1]; [1]US Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

Recently, there has been renewed interest in low-cost titanium. Near-net-shape powder metallurgy offers the potential of manufacturing titanium articles without costly and difficult forming and machining operations; hence, processing methods such as conventional press-and-sinter, powder forging and powder injection molding are of interest. The sintering behavior of a variety of commercial and experimental titanium powders was studied. Commercial powders were acquired that were produced different routes: (i) sponge fines from the primary titanium processing; (ii) via the hydride-dehydride process; and (iii) gas atomization. The influence of vacuum sintering time (0.5 to 32 hrs) and temperature (1200, 1275 or 1350°C) on the microstructure (porosity present) of cold pressed powders was studied. The results are discussed in terms of the difference in powder characteristics; with the aim of identify the characteristics required for full density via press-and-sinter processing. Near-net-shape tensile bars were consolidated via cold pressed and sintered. After sintering, a sub-set of the tensile bars was hot-isostatic pressed (HIPed). The microstructure and properties of the bars were compared in the sintered and HIPed conditions.

11:10 AM
A Novel Method of Injection Molding of Titanium Components: *K. Scott Weil*[1]; Eric Nyberg[1]; Kevin Simmons[1]; [1]Pacific Northwest National Laboratory, Matls. Dept., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

One of the key problems in the powder injection molding (PIM) of titanium components is to minimize the introduction of carbon impurities, because of the deleterious effect they impart on subsequent mechanical properties. We have developed a unique blend of PIM constituents where only a small volume fraction of binder (~5 - 10 vol%) is required for injection molding; the remainder of the mixture consists of the metal powder and binder solvent. Because of the nature of decomposition in the binder system and the relatively small amount used, the binder is eliminated almost completely from the pre-sintered component during the initial stage of a two-step heat treatment process. Results will be presented on the first phase of this research, in which the binder, injection molding, de-binding and sintering schedule were developed. Additional data on the mechanical and physical properties of the material produced will be discussed.

Products, Services, Suppliers Showcase

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

Wednesday AM Room: 2000
February 16, 2005 Location: Moscone West Convention Center

Session Chair: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

8:30 AM
Production of High Performance Aluminum Alloy Billet: *Ravi Tilak*[1]; [1]Almex USA, Inc., 1 World Trade Ctr., 8th Fl., Long Beach, CA 90831 USA

Use of Almex LARS TM molten aluminum purification technology along with Almex's Castright II TM casting equipment technology enables cast houses to produce quality products to meet today's increasingly demanding market place for high quality value added products. These innovative technologies address metallurgical issues facing cast houses daily enabling them to produce high quality product on a consistent cost effective basis.

8:50 AM
EcoReg® Continuous Rotating Regenerator - Central Heat Recovery on Large Capacity Aluminum Processing Furnaces: *Michael Shay*[1]; [1]Hauck Manufacturing Company, PO Box 90, Lebanon, PA 17042 USA

The EcoReg® Continuous Rotary Regenerator represents the latest technology in regenerative combustion systems offering higher heat recovery than conventional regenerative systems. Designed to be used for central heat recovery on large capacity furnaces, the EcoReg® replaces the classic recuperator and pulsating dual burner regenerator sets. In the EcoReg®, both principals of a classical regenerator are physically combined in one rotating generator with stationary upper and lower sections. Each section is equipped with two chambers - one for waste gas and one for combustion air. Offering excellent efficiency and productivity increases with low emissions, the regenerator handles 100% of the flue gas, eliminating the need for ancillary flues. As an added benefit, the EcoReg® design does not require burner pairs, allowing for optimum burner number and placement within the furnace design. This paper will present additional information on the EcoReg® technology and a discussion of its application to large capacity aluminum processing furnaces.

9:10 AM
Metaullics Systems Introduces a New High Capacity Pump for Today's Large Reverberatory Furnaces: *Paul Campbell*[1]; [1]Metaullics Systems Co. LP

Forced circulation in a reverberatory furnace by a molten metal pump has long been known to provide benefits in melting rates, reduced energy consumption, higher metal recovery rates, and longer refractory life. The historical recommendation of circulating the volume of the furnace four times per hour is being reconsidered as higher circulation rates are being found to be beneficial. To achieve these higher circulation rates in today's large furnaces new, higher capacity pumps are being developed. Metaullics has recently introduced a pump that has the capacity to circulate a 150 ton furnace ten times per hour.

9:30 AM
Combined Metal Skimming and Melt Treatment System for Metal Transport: *Jan Magnusson*[1]; [1]Altech SMV ehf., Hlidasmara 17, Kopavogur, Reykjavik Area IS 201 Iceland

Abstract not available.

9:50 AM
Brochot Casting System for Aluminum Alloys: *Jean-Jacque Grunspan*[1]; [1]Brochot SA, Tremblay en France 93297 France

The paper defines aluminium alloy families and their characteristics and outlines advantages of Brochot casting system for aluminium alloys. Emphasis is on turbulence-free, non-drossing casting wheel giving controlled and uniforrn ingot filling, constant metal level in

moulds and ingot size. In addition, Brochot's plant-tested programme, which adjusts cooling rate to specific alloys being cast, ensures fine grain, low porosity and minimal shrinkage. Achievenient based on a real industrial scale.

10:10 AM
Main Advantages of the Riedhammer Advanced Baking Furnace System for Production of High Quality Anodes for the Aluminium Industry: *Carmen Porzelt*[1]; [1]Riedhammer GmbH, Industrial Kiln Plants, 90332 Nürnberg Germany

In the last 80 years Riedhammer has been designing and developing Closed Ring Pit Furnaces for t h e baking of high quality anodes for the aluminium industry and more than 105 furnaces have been built or revamped on the basis of this concept in 25 different countries. The most relevant advantages which will be presented are directly related to the achievement of a significant reduction of the capital investment aiming at an increased competitiveness. The introduction of "state of the art" anode baking technology ensures first quality baked anodes in view of the physical and chemical properties directly connected, among others, with the baking process, i.e. heating-up gradients, final baking temperature, temperature homogeneity. Another important improvement is the increase of baking and thermal efficiency positively influencing the fuel consumption as well as the cycle time. A computerised auto firing system permits very high equipment and process reliability together with the optimisation of baking parameters, cycle time ("fast baking") and overall energy balance. Thereby a further strong reduction of the emission levels could be realised emphasising our concern to maintain and improve the environmental friendly characteristics of the equipment.

10:30 AM
Anode Slot Cutting Machine: *Jean-Jacque Grunspan*[1]; [1]Brochot SA, Tremblay en France 93297 France

The paper outlines BROCHOT and ALCO-TEK full pot-rooms installafion project at Alouette sept-Iles in Quebec Canada. It will present four main tasks accurately under progress: Installation and welding of BUS BARS; Installation and alignment of POT Shells; Full lining of the pots; and Superstructure Installation. Moreover, describes BROCHOT/ALCO-TEK contract system for carrying out the plan work schedule along with the specific equipment used for the purpose as well as their relevant appropriate maintenance.

10:50 AM
Latest Developments with TAC: *Martin Taylor*[1]; [1]STAS, Chicoutimi, QC G7K 1H1 Canada

Stas has recently supplied TAC (Treatment of Aluminium in a Crucible) to large aluminium primary smelters, for both <Brownfield> and existing plants. These TAC units are used to remove alkalines, especially sodium, from molten metal using aluminium fluoride. There have also been developments to use a similar technology to remove alkalines .from molten metal in Cast Houses where only recycled metal is processed. Stas will report on the latest developments in this field.

11:10 AM
Bath Crucible Handler, Vehicle Designed to Manipulate Bath Crucibles in the Potroom: *Eloise Harvey*[1]; [1]Mecfor Inc., Chicoutimi, QC G7K 1H1 Canada

Maintaining an adequate level of bath in pots is crucial when a primary smelter wants to achieve high purity aluminium. Indeed, the bath level must be kept just below the cast iron or steel rod in order to minimise its corrosion thus, minimising the deposit of Fe in the aluminium. Mecfor's bath crucible handler allows for precise siphoning and pouring operations to balance bath between pots in primary smelters potrooms. To perform the bath balancing operation, an articulated or rigid bath crucible handling vehicle specially designed for the harsh working environment of aluminium smelter is used. The vehicle is fully autonomous and can pick up a crucible, even from the floor, rotate it and tilt it both ways towards the left or the right of the vehicle to pour bath. The operator can complete his work without ever having to leave his vehicle since the vehicle is equipped with an on-board compressor. Its highly efficient handling system requires only a few seconds to load a crucible. The grip is secured by a manual lock operated from each side of the handler. The rotation allows for a 135° movement, both ways, executed with a hydraulic motor and a slewing bearing. The tilting position is performed by two hydraulic cylinders up to 96°. The bath crucible handler also possesses a high-tech weight scale that indicates the deflection on the horizontal arm of the handler thus providing extremely precise measurements. The system indicates the weight of each load during the siphoning and even during the pouring operation (+/-2Okg or 45 Ibs.). The weight precision is crucial to obtain an adequate mix. The system is composed of strain gauges installed on the horizontal arms and of two weight displays: one on the mast and another one inside the cabin. Another advantage of the bath crucible handler is the bi-directional steering that allows the operator to drive in the forward direction at all time. It provides unobstructed view during the transportation with the bath crucible.

11:30 AM
Cast Aluminium Developments in Automotive Applications: *Nnamdi Anyadike*[1]; [1]APT Aluminum Process and Product Technology, London TW10 6QX UK

Cast aluminium is increasingly gaining in importance, particularly with the growth of highly precise and complex cast products. Each year in Europe alone, more than two million tonnes of aluminium are processed. The shift from grey cast-iron to aluminium casting began in the 1970s and since then weight reductions in the engine block have been in the order of 40-55% compared with the mid-1970s. Further substitutions have been made in areas such as the axle, leading to considerable performance enhancements. Meanwhile, casting methods have been developed in the bid to keep pace with demands from the automotive industry for a lightweight yet robust alternative to iron and steel castings. For aluminium engine blocks the low-pressure casting process has proved to be a high quality technique that satisfies the automotive industry's requiurements. In the case of the 'all aluminium car body' a whole new space frame technology needed to be developed with vacuum pressure die casting at its heart. Automotive aluminium castings are continuing to improve as the technology leaps forward. This paper will look at some of these developments and provide some indication of where the 'next generation' castings are heading.

Recycling - General Sessions: Aluminum and Consumer Goods Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, LMD/EPD-Recycling Committee
Program Organizer: Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA

Wednesday AM Room: 2011
February 16, 2005 Location: Moscone West Convention Center

Session Chair: TBA

8:30 AM
Consolidation of Fine Aluminum Scrap Via the Extrusion Process: *William H. Van Geertruyden*[1]; Wojciech Z. Misiolek[2]; Clifford A. Prescott[2]; [1]EMV Technologies, LLC, 115 Research Dr., Bethlehem, PA 18015 USA; [2]Lehigh University, Inst. for Metal Forming, 5 E. Packer Ave., Bethlehem, PA 18015 USA

In the current aluminum processing industry, there does not exist a cost effective method for remelting fine aluminum scrap. Fine aluminum scrap may include machine chips and swarfs, fine gauge wire, or other particles. Additionally, remelting this type of material is many times not feasible because of considerable oxidation due to the high surface to volume ratio. A new method for consolidation fine aluminum scrap by cold extrusion is proposed in this paper. Cold extrusion experiments have been performed using fine aluminum powder and chopped aluminum wire to understand the effect of particle size and shape on the pressure necessary to produce consolidated aluminum material. These extrusion experiments were performed using a front pad to introduce different levels of counter pressure to control the degree of densification.

9:00 AM
New Experimental Approach in the Search of Intermetallic Compounds for Fe, Mn and Si Removal in Aluminum Recycling: *Alexander Pisch*[1]; Christoph Kraeutlein[2]; Pierre Le Brun[3]; Georg Rombach[4]; Paul de Vries[5]; Marc Ryckeboer[6]; Christian J. Simensen[7]; [1]INPG, LTPCM, Domaine Universitaire, BP 75, St. Martin d'Heres Cedex 38000 France; [2]RWTH Aachen, IME, Intzestrasse 3, Aachen 52056 Germany; [3]Alcan, Voreppe Rsch. Ctr., BP 27, 725 rue Aristide Bergès, Voreppe 38341 France; [4]Hydro Aluminium Deutschland GmbH, Georg-von-Boeselager-Str. 21, Bonn 53117 Germany; [5]Corus Technology B.V., PO Box 10000, CA Ijmuiden 1970 The Netherlands; [6]Remi Claeys Aluminium NV, Kortemarkstraat 52, Lichtervelde 8810 Belgium; [7]SINTEF, Matls. Tech., PO Box 124 Blindern, Oslo 0324 Norway

A new experimental approach has been developed to identify new, unknown higher order intermetallic compounds which can precipitate

and segregate Fe, Mn and Si from a liquid aluminum melt during recycling. For this basic investigation, a complete experimental procedure has been defined and tested in terms of addition elements, melt processing, cooling conditions and crucible material. Large scale analysis has been performed by XRF and ICP by comparing the Fe, Mn and Si content on the top and the bottom of the samples. Several specimens have been analyzed by SEM to investigate the shape and to identify the composition of the observed intermetallics. A list of additional elements have been defined and a maximum amount of nine elements were added at a time. This work was founded by the EEC in the Growth programme (G1RD-CT-2002-00728).

9:30 AM Break

10:00 AM
About the Coalescence Mechanism of Aluminum, the Analysis of the Recent Conceptions: *Anatoly Georgievich Zholnin*[1]; Sergey Borisovich Novichkov[2]; [1]Mosoblprommontazh, Voskresensky region der.Ratmirovo, Ul. Naberezhnaya, 4, Moskovskaya oblast 140207 Russia; [2]Russkiy Aluminiy, Prokatniy Div., Verhniy Taganskiy Typic, 4, Moscow Russia

The overview of articles about confluence of molten aluminum droplets in liquid flux was made. We have reviewed previously published experimental data. The contradiction between conceptions about coalescence mechanism and experimental data was revealed. Mechanisms are proposed which improve conceptions about coalescence. We have shown experimentally that the changes of fusion conditions may principal modify our conceptions about the role of fluoride and chloride additions to the flux. These were revealed to be the determining factors at melting of low-yield aluminum raw material. The basic source of metal loss in reverberatory and rotary furnaces is the mixing of the metal in flux and oxides. Therefore, the determining factors in real fusions are not the flux composition and its behavior, but is instead the viscosity and the wetting ability of flux-oxides substance. On the aluminum drops and flux-oxides substance interaction will be imposed upon mechanical effect, which presents on any fusions.

10:30 AM
The Recovery of Aluminium Metal from Primary and Secondary Drosses by Grinding and Screening: *Warren John Bruckard*[1]; Patrick Walta[1]; James Thorpe Woodcock[1]; [1]CSIRO Minerals, Box 312, Clayton S., Melbourne, Victoria 3169 Australia

Dross is formed during the smelting and remelting of aluminium. It is toxic, and expensive to dump, but contains valuable metallic aluminium. Tests on Australian drosses obtained high recoveries of metallic aluminium in high-grade concentrates by wet milling and screening. Characterisation of the drosses showed mixtures of metallics, oxides, nitrides, carbides, and soluble salts. P80 ranged from 290 µm to 750 µm. After wet grinding in a ball mill the P80 was reduced to 38-74 µm. Assay of the size fractions showed that the malleable aluminium particles had been flattened and reported in the coarser fractions, and that the softer or more brittle non-metallics reported to the fine fractions. For primary dross, screening at 150 µm recovered 95% of the metallic aluminium in a product assaying 90% Al0. Secondary drosses gave about 54% recovery at 70% Al0. It is believed that these products can be directly remelted, thus providing an economic benefit.

11:00 AM
Extraction of Value Added Products from Aluminium Dross Material to Achieve Zero Waste: *Jyoti Mukhopadhyay*[1]; Y. V. Ramana[2]; Upendra Singh[2]; [1]Jawaharlal Nehru Aluminium Research Development and Design Centre, Techl., Amravati Rd., Wadi, Nagpur, Maharashtra 440 023 India; [2]HINDALCO Industries Limited, R&D, PO Renukoot, Renukoot, Uttar Pradesh 231 217 India

Hindalco generates 4,000 tons of dross annually while producing 340,000 tons of aluminium. The dross is disposed as process reject for further processing to downstream industries. The lowest grade of dross normally contains less than 10% metallic aluminium, which is further processed to generate alum as a by-product. Since Hindalco consumed substantial amount of alum for water purification, it was decided to study the scope for internal generation of alum. Accordingly, alum was produced from dross and compared with the alum obtained from the market. It was observed that the cost of indigenous alum is less than half of alum procured from the market. Furthermore, the properties of indigenously produced alum compared very well with commercial alum. The remaining process reject after alum production will be used for making high quality refractory. As a result, a zero waste concept could be achieved, while using waste dross material.

11:30 AM
Characterization of the Hazardous Components in End-of-Life Notebook Displays: A. Mester[1]; N. Fraunholcz[2]; A. van Schaik[1]; *M. A. Reuter*[1]; [1]Delft University of Technology, Mijnbouwstraat 120, Delft 2628 RX Netherlands; [2]Recycling Avenue, Rotterdamseweg 145, Delft 2628 AL Netherlands

The use of notebooks has been strongly and steadily increasing for years in Europe. A corresponding increase in the amount of end-of-life notebooks available for recycling is expected in the coming years. Notebooks are equipped with liquid crystal displays mainly using mercury-containing backlights. The European Directive on waste electrical and electronic equipment (WEEE) requires the save removal of both mercury and liquid crystals. Therefore, the treatment of end-of-life notebooks requires special treatment for which the existing infrastructure for the recycling of traditional desktop computers based on cathode ray tube monitors is not suited. We studied the composition of end-of-life notebook displays in terms of number and mercury content of backlights and main parts such as printed circuit boards. For this, an actual sample of 150 end-of-life notebooks was dismantled manually. Our results indicate that the best option to safely remove mercury is shredding with controlled vapor extraction. Moreover, literature data show that the toxicity of liquid crystals is very low. Therefore, the legislative requirement of their controlled removal appears not justified. The data were also used to predict the material composition of notebook displays for a few years ahead.

Refractory Metals in Electronic Applications: Applications

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee, EMPMD-Thin Films & Interfaces Committee, SMD-Refractory Metals Committee, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Gary A. Rozak, Fabricated Products, Cleveland, OH 44117 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA

Wednesday AM Room: 3010
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Gary A. Rozak, H.C. Starck, Cleveland, OH 44117-1117 USA; Don Mitchell, Rhenium Alloys Inc., Elyria, OH 44036-0245 USA

8:30 AM
Rhenium, Tungsten and Molybdenum in Electronics: *Don Mitchell*[1]; Todd Leonhardt[1]; James Downs[1]; [1]Rhenium Alloys Inc., 1329 Taylor St., Elyria, OH 44036 USA

Rhenium, tungsten, and molybdenum are used in a wide range of applications for the electronics industry because of the inherent properties of these pure metals and their alloys. These refractory metals have unique properties of high melting points, high temperature strength, high modulus of elasticity, good ductility, and high recrystallization temperatures. A discussion of some of the applications and properties will be presented.

9:00 AM
Applications of Molybdenum in Electronic Materials: *John A. Shields*[1]; Gary A. Rozak[1]; [1]H.C. Starck, Mo/W Ops., Fabricated Products Business Grp., 21801 Tungsten Rd., Cleveland, OH 44117 USA

Molybdenum and molybdenum alloys have thermal, mechanical, electrical and chemical properties that are uniquely applied as electronic materials and in the production of electronic materials. Componant applications include thermal management in electronic packaging, thick film multi-layer ceramic packaging, and thin film metallization and in flat panel displays. Mo and Mo alloys are also employed in manufacturing equipment and tooling for the production of electronic materials. For example, molybdenum is used in masks, ion-implantation equipment and crystal growth furnaces. This presentation will summarize the properties of molybdenum for particular electronic material applications.

9:30 AM
Iridium/Iridium Silicide as an Oxidation Resistant Capping Layer for Soft X-Ray Mirrors: *Shon T. Prisbrey*[1]; Stephen P. Vernon[2];

[1]Lawrence Livermore National Laboratory/University of California, Davis, L-174, 7000 E. Ave., Livermore, CA 94551 USA; [2]Lawrence Livermore National Laboratory, 7000 E. Ave., Livermore, CA 94551 USA

Quarter wavelength multilayer stacks for 13.4 nm light were employed to probe the oxidation resistance of an [Ir/Si] capping layer. Ir thickness values ($7.5 < d_{Ir} < 20$ Å) were investigated. Reflected standing waves were modeled and correlated to Ir thickness, sputtering auger depth profiles, and atomic force microscopy surface measurements. Peak reflectivity determined stability. $7.5 < d_{Ir} < \sim 10$ Å and $15 < d_{Ir} < 20$ Å capping layers were stable after exposure to air for the time measured in this study (~140 days) whereas $\sim 10 < d_{Ir} < 15$ Å capping layers were not. The instability coincides with increased surface roughness (2 -> 5 Å RMS) and the formation of a SiO_2 surface layer. Modeling implied: the formation of iridium silicide, formation of a SiO_2 surface layer for $\sim 10 < d_{Ir} < 15$ Å, and that for $d_{Ir} > 16$ Å, $IrSi_y$ ($y<<1$) is present on the surface.

10:00 AM Break

10:15 AM
Tantalum and Niobium Powder for Electronics Applications: *Leah F. Simkins*[1]; Michael J. Albarelli[1]; [1]H. C. Starck, Inc., 45 Industl. Place, Newton, MA 02161 USA

The tantalum pentoxide as a dielectric film has been the subject of intensive investigation for the last 50 years because of the importance of these insulators in capacitors. Over the past 10 years, capacitors have gotten smaller as technology has developed to make higher surface area powders. In 2002, due to a technology developed at H.C. Starck niobium finally entered the capacitor industry as a viable capacitor substrate. This paper will explore the properties, characteristics, and applications of tantalum and niobium in the electronics industry.

10:45 AM
Tantalum "Spine Anodes" for High Power Electrolytic Capacitors: *Gerhard E. Welsch*[1]; Donald McGervey[1]; [1]CWRU, Matls. Sci. & Engrg., 10900 Euclid Ave., Cleveland, OH 44106 USA

The paper describes how performance criteria of electrolytic capacitors depend on material properties and on capacitor anode design. Various designs will be discussed and a new method for the fabrication of spine-anodes(TM) will be presented using solid tantalum substrates on which layers of high-surface-area tantalum sponge are grown. We will show that capacitors made from thin spine-anodes have higher energy-density and higher power than capacitors made from pressed and sintered powder.

Shape Casting — The John Campbell Symposium: Modeling

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM Room: 2008
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Mark R. Jolly, University of Birmingham, IRC in Matls., Edgbaston, Birmingham B15 2TT UK

8:30 AM
Computer Simulation of Shrinkage-Related Defects in Castings - A Review: *Doru Michael Stefanescu*[1]; [1]University of Alabama, PO Box 870202, Tuscaloosa, AL 35487 USA

Simulation of shrinkage-related defects in shape castings has been extensively studied because of its potential contribution to quality improvement of cast products. The location of macro-shrinkage can be estimated rather easily by present solidification models in most casting alloys. The exception is ductile iron, where graphite expansion complicates the physics. For all alloys it is still difficult to quantify macroshrinkage size. In spite of concentrated efforts and numerous claims of success, microporosity prediction is still a problem looking for a solution. After a succinct analysis of the physics of the problem, the paper reviews the various approaches to modeling macro- and micro-porosity evolution, from simple thermal models and criteria functions, to channel and porous medium models based on hydrogen diffusion, and finally to a most recent model based on oxide entrapment. Recent experimental data on microporosity formation in thin-wall ductile iron is also included in the analysis.

9:00 AM
Computational Modelling of Mould Filling and Related Free Surface Flows in Shape Casting: *Mark Cross*[1]; Koulis A. Pericleous[1]; T. Nick Croft[1]; Diane McBride[1]; James Lawrence[1]; Alison J. Williams[1]; [1]University of Greenwich, Ctr. for Numerical Modlg. & Process Analysis, Old Royal Naval Coll., Park Row, London SE10 9LS UK

Accurate representation of the coupled effects between turbulent fluid flow with a free surface, heat transfer, solidification and mould deformation has been shown to be necessary for the realistic prediction of several defects in castings and also for determining the final crystalline structure. A core component of the computational modelling of casting processes involves mould filling which is the most compute intensive aspect of casting simulation at the continuum level. Considering the complex geometries involved in shape casting, the evolution of the free surface, gas entrapment and the entrainment of oxide layers into the casting make this a very challenging task in every respect. Despite well over 30 years of effort in developing algorithms this is by no means a closed subject. In this paper we will review the full range of computational methods used from unstructured finite element and finite volume methods through fully structured and block structured approaches utilising the cut cell family of techniques to capture the geometric complexity inherent in shape casting. This discussion will include the challenges of generating rapid solutions on high performance parallel cluster technology, and how mould filling links in with the full spectrum of physics involved shape casting. Finally, some indications as to novel techniques emerging now that can address genuinely arbitrarily complex geometries are briefly outlined and their advantages and disadvantages discussed.

9:30 AM
Simulation of Casting Complex Shaped Objects Using SPH: *Paul Cleary*[1]; Joseph Ha[1]; Mahesh Prakash[1]; Thang Nguyen[2]; [1]CSIRO, Math. & Info. Scis., PB 10, Clayton S., Victoria Vic 3169 Australia; [2]CSIRO, Mfg. & Infrastruct. Tech., Preston, Victoria Vic 3072 Australia

The geometric complexity and high fluid speeds involved in High Pressure Die Casting (HPDC) combine to give strongly three dimensional fluid flow, with significant free surface fragmentation and splashing. A Lagrangian simulation technique that is particularly well suited to modelling HPDC is Smoothed Particle Hydrodynamics (SPH). Materials are approximated by particles that are free to move around rather than by fixed grids, enabling the accurate prediction of fluid flows involving complex free surface motion. The SPH method will be summarized and its relative strengths and weaknesses for die will be discussed. SPH has other attractive features for the prediction of casting, including ready prediction of shrinkage, feeding, some forms of porosity generation and surface oxide formation. These will be demonstrated using a simple two dimensional Low Pressure Die Casting (LPDC) example. Several examples of SPH simulated HPDC flows for highly complex real three dimensional industrial components/products will be presented. Automotive parts (usually cast with aluminium) range from simple cases such as a servo piston to structural cross members and a full engine rocker cover. Simulation of casting of many household products such as door handles remain large calculations because of the very small gates employed with zinc. A example of zinc die casting will be presented. Together these show unprecedented detail in the fluid free surfaces, particularly in the extent of fragmentation and void formation for the casting of complex shapes. Validation of isothermal SPH flow predictions by comparison with water analogue experiment and MAGMAsoft predictions are summarized for the flow in a single servo piston head. The SPH simulations were better able to capture the key details of the fluid motion and splashing, particularly the relative rates of flow around sharp bends and through thin sections. Some initial validation of flow predictions coupled to temperature and solidification using short shots are also presented. The bulk features of the final solid castings are in good agreement with the predictions. These results together combine to demonstrate that SPH modelling of die casting has now reached a level where both isothermal and thermal simulations can be performed in reasonable computation times for large scale automotive castings and provide a good degree of predictive accuracy.

10:00 AM
Review of Optimisation Methods for Casting Simulation: *Rajesh S. Ransing*[1]; [1]University of Wales Swansea, Civil & Computational Engrg., Sch. of Engrg., Singleton Park, Swansea, W. Glamorgan SA2 8PP UK

The manufacture of defect free components at low cost and high productivity is as important to the casting industry today, as it was thirty years ago. In old days, experience was gained either by using a "trial and error" method or by undertaking expensive experiments. Many "do's" and "don'ts" in the casting process have been evolved over a period of time. However, the important ones that come to mind are so fundamental that they challenge the "academic mind" to think all over again. The rules proposed by Professor John Campbell are classic examples. The message is simple: mathematical complexity in computer models needs to go hand in hand with the rules derived from "first principles." In the optimisation domain, a range of methods have been evolved over a period of years. At the start of the optimisation study, a foundryman has the first option of using simple and well established methods such as the use of orthogonal arrays for optimal design of process conditions or the famous Inscribed or Heuvers circle method for optimal feeding design. With the maturity of computer simulation software, the inverse analysis has also become a popular tool to determine optimal specification of some of the process parameters. The computer simulation software can be based on a variety of computational methods ranging from geometric reasoning techniques (the famous Chvorinov rule and its variants) to solving complex partial differential equations using one of the numerical methods. Optimisation methods based on solving partial differential methods was an active area of research in mid nineties with pioneering work originating from Professor Dantzig's group. This paper will review a variety of optimization methods highlighting their advantages and disadvantages. The paper will raise some of the challenging issues that the optimization community is facing today and also report on our recent work on linking geometric reasoning techniques with the finite element method to achieve optimal and computationally efficient feeding design of castings.

10:30 AM Break

10:40 AM

State of the Art Review of Use of Modelling Software for Shape Casting: *Mark R. Jolly*[1]; [1]University of Birmingham, IRC in Matls. Procg., Edgbaston, Birmingham B15 2TT UK

During the time that John Campbell was developing the Cosworth process for giving quiescent filling in aluminium castings other workers were using the newly developed PCs to help them in the foundry. In the mid-1980s the first 3D PC based casting modelling software, SOLSTAR, was launched into the market. It was developed by a foundryman and used a series of rules to model casting solidification on a cellular automaton based approach. A finite difference type mesh described the geometry and by the early 1990s was using up to 4 million cells. It was solely applicable to prediction primary shrinkage cavities mainly in the ferrous castings. Initially it was sold only to steel foundries, then cast iron and copper and eventually in the early 1990's an aluminium version was developed. However for John Campbell modelling solidification shrinkage was a secondary consideration and it wasn't until software codes were developed that could model flow that he started to take modelling seriously. This was demonstrated in the 1995 conference Modelling of Casting, Welding and Advanced Solidification Processes where, as co-chair, he set up a benchmarking exercise to test the filling algorithms of the software developers. This was made possible by the real-time x-ray equipment that has proved to be invaluable in his recent research work in helping validate and develop his own theories for oxide generation aswell as validate, or not, casting filling simulation software codes. During the following fifteen or so years, many man-hours have been dedicated to developing methods for simulating the whole casting process and to other supplementary processes that exist within the foundry. This review attempts to give the reader an understanding of the special problems that exist within the foundry process and how simulation software has been applied to help give solutions to manufacturing problems.

11:00 AM

Analysis and Optimization of the Transient Stage of Stopper-Rod Pour: *David Goettsch*[1]; Michael Barkhudarov[2]; [1]General Motors, Powertrain, 895 Joslyn Ave., Pontiac, MI 48340-2925 USA; [2]Flow Science, Inc., 683 Harkle Rd., Ste. A, Santa Fe, NM 87505 USA

Stopper-rod controlled pouring system is a simple, efficient method for metal delivery in high production cast lines. However, the raising of the rod from the nozzle creates a complex flow front that results in air entrainment and oxide film creation that are detrimental to casting quality. Three primary factors resulting in defect creation which compromise casting quality are: 1) metallostatic head pressure, 2) shape of the rod, and 3) the speed of its removal from the nozzle. Our present work aims to employ numerical modeling to enhance the understanding of this essentially transient flow. The model will then be applied to optimize the process to minimize the potential for such defects. Real time X-ray visualization will be used to validate the results.

11:20 AM

"Designing-In" Controlled Filling Using Numerical Simulation for Gravity Sand Casting of Aluminium Alloys: *Jean-Christophe Gebelin*[1]; Fu-Yuan Hsu[2]; Mark R. Jolly[1]; John Campbell[1]; [1]University of Birmingham, IRC in Matls. Procg., Edgbaston, Birmingham, W. Midlands B15 2TT UK; [2]Auspicium Co. Ltd., 2F-3, No.4, Sec. 1, Jen-Ai Rd., Taipei Taiwan

In order to achieve good reliable aluminium alloy casting components using gravity sand casting process it is essential to have a good part design, good quality ingots, a good melting process, a good metal transfer system from the furnace to the mould, and use a good running system design. In developing the running system design, and especially in the case of gravity sand casting of aluminium alloys, the first aim of the designer should be the control of the metal stream during the filling. From this statement, it would appear that the only reasonable way forward is to use bottom-gating system, where the liquid metal travels in opposition to the direction of the gravitational force. Only then can the metal stream truly be controlled. This immediately creates a problem in that the metal is going upward in the casting, but in the running system it has first to go downward, in the most controlled possible way. In order to achieve that a number of studies of different parts of the running system has been carried out, such as investigating the pouring basin,[1] down-sprue,[2-3] runner[4] and gating system,[5-6] but putting everything together and looking at the 'junctions' is still a challenging task. In this paper we will present the main results on the 'right' design for the different parts of the running system and some recent work on the "junction" where process modelling and experimental validation have been widely used. [1]Yang X. and Campbell J., Liquid metal flow in a pouring basin, Int. J. Cast Metals Res., 10, pp-239-253, 1998. [2]Campbell J., Castings, Butterworth-Heinemann, 1991. [3]Sirrell B.R., The optimisation of the running and gating of sand casting, M. Ph. Thesis, The University of Birmingham, UK, 1995. [4]Sirrell B. and Campbell J., Mechanism of filtration in reduction of Casting defects due to surface turbulence during mould filling, Trans. AFS, pp 645-654, 1997. [5]Jeancolas M., Cohen de Lara G., Hanf H., Hydrodynamics study of horizontal gating systems, Trans. AFS, v70. 1962. [6]Srinivasan M. N., Applied hydraulics to gating systems, PhD thesis, The University of Birmingham, 1962.

11:40 AM

A Front-Tracking Method of Predicting the Solidification Microstructure in Shape Castings: *Shaun McFadden*[1]; David J. Browne[1]; [1]University College Dublin, Mechl. Engrg., Engrg. Bldg., Belfield, Dublin 4, Co Dublin Ireland

A novel numerical model has been developed that simulates the nucleation and growth of columnar and equiaxed grains from the liquid phase in a casting. This model uses a control volume finite difference method to solve conduction heat transfer over a two-dimensional domain. The evolving columnar front and equiaxed grain boundaries are represented by sharp fronts across the mesh. A front-tracking algorithm is used to predict the evolution of the grains across the grid according to dendrite kinetics. The heat equation is coupled to the front-tracking algorithm by a source term that represents the latent heat that evolves as the dendrite tips advance and the mushy zone solidifies. This model predicts the extent of liquid undercooling ahead of the growing columnar front and this computation is used to determine the likelihood of nucleation and growth of equiaxed grains in the liquid; in particular the columnar-to-equiaxed transition is predicted.

Superalloys and Coatings for High Temperature Applications: Superalloys - I

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Wednesday AM Room: Nob Hill A/B
February 16, 2005 Location: San Francisco Marriott

Session Chairs: Malcolm McLean, Imperial College London, London SW72AZ UK; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA

8:30 AM Invited
High Temperature Protective Coating Systems, The First 60 Years: *Donald H. Boone*[1]; [1]BWD Turbines, Ltd., 2412 Cascade Dr., Walnut Creek, CA 94598-4300 USA

Protective coating for superalloy components operating in gas turbine engines have been in successful use for over 60 years. Their need and use continues as greater dependencies are placed on coatings for present and advanced designs. A review of this history and the significant understanding attained will be presented as a background to thoughts on present activities and future goals. Some critical achievements, recurring myths, and stumbling blocks will be suggested.

9:00 AM Invited
Improved Single Crystal Superalloy, CMSX-4® (SLS)[La+Y]: *Jacqueline B. Wahl*[1]; Ken Harris[1]; [1]Cannon-Muskegon Corporation, Box 506, Muskegon, MI 49443-0506 USA

Modern turbine engine designs place demanding requirements on single crystal superalloy turbine components. The most stringent applications, such as first stage turbine blades and vanes, require a fully solutioned microstructure to achieve superior high temperature creep and fatigue strength, with excellent oxidation and coating performance, including the use of prime reliant thermal barrier coatings. CMSX-4® [SLS][La+Y] is an improved version of CMSX-4® alloy, with typical sulfur content of 1 ppm and pre-alloyed with lanthanum and yttrium to address these requirements. Significant experience through the manufacture of fifteen heats of alloy and numerous component casting trials will be discussed along with the benefits of lower reactive element additions, pre-alloyed ingot and composite remelt charge blending. This technology has been scaled to 4000 lb. heats to meet initial development and production applications. Current status, including engine service results, will be discussed.

9:30 AM
Surface Modification of Nickel Based Alloys for Improved Oxidation Resistance: *Paul D. Jablonski*[1]; David E. Alman[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321 USA

The present research is aimed at the evaluation of a surface modification treatment to enhance the high temperature stability of nickel-base superalloys. A low Coefficient Thermal Expansion (CTE ~ 12.5x10-6/°C) alloy based on the composition (in weight %) of Ni-22Mo-12.5Cr was produced by Vacuum Induction Melting and Vacuum Arc Melting and reduced to sheet by conventional thermal-mechanical processing. A surface treatment was devised to enhance the oxidation resistance of the alloys at high temperature. Oxidation tests (in dry and wet air; treated and untreated) were conducted 800°C to evaluate the oxidation resistance of the alloys. The results were compared to the behavior of Haynes 230 (Ni-22Cr) in the treated and untreated conditions. The treatment was not very effective for Haynes 230, as this alloy had similar oxidation behavior in both the treated and untreated conditions. However, the treatment had a significant effect on the behavior of the low CTE alloy. At 800°C, the untreated Ni-12.5Cr alloy was 5 times less oxidation resistant than Haynes 230. However, in the treated condition, the Ni-12.5Cr alloy had comparable oxidation resistance to the Haynes 230 alloy.

9:55 AM
Effect of Alloying Elements on the Oxidation Behavior of 4th Generation Ni-Base Single-Crystal Superalloys: *Kyoko Hashi Kawagishi*[1]; Atsushi Sato[2]; Toshiharu Kobayashi[1]; Hiroshi Harada[1]; [1]National Institute for Materials Science, High Temp. Matls. Grp., 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Shibaura Institute of Technology, Dept. of Matls. Sci. & Engrg., 3-9-14, Shibaura, Minato-ku, Tokyo 108-0023 Japan

The 4th generation Ni-base single crystal superalloys, which contain large amounts of refractory metals for strengthening and platinum group metals, e.g., Ru, for TCP-phase prevention, show excellent high-temperature strengths. However, these alloying elements seem to decrease high-temperature oxidation resistance, and the improvement of high-temperature oxidation resistance is one of the important issues for practical use of these superalloys. In this study, Ni-base superalloys with various amounts of Hf, Ta, Re and Ru were examined in isothermal and cyclic exposures at 1100°C to investigate the effect on the oxide growth rate and resistance to scale spallation. Structures of the oxide for the alloys were analyzed by XRD etc, and the oxidation kinetics is discussed.

10:20 AM
High Temperature Corrosion of Ni-Cr Based Superalloys in a LiCl-6% Li2O Molten Salt System: Alberto Polar[1]; Francisco Rumiche[1]; *J. Ernesto Indacochea*[1]; Christine T. Snyder[2]; Leonard Leibowitz[2]; [1]University of Illinois, Civil & Matls. Engrg., 842 W. Taylor St., Chicago, IL 60607 USA; [2]Argonne National Laboratory, Cheml. Engrg. Div., 9700 Cass Ave., Bldg. 205, Argonne, IL 60439 USA

Corrosion tests in molten LiCl-6% Li_2O were conducted on a set of Ni-Cr based superalloys at temperatures ranging from 625°C to 725°C at the Argonne National Laboratory to evaluate their capability to be used for vessels in the electrometallurgical treatment of spent oxide fuels. Ar-10% O_2 gas was bubbled inside the molten salt to simulate the effect of oxygen emission during the reduction phase of the process. Microstructural examination under optical and scanning electron microscopes was performed, and the oxide layers were analyzed using EDS and XRD techniques. Ni-Cr alloys with high Fe content, as well as those where refractory metals were present, produce a highly porous non protective layer. Chlorides formation seems to contribute to the porous character of these layers. Even though different layer morphologies were observed, Ni-Cr alloys with only other minor alloying elements, formed a dense protective layer and they showed the best performance among the analyzed samples.

10:45 AM Break

11:10 AM Invited
An Overview of Modeling Approaches for High Temperature Materials and Coating Design, Processing and Durability for Gas Turbine Applications: *Prakash C. Patnaik*[1]; [1]National Research Council Canada, Inst. for Aeros. Rsch., Bldg. M13, Montreal Rd. Campus, Ottawa, Ontario K1A 0R6 Canada

This paper will present an overview of modeling efforts in design, processing and durability of gas turbine materials and coatings as work continues to advance in this laboratory. Attempts are being made to systematically use first principle calculations of alloying strengthening effects in binary, ternary, quaternary and higher order (up to 5 alloying elements) Ni alloys representing the gamma solid solution and the alloying representing the transition or refractory metals. From superalloy design point of view, one needs to select the alloying additions wisely in order to achieve effective solid strengthening in the gamma matrix. On the processing of materials, understanding the behaviour of chemical microsegregation and the addition of Re, W and Ru on the microsegregation is of paramount importance for modeling purposes. Utilizing a solid-liquid phase equilibria model and a customized chemical thermodynamic database, chemical partitioning predictions are carried out. Future generation of advanced coatings for high temperature applications are addressed through the use of ceramic Thermal Barrier Coatings to increase engine efficiency and enhance component durability.Understanding the physics of the conductive and radiative heat transfer through modeling and the physics of failure have also provided the basis to improve the TBC properties further.

11:35 AM Invited
Effect of Environment on Notch Fatigue Initiation Resistance in CMSX4: *Philippa Ann Reed*[1]; Mark Miller[1]; Mark Joyce[1]; [1]University of Southampton, Sch. of Engrg. Scis., Highfield, Southampton, Hants SO17 1BJ UK

Fatigue initiation in the stress-concentrating fir-tree root fixing of turbine blades is of some concern, and as the turbine blades are often coated, it is assumed that the Ni-base blade will be relatively protected - however fretting may remove this protective coating. Crack initiation at high temperatures has been studied in CMSX4 in both air and vacuum environments, to elucidate the effect of oxidation on the notch fatigue initiation process. In air, crack initiation occurred at sub-surface interdendritic pores in all cases. The sub-surface crack grows initially under vacuum conditions, before breaking out to the top surface. Lifetime is then critically dependent on initiating pore size and distance from the notch root surface. In vacuum conditions, crack initiation has been observed more consistently from surface or close-to-surface pores - indicating that surface oxidation is in-filling/ "healing" surface pores or providing significant local stress transfer to shift initiation to sub-surface pores.

12:00 PM Invited
Mechanical Behaviour of Single Crystal Superalloys Under Cyclic Loading: *Pedro D. Portella*[1]; [1]Federal Institute for Materials Research and Testing, Matls. Engrg., Unter den Eichen 87, Berlin D-12205 Germany

Single crystal superalloy find a wide application as blades or vanes in landbased and airborne gas turbines, especially in the highly loaded first stages. Due to their very regular microstructure it is possible to characterize the changes due to high temperature deformation and correlate them to the mechanical response of these materials. In this work we discuss the microstructural changes in single crystal superalloys observed during cyclic loading at high temperatures. The influence of hold times on cyclic softening, asimmetry in the hysteresis loops and failure mechanisms can be derived from the coarsening in the gamma/gamma' structure. Parallel to this discussion it is possible to modify consistently the constitutive equations designed to predict the mechanical behaviour of these materials in order to simulate these phenomena. We further show the effect of crystal anisotropy and artificially introduced defects on crack initiation in uncoated specimens of single crystal superalloys. We finally propose an approach for lifetime estimation using the constitutive equations derived for these materials.

Surface Engineering in Materials Science - III: Characterization of Surfaces and Films/Coating

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Wednesday AM Room: 2022
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Sharmila Mukhopadhyay, Wright State University, Dept. Mech. & Matls. Engrg., Dayton, OH 45435 USA; Craig A. Blue, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6083 USA

8:30 AM Invited
Improving the Performance of Rolling Contact Bearings With Tribological Coatings: *Gary Doll*[1]; R. D. Evans[1]; C. R. Ribaudo[1]; [1]The Timken Company , Timken Rsch., 1835 Dueber Ave. SW, RES05, Canton, OH 44706 USA

Tribological coatings can improve the performance of rolling contact bearings especially those bearings that are poorly lubricated, subjected to debris, or experience high-frequency oscillatory motion. Whereas coatings such as Ag, MoS2, TiN, and TiC have been used on bearings in niche applications for many years, coatings comprised of amorphous hydrocarbon or diamond-like carbon reinforced with nanocrystalline metal carbides are becoming more broadly used on rolling contact bearings.

8:55 AM
Degradation Mechanisms of Die Coatings Used in Aluminum Pressure Die Casting: *John J. Moore*[1]; *J. L. Lin*[1]; S. L. Meyers[1]; O. Salas[2]; S. Carrera[1]; B. Mishra[1]; [1]Colorado School of Mines, ACSEL, 1500 Illinois St., Golden, CO 80401-1887 USA; [2]Instituto Tecnologico y de Estudios Superiores de Monterrey-CEM, Carretera a Lago de Guadalupe km 3.5, Atizapan Mexico 52926 Mexico

The degradation behavior of five optimized commercial coating systems, developed by different PVD processes, on the H13 hot working steel, has been studied. Data have been generated from both laboratory and in-plant trials with the primary objective of systematically understanding coating degradation mechanisms that occur during aluminum pressure die-casting. The coatings were characterized both before and after subjection to die casting with respect to stress, failure mechanisms and chemical interaction using optical microscopy (OM), scanning electron microscope (SEM), energy dispersive X-ray spectrometry (EDS), and glancing incident XRD. The results show that each of the commercial coatings provided improved die performance and die life compared with the ferritic nitrocarburized surface treatment only of the H13 tool steel. The mechanism of degradation, cracking and failure of uncoated and coated H13 dies during aluminum pressure die-casting is proposed. The conjoint action between chemical attack of the liquid aluminum alloy and thermal fatigue cracking in the H13 substrate played an important role in the degradation process.

9:15 AM
Advanced Technologies for Anticorrosive Protection of Canned-Food Steel Sheet: *Oleg B. Girin*[1]; Igor D. Zakharov[2]; Yevgen V. Kolesnyk[1]; Volodymyr I. Ovcharenko[1]; Yuliya O. Proshenko[2]; [1]Ukrainian State University of Chemical Engineering, Dept. of Matls. Sci., Prospekt Gagarina, 8, Dnepropetrovsk 49005 Ukraine; [2]Polimet Research and Technology Center, Lab. of Protective Coatings, Ul. Mandrykovskaya, 169, Dnepropetrovsk 49049 Ukraine

Presented are the developed technologies for anticorrosive protection of canned-food steel sheet that are intended both to upgrade its quality and to reduce the prime cost and the environmental hazards of its production. Discussed are the following technologies for producing on the canned-food steel sheet of: 1) a protective nanostructural composite super-thin chromium coat from a low concentration electrolyte based on hexavalent chromium compounds; 2) a protective amorphous composite super-thin chromium coat from a low toxicity electrolyte based on trivalent chromium compounds, and 3) a protective textural composite super-thin tin coat from a low toxicity electrolyte. In comparison to the conventional these technologies are: 1) more environmentally friendly due to the use of less toxic electrolytes, and 2) more cost and material saving due to the reduction of the coat thickness on the steel sheet. This research project is financed by Science & Technology Center in Ukraine, Project No. 2520.

9:35 AM
Ti Dental Implant Surface Modification by Micro-Arc Oxidation: *Carlos Nelson Elias*[1]; Jose Henrique Lima[2]; Fernando Costa Silva Filho[3]; Carlos Alberto Muller[4]; [1]Military Institute of Engineering, Pr Gen Tibúrcio 80, Rio de Janeiro, RJ Brazil; [2]Odontoclinica Central Exercito, Rua Moncorvo Filho, Rio de Janeiro, RJ Brazil; [3]Intituto de Biofísica, Universidade Federal do Rio de Janeiro, Rio de Janeiro Brazil; [4]Bioterio, Instituto Oswaldo Cruz, Rio de Janeiro, RJ Brazil

Commercial pure titanium (CP Ti) is used as dental implant material because of its excellent chemical stability and biocompatibility. The biocompatibility is closely related to the titanium oxide layer. To increase the biocompatibility of titanium various mechanical and chemical treatments has been proposed. One possibility is increasing the surface roughness and the titanium oxide thickness. In the present work, an electrochemical procedure for modifying the Ti surface was presented. A positive voltage to a CP Ti implant immersed in an electrolyte was applied, anodic oxidation of Ti occurred to form a oxide layer. The morphology of oxide layer was analyzed and the biological properties of the layers were evaluated by in vitro tests, in terms of cell culture. In vivo tests were also done to confirm the biological in vitro tests. The results showed that the dental implant surface modification by oxidation had a beneficial effect on the implant biocompatibility.

9:55 AM Break

10:30 AM Invited
Surface Engineering for Improved Adhesion to Biomedical Implants and Biosensors: *Wole O. Soboyejo*[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton Inst. of Sci. & Tech. of Matls., Princeton, NJ 08544 USA

This paper presents an overview of recent efforts to develop biocompatible surface coatings and textures that promote increased

adhesion to biomedical surfaces. These include surfaces that are relevant to orthopedic implants and biosensors at the micro- and nanoscale. In the case of orthopedic implants, alkane phosphonic acid/RGD tethers and laser textures are shown to promote increased adhesion to Ti-6Al-4V surfaces. The adhesion is quantified using shear assay experiments. Similarly, in the case of implantable bio-micro-electro-mechanical structures (bioMEMS), nanoscale biocompatible titanium coatings and alkane phosphonics/RGD tethers are shown to promote increased adhesion to silicon surfaces that are most commonly used in MEMS structures. Finally, the paper highlights the coating of magnetic nanoparticles for specific in-vivo attachment to cancer cells. Examples of such attachment are presented for breast/prostate cancer. The implications of the research are then discussed for the early detection of cancer.

10:55 AM
Plasma Sterilisation and Surface Modification of Thermolabile Materials: *Peter Messerer*[1]; Helmut Halfmann[1]; Marc Czichy[1]; Martin Schulze[1]; Peter Awakowicz[1]; [1]Ruhr-Universitaet Bochum, Inst. for Electl. Engrg. & Plasma Tech. (AEPT), Universitaetsstr. 150, Bochum 44780 Germany

At the institute for Electrical Engineering and Plasma Technology (AEPT) investigations on plasma sterilisation and surface modification of thermolabile materials are performed. Our first field is plasma treatment of medical implants made of titan, UHMWPE and degradable polylactide. In a total treatment time of two minutes the developed process reduces germs and pyrogens by six and more than four decades, respectively. A modified plasma discharge hardens the surface of UHMWPE. Gel content measurements indicate the improvement of abrasion resistance while the bulk material is unaffected. An increasing part of non-carbonated and non-acidic beverages have been bottled in PET. Sensitive products require aseptic filling. Plasma treatment prepares bottles without toxic residua. To improve the shelf-live of oxygen-sensitive soft drinks a diffusion barrier made of a SiOx-layer can be deposited on the inner side of a PET bottle. The combined sterilisation and coating process takes ten seconds.

11:15 AM
Corrosion Resistance Mechanism of Plasma-Based Low-Energy Nitrogen Ion Implanted Austenitic Stainless Steel: *M. K. Lei*[1]; X. M. Zhu[2]; [1]Dalian University of Technology, Surface Engrg. Lab., Dept. of Matls. Engrg., Dalian 116024 China; [2]Dalian Jiaotong University, Dept. of Matls. Sci. & Engrg., Dalian 116028 China

Plasma-based low-energy ion implantation (PBLEII), including plasma source ion nitriding/carburizing and plasma source low-energy ion enhanced deposition of thin films, has emerged as a low-temperature surface engineering technique. A high nitrogen face-centered-cubic phase (gamma-N) layer with peak nitrogen concentration up to 32 at.% was obtained on the nitrided 1Cr18Ni9Ti (18-8 type) austenitic stainless steel. No pitting corrosion for the gamma-N phase was confirmed by electrochemical polarization measurement and immersion corrosion test in a series of NaCl aqueous solutions. In order to explain the pitting corrosion resistance of the gamma-N phase, the passive film formed on the gamma-N phase in 3% NaCl solution was investigated by Auger electron spectroscopy and x-ray photoelectron spectroscopy in conjunction with ion beam sputtering. The protective passive film with a duplex character was by 2-3 times thicker than that of original stainless steel. It was essentially composed of two regions: iron hydroxide/oxides in the outer region and chromium hydroxide/oxides and iron oxides accompanying chromium and iron nitrides in the inner region. The conventional bilayer substructure, e.g. outer hydroxide layer and inner oxide layer, was also detected in the each region. During anodic polarization the chromium nitride with weak Cr-N ionic-type bonds in the gamma-N phase was prone to oxidation and release of nitrogen to form stable oxides and ammonia on the passivating surface. The thick iron hydroxide/oxides region formed on the chromium hydroxide/oxides region due to the increase of alkalinity in the solution, which led to completely barrier the penetration of localized attack of aggressive ions. The role of nitrogen in pitting corrosion resistance of austenitic stainless steel was proved.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session V

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Wednesday AM Room: 3003
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Greg Olson, Northwestern University, MSE, Evanston, IL 60015 USA; Long-Qing Chen, Pennsylvania State University, MSE, Univ. Park, PA 16802 USA

8:30 AM Opening Remarks

8:35 AM Invited
Surface-Induced Ordering and Epitaxt-Induced Ordering: The Role of Strain: *Alex Zunger*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

It was once thought that the growth method of an alloy does not affect the growth product, as long as one is close enough to equilibrium. It is now realized, however, that while melt-growth and LPE growth of semiconductor alloys produces phase-separation microstructure, vapor-phase (MOCVD, MBE) growth of III-V alloys such as GaP-InP produces ordered atomic arrangements in a spontaneous fashion. The structures obtained are short-period AnBn superlattices along <111> ("CuPt structure") or <001> ("CuAu structure"), and exist even though during growth all atoms are deposited simultaneously. First-principles theoretical calculations have shown that these structures are produced because of surface reconstruction and are, therefore, thermodynamically stable surface-phases that are frozen-in during subsequent growth. I will discuss how strain selects different types of ordering in semiconductor and metal alloys, a field to which A. Khachaturyan contributed significantly. Spontaneous ordering changes the point-group symmetry relative to the random alloy, leading to band gap changes, crystal-field splitting, novel polarizations, as well as changes in effective-masses, pressure coefficients, etc.

9:00 AM Invited
Ab Initio Contribution to Alloy Phase Transformations: Prospects and Challenges: *Patrice E.A. Turchi*[1]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci. Direct. (L-353), PO Box 808, Livermore, CA 94551 USA

On one hand, first-principles approaches are routinely applied to the study of the statics of phase transformations in alloys. These methodologies seem to impact the field despite, as should often be acknowledged, a lack of experimental validations. On the other hand, phase-field simulations (PFS) have shown in recent years great versatility in predicting various aspects of phase transformations in solids. Moreover the interface between ab initio and the phenomenological CALPHAD approach to thermodynamics and kinetics has recently shown some promise in bringing realism to PFS. Despite this progress, questions remain to be answered on lattice stability, the definition of thermodynamics functions in a broad range of fields, and on the acquisition of realistic kinetic parameters. After a brief survey of the contribution of ab initio to phase transformations in solids, future prospects and challenges are discussed. Work performed under the auspices of the U. S. Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

9:25 AM Invited
Aluminum Alloy Thermodynamics and Kinetics from First Principles: *Chris M. Wolverton*[1]; Vidvuds Ozolins[2]; [1]Ford Motor Company, Physl. & Environml. Sci., MD 3083/SRL, PO Box 2053, Dearborn, MI 48121-2053 USA; [2]University of California, Dept. of Matls. Sci., Los Angeles, CA 90095-1595 USA

We present an extensive survey of the thermodynamic and kinetic properties of binary Al alloys, as obtained from first-principles atomistic calculations. We consider a wide range of properties: 1) Energetic properties of ordered compounds, impurities, and mixing energies of solid solutions, 2) first-principles calculations of interatomic force constants, phonon spectra and vibrational entropies, 3) thermodynamic properties and solubility, and 4) kinetic quantities such as solute-vacancy binding, migration energies, and diffusion coefficients. We compare our results critically with experimental and CALPHAD databases to ascertain inaccuracies in the theoretical methods, and cases in which experimental data should be re-evaluated. In addition, the extensive nature of the database facilitates understanding the trends in energetic, thermodynamic, and kinetic properties. In addition, this large first-principles database should enable many future applications, such as improving existing CALPHAD databases, as well as providing key information to phase-field models of microstructural evolution.

9:50 AM
How Well are Higher-Order Correlations in Alloys Determined by Pair Correlations?: Gene Ice[1]; *Rosa Barabash*[1]; Don Nicholson[1]; Lee Robertson[1]; Cullie Sparks[1]; Christopher M. Wolverton[2]; [1]Oak Ridge National Laboratory, Metals & Ceram., One Bethel Valley Rd., MS6118, Oak Ridge, TN 37831-6118 USA; [2]Ford Motor Co., MD 3083/SRL, PO Box 2053, Dearborn, MI 48121-2053 USA

Diffusely scattered x-rays (neutrons) are sensitive to the correlations among atom positions. Only pair correlations can be recovered from diffuse scattering measurements as a measure of the average chemical neighborhood and average spacing between the different atom species. Though it has never been shown that these average pair correlation functions can uniquely determine the actual atomic arrangements, it has been shown that pair correlations do restrict the value of higher order correlations.[1] Here we present numerical analyses of the reconstruction of substitutional crystalline alloys from pair correlations derived from both pair and many-body interactions. With regular, forward Monte Carlo, pair and many-body interactions were used to calculate snapshot images of short-range ordered 64,000-atom periodic cells. We refer to these cells as "input". The averaged Warren-Cowley pair correlations were obtained for each "input" and these pair correlations were then used in a reverse Monte Carlo simulation to reconstruct "output" 64,000-atom configurations. We compare in detail the pair and higher-order (triplets and higher configurations containing up to six atoms) probabilities between the input and output configurations. Pair correlations are shown to strongly constrain the higher-order correlations. For "input" derived from pair-only interactions, the higher-order correlations of the "input" and "output" were in good agreement. For our model system (fcc/AB3), pair correlations are shown to define the morphology of the locally ordered or clustered domains. However, the higher-order correlations in the many body case appear to be restricted but not determined by the pair correlations. Issues of uniqueness are discussed as they relate to pair and many-body interactions. Work supported by DOE Office of Basic Energy Science under subcontract DEAC05-00OR22R725464 with UT-Battelle, LLC. [1]J. Gragg, J. Bardham & J. Cohen, in "Critical phenomena in Alloys, magnets and superconductors", R. Mills, E. Ascher and R. Jaffee, eds., McGraw-Hill NY (1977), pp. 309-337.

10:05 AM Break

10:30 AM Invited
Short-Range Order and Phase Evolution in Au-Ni Alloys: *J.-C. Zhao*[1]; [1]GE Global Research, One Rsch. Cir., K1-MB239, Niskayuna, NY 12309 USA

The Au-Ni system has one of the simplest phase diagrams: complete miscibility at high temperatures and a miscibility gap at low temperatures. Yet it presents one of the very complex systems in terms of short-range order (SRO), and remains one of the most intensively studied binary systems to date. Electron diffraction was performed on two Au-Ni alloys using transmission electron microscopy. Very complex SRO intensities were observed in the diffraction patterns. These intensities match very well with results from first-principles simulation. The good agreement between experimental and simulated patterns indicates that the first-principles calculations have made good progress in modeling the complex behavior in the Au-Ni alloys. In addition to SRO, spinodal decomposition, transient ordering, and continuous and discontinuous precipitation were observed under different annealing conditions.

10:55 AM
Experimental Vertifications of a Ginzburg-Landau Second Order Theory of Autonomous Nanostructural Clustering and Ordering: G. C. Weatherly[1]; K. Janghorbian[1]; G. Radtke[1]; G. A. Botton[1]; *J. S. Jirkaldy*[1]; [1]McMaster University, Brockhouse Inst. for Matls. Rsch., Hamilton, ON L8S-4M1 Canada

An elementary real space theory of clustering (spinodal decomposition) as an autonomous isothermal process starts with the construction of a free energy functional (an integral or lattice sum over a density) which at stationary defines a variational derivative to be entered into a 2nd order non-linear time-dependant Ginzburg-Landau (TDGL) partial differential equation (PDE). Solute conservation is in general entered via a Lagrange Multiplier. Since commonly in cubic crystals the Lagrangian density possesses an even symmetry which is reflected in solute-conserving solutions of the PDE, the additional application of a conserving flux balance represents an over determination which results in a 4th order PDE. Indeed, such a character in a ternary generalization denies a necessary simultaneous diagonalization of the coefficient matrices and so illogically prohibits the mounting of an initial value problem. Unlike the 4th order case, the 2nd order representation in Au-Ni following the Friedel and Eshelby atomistics incorporates coherency strain without a complicating depression of the effective critical point. Our binary TFDL equation(s) incorporating free energy densities associated with Hillert, Khachaturyan, Goryachev and Maugis has been subjected to experimental tests for high temperature clustering, the obligatory coupling of clustering and local long range ordering and extra-spinodal G-P zones in Au-Ni alloys as predicted by Morris and Khachaturyan.

11:10 AM
Ordering in Ternary CuNiZn Alloys: Concentration Wave Consideration and Monte Carlo Simulations: *Andrei V. Ruban*[1]; Sergei Simak[2]; [1]Royal Institute of Technology, Dept. of Matls. Sci. & Engrg, Brinellvagen 23, Stockholm 10044 Sweden; [2]Uppsala University, Condensed Matter Theory Grp., Box-530, Uppsala 75121 Sweden

Concentration wave theory is applied to the case of ternary CuNiZn alloys. Possible ordering scenario are considered. The investigation of the ordering in real system has been performed by Monte Carlo method with effective interactions obtained in the first-principles calculations. We demonstrate that there exist three ordering transitions in contrast to the generally accepted picture, which assumes the existence of only two. We demonstrate that this sequence of phases is a consequence of the symmetry of the ground state and the magnitude of the dominating pair interactions. It agrees with available experimental data.

11:25 AM
Effects of Ordering Phase Transformation on the Evolution of Microstructure During Annealing of Cold Deformed Equiatomic FePd: *Anirudha R. Deshpande*[1]; Jorg M.K. Wiezorek[1]; [1]University of Pittsburgh, Matl. Scis. & Engrg., 864, Benedum Hall, Pittsburgh, PA 15261 USA

Equiatomic L1o ordered FePd intermetallics form a model system for the investigation of the effects of microstructure on hard magnetic properties in the class of L1o ordered ferromagnetic intermetallics. In this system, a microstructural morphology consisting entirely of equiaxed, defect free, L1o ordered grains has been previously reported to exhibit enhanced magnetic properties relative to a characteristic lamellar-polytwin morphology. A combined reaction of FCC to L1o ordering concomitant with annealing of crystal defects introduced during cold working in the FCC stage has been employed to achieve an equiaxed, L1o ordered grain structure. A synergy between the ordering process and the annealing of crystal defects is expected to exist, since previously it has been reported that the preferential nucleation and growth of L1o ordered grains would occur at sites in the microstructure with an appropriate, favorable, localized stress state. Microstructural textures are appropriate signatures of the evolution of the microstructural state. In this study the synergy between the processes of ordering and annealing has been studied using relevant texture evolution. Orientation Distribution Functions coupled with pole figures obtained using X-ray diffraction and orientation imaging microscopy by SEM have been utilized to study the evolution of global and local textures. Local textures have also been probed using TEM. The texture evolution during the combined reaction has been compared with texture evolution during a microstructural change involving solely the annealing of crystal defects in the cold-deformed L1o state. This comparison has yielded new insights into the effects of the ordering phase transformation on the evolution of textures during annealing of these intermetallics after cold deformation.

11:40 AM
Low-Temperature Aging Mechanisms in an U-6Nb Alloy: *Luke L. Hsiung*[1]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci. Direct., PO Box 808, L-352, Livermore, CA 94551-9900 USA

Aging mechanisms in a water-quenched U-6wt.%Nb alloy isothermally aged at 200°C and naturally aged at an ambient temperature for 15 years have been investigated. The hardness of the isothermally aged samples initially increases from HV 190 (WQ) to HV 255 (200°C, 8h) and subsequently decreases to HV 237 (200°C, 16h). The age hardening phenomenon can be rationalized by the occurrence of spinodal decomposition of the α " matensite phase within the aged alloy. The occurrence of order-disorder transformation is found within the naturally aged alloy based upon the observations of antiphase boundaries (APBs) within the α " phase. An ordered α " phase and a transformation pathway for the aging of U-6Nb at low temperatures are accordingly proposed. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

11:55 AM
Phase Transformations and Microstructure Evolution in Mutlicomponent Nb-Ti-Si-Cr-Al-X Alloys: Raghvendra Tewari[1]; Hyo-jin Song[1]; Amit Chatterjee[2]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Cheml. & Matls. Engrg., Cincinnati, OH 45221-0012 USA; [2]Rolls Royce Corporation, 2001 S. Tibbs Ave., Indianapolis, IN 46206 USA

Recently, alloys based on the Nb-Ti-Si system have become of interest for high temperature structural applications. In the present work, the microstructure of multicomponent Nb-30Ti-8Si-10Cr-10Al-X (in at.%) alloys in the as-cast and heat treated conditions was studied utilizing x-ray diffraction, electron probe microanalysis, and scanning and transmission electron microscopy. The effect of temperature and time on phase evolution was examined in detail. The as-cast microstructure was found to be composed of three phases: the matrix b, silicides (of type (Nb,Ti)5Si3)) and a Cr-rich Laves phase. The b phase was found to display in B2-type ordering. The silicides in these alloys were generally quite stable during heat treatment, whereas the Cr-rich Laves phase was observed to dissolve on solutionization at temperatures above 1300°C. Aging of the solutionized materials between 900–1100°C led to the precipitation of fine particles of another Laves phase in b matrix. In addition, the b matrix revealed a tendency toward phase separation into Ti-rich and Nb-rich regions. The volume percentage and chemical composition of each phase has been determined as a function of time and temperature. The changes in the phase constituents have been rationalized in terms of the distribution of elements in various phases. The role of different alloying elements on the formation of these phases has also been critically examined.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Ultrafine-Grained Materials I

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Wednesday AM Room: 3024
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Alexander H. King, Purdue University, Sch. of Matls. Engrg., W. Lafayette, IN 47907-2044 USA; Yuri Estrin, Clausthal University of Technology, Matls. Sci. & Tech., Clausthal-Zellerfeld 38678 Germany; Evan Ma, Johns Hopkins University, Matls. Sci. & Engrg., Baltimore, MD 21218 USA

8:30 AM
Shear Deformation and the Evolution of Deformation Bands During ECAP of Pure Aluminum: The Formation of High-Angle Boundaries: Keiichiro Oh-ishi[1]; Alex P. Zhilyaev[2]; *Terry R. McNelley*[1]; [1]Naval Postgraduate School, Mechl. & Astronautical Engrg., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]Russian Academy of Sciences, Inst. of Mech., Ufa 450000 Russia

Orientation imaging microscopy (OIM) methods have been applied to the evolution of microstructure and microtexture during ECAP of pure aluminum. The influence of strain path has been considered by examination of pure aluminum after four pressing operations by route BC in a die having a 90° die angle, or eight pressing operations by route BC in a die have a 135° die angle. The von Mises equivalent strains were essentially the same for these two procedures. Microtexture results indicate that the distortion during ECAP corresponds to a simple shear in a direction approximately parallel to die-channel exit and on a plane perpendicular to the flow plane and containing the shear direction. Two orientation in the texture correspond to bands of common orientation in the microstructure, and the interfaces between the bands are high-angle in nature. The role of these bands in the formation of high-angle grain boundaries will be discussed.

8:45 AM
Processing Routes Leading to Superplatic Behaviour of ZK60 Magmesium Alloy: *Rimma Ye. Lapovok*[1]; Peter F. Thomson[1]; Ryan E. Cottam[1]; Yuri Estrin[2]; [1]Monash University, Sch. of Physics & Matl. Engrg., Clayton, Melbourne, Vic 3800 Australia; [2]Technical University of Clausthal, Inst. of Matls. Sci. & Tech., 6 Agricolastr., Clausthal, Clausthal-Zellerfeld 38678 Germany

There has recently been discussion on processing of magnesium alloys by ECAE to achieve a high elongation-to-failure during a superplastic tensile test. A two-step processing route was suggested to improve superplastic capabilities of such alloys. In this work superplastic behaviour samples of ZK60 processed by ECAE only and samples which were rolled prior the ECAE processing have been compared. It was shown that the benefit of preliminary deformation is significant for superplastic behaviour at temperatures close to 300°C and low strain rates, while in the area of low temperature superplasticity around 200°C and higher strain rates ECAE processing by itself leads to an elongation of 1700% at a strain rate of 3x10-3.

9:00 AM
Mechanical Properties in Aluminium Alloys Processed by SPD: Comparison of Different Alloy Systems and Possible Product Areas: *Hans J. Roven*[1]; Hakon A. Nesboe[1]; Jens C. Werenskiold[1]; Tanja Seibert[2]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway; [2]Friedrich-Alexander-Universitat Erlangen-Nurnberg, Inst. fur Werkstoffwissenschaften, Erlangen D-91058 Germany

The present work compares the strength and ductility of a wide range of alloys processed from N= 1 to N= 6 (8) by ECAP: AlMg-, AlMn-, AlMgSi-, AlMnSc-, AlMgSc- and high strength AlZnMg alloys. The plastic deformation has been conducted under controlled conditions with the same deformation tool (ECAP rectangular cross section) in order to make sound comparisons. The initial material conditions are changed, ranging from soft annealed and homogenized to solution heat treated and naturally aged. Post-ECAP heat treatments at different temperatures are also systematically studied. Generally, ECAP route A gives significantly increased strength and combined with non-conventional temperature artificial aging, excellent combinations of strength and ductilty is achieved for age hardening alloys. Generally, the effect of severe plastic deformation is mostly beneficial with high alloy contents, whereas lean alloys tend to be less reacting to SPD. Finally, some believed product areas for selected alloys will be discussed briefly.

9:15 AM
Texture and Microstructural Evolution in Pure Aluminum During High-Pressure Torsion: *Alexander P. Zhilyaev*[1]; Keiichiro Oishi[1]; Terence G. Langdon[2]; Terry R. McNelley[1]; [1]Naval Postgraduate School, Dept. Mechl. & Astronautical Engrg., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., 3650 McClintock Ave., Los Angeles, CA 90089-1453 USA

An orientation imaging microscopy (OIM) investigation was conducted to evaluate the microstructural characteristics in samples of pure aluminum processed by high-pressure torsion (HPT) under both constrained and unconstrained conditions. Electron backscattering diffraction (EBSD) techniques were employed to measure the microtextures and the distributions of the misorientation angles. A thorough analysis of the microtexture has revealed the shear plane normal and direction for aluminum specimens. This paper discusses the common features of the texture components by comparison with aluminum deformed by equal-channel angular pressing. It is shown that the magnitude of the grain refinement depends upon the location in the HPT disc and the pressing conditions.

9:30 AM

Determination of Deformation Mechanisms in Al5083 by Neutron Diffraction: *Sven C. Vogel*[1]; David J. Alexander[1]; Mark A.M. Bourke[1]; Donald W. Brown[1]; Bjorn Clausen[1]; Thomas A. Sisneros[1]; Enrique J. Lavernia[2]; David B. Witkin[3]; [1]Los Alamos National Laboratory, LANSCE, PO Box 1663, MS H805, Los Alamos, NM 87545 USA; [2]University of California, Cheml. Engrg. & Matls. Sci., Davis, CA 95616-5294 USA; [3]University of California, Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA

The activity of deformation mechanisms like slip, twinning, diffusion or grain boundary sliding depends on parameters such as grain-size and shape, deformation temperature and strain rate. These activities are typically determined indirectly by fitting parameters of functions describing the strain rate as a function of diffusion constants, stress exponents, flow stress and Young's modulus against experimental results for flow stress vs. strain rate. In the present paper, we describe an attempt to determine the active deformation mechanism directly by neutron diffraction on the SMARTS neutron diffractometer at LANSCE. Available data are peak shifts (lattice strains), peak intensity (texture) and peak width (inter- and intragranuler strains). The various deformation mechanisms have different signatures for these parameters. We will report our initial results on a series of measurements on Al5083. As-received (extruded), 8 pass ECAP and bulk nanograined specimen of <100 nm grain size were tested in tension at room temperature and 275°C. Each specimen was plastically deformed to a logarithmically increasing degree and neutron diffraction data were collected after unloading. Bulk texture was measured on the neutron diffractometer HIPPO before and after the deformation.

9:45 AM

Grain Refinement of Pure Al by ECAP and Simulation of the Process: *Hua Ding*[1]; Wenjuan Zhao[1]; Yuping Ren[1]; Shiming Hao[1]; [1]Northeastern University, Sch. of Matls. & Metall., 3-11,Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

In this paper, microstructures of pure Al after equal channel angular pressing(ECAP) were observed and the effects of purity, initial states of the material and deformation routes on the microstructures were investigated. The results showed that the purity of Al had obvious influnce on the final grain size of the material. The material with 99.999% Al had a tendency to grow after ECAP; while the one with 99% Al remained unchanged. It is also shown that the methods by which the samples were prepared before ECAP and deformation routes affected the microstructures of the material. Meanwhile, the ECAP process was simulated by DEFORM. The results of the simulation can be used to predict the process quite well.

10:00 AM

Elevated Temperature Behavior of SPD Materials: Superplasticity or Enhanced Ductility?: *Alla V. Sergueeva*[1]; Ruslan Z. Valiev[2]; Nathan A. Mara[1]; Amya K. Mukherjee[1]; [1]University of California, Cheml. Engrg. & Matl. Sci., Davis, CA 95616 USA; [2]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., Ufa 450059 Russia

The generated results on elevated temperature mechanical behavior of the materials produced by severe plastic deformation (SPD) have established a number of trends: extensive strain hardening; high flow stresses; a correlation between microstructural instability and enhanced plasticity. Moreover, the parametric dependencies such as strain rate exponent and activation energy of the deformation process can range widely. In many materials high elongation (more than 200%) was detected and the resulting structure still had equaxied grains while the values of the strain rate sensitivity and activation energy did not support an implication of superplastic deformation. In most cases, when enhanced ductility was observed, an intensive grain growth occurred in the materials. In current investigation the elevated temperature deformation behavior of metals and intermetallics subjected to SPD was analyzed in order to establish possible deformation mechanisms and microstructural effects. This investigation is supported by NSF, Division of Materials Research (grant NSF-DMR-0240144).

10:15 AM

Grain Boundary Diffusion and Plasticity/Superplasticity of Polycrystalline and Nanostructured Metals and Alloys: *Yury Romanovich Kolobov*[1]; Ilya Vasil'evich Ratochka[1]; [1]Institute of Strength Physics and Materials Science, Physl. Matl. Sci., Academichesky 2/1, Tomsk 634021 Russia

Characteristic features of diffusion and diffusion controlled processes (grain boundary sliding, dislocation accommodation and grain boundary migration) in polycrystalline metals and alloys with bcc and fcc crystal lattice at annealing and creep have been examined. The interconnection and intereffect of diffusion processes, sliding and grain boundary migration as factors, determining the development of plastic deformation at examined conditions are analyzed. The peculiarities of grain boundary diffusion of substitution from impurity environment (coating) in nanostructured metals and alloys relative to the respective ones in coarse grained metals and alloys have been investigated. The physical reasons for considerable (by some orders of magnitude) increase of diffusion penetration of grain boundaries in nanostructured state are discussed. The features of manifestation of low temperature and/or high-strain rata superplasticity in nanostructured metals and alloys have been considered.

10:30 AM Break

10:45 AM

Micro SPD: *Yuri Estrin*[1]; Eugen Rabkin[2]; Ralph Hellmig[1]; Michael Kazakevich[2]; Aikaterini Zi[1]; [1]Clausthal University of Technology, Matls. Sci. & Tech., Agricolastr. 6, Clausthal-Zellerfeld 38678 Germany; [2]Technion, Matls. Engrg., Haifa 32000 Israel

It is proposed to use porous steel 'dies' to infiltrate them with a metallic material by a variant of the known forcefill process. Severe plastic deformation (SPD) that occurs in the material due to its flow through a tortuous channel is believed to lead to grain refinement and an improvement of mechanical properties. This process will be referred to as micro SPD. First experimental results on infiltration of steel with aluminum will be presented. The dependence of the penetration depth on the average pore size of the die material in the micrometer range will be discussed along with the results on the effect of the process parameters on the microhardness of Al.

11:00 AM

Processing and Microstructural Modelling of Equal Channel Angular Pressing for Ultrafine Grained Materials: *Hyoung Seop Kim*[1]; Yuri Estrin[2]; [1]Chungnam National University, Dept. of Metallurgl. Engrg., Yuseong, Daejeon 305-764 Korea; [2]Clausthal University of Technology, Inst. of Matls. Sci. & Tech., Agricolastr. 6, Clausthal-Zellerfeld D-38678 Germany

ECAP is a convenient forming procedure among various methods of severe plastic deformation to extrude material by use of specially designed channel dies to make an ultrafine grained material. The properties of the materials are strongly dependent on the plastic deformation behaviour during pressing, which is governed mainly by die geometries, material properties, and process variables. Because the evolution of microstructures and the mechanical properties of the deformed material are directly related to the plastic deformation, the understanding of the phenomenon associated with strain development is very important in the ECAP process. In this study, we describe a range of our continuum modelling and microstuctural modelling results of ECAP in order to illustrate the modelling applicability. For this purpose, the finite element results of ECAP for various geometric factors are described. The inhomogeneous deformation due to the hardening property of the material is explained. Lastly, modelling the microstructural evolution using a dislocation cell model during ECAP is presented.

11:15 AM

Grain Refinement Mechanisms During Severe Deformation of Aluminium Alloys: Effect of Material Variables: *Philip B. Prangnell*[1]; Chris P. Heason[1]; Pete J. Apps[2]; [1]University of Manchester, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2]Health and Safety Laboratory, Broad Ln., Sheffield S3 7HQ UK

The evolution of the deformed state during the severe deformation of aluminium alloys is discussed, focusing on the mechanisms involved and the effect of important material variables. High spatial and orientation resolution data is presented, obtained using FEG-SEM EBSD orientation mapping and Kuwahara filtering. It has been confirmed that microshear bands are responsible for the majority of new high angle boundary formed at moderate strains. By studying simple model alloys, it has been found that a fine initial grain size increases the rate of grain refinement at low strains, but has little influence at ultra high strains. It has also been show that coarse second phase particles can greatly accelerate the rate of grain refinement, whereas fine coherent particles inhibit the rate of grain refinement. These effects are most noticeable at moderate strains, but still lead to significant differences in the ultra-fine grain structure at very high strains (e.g. strain = 10). The development of strong textures has been found to inhibit, and in some cases, reverse grain refinement.

11:30 AM

Severe Plastic Deformation of Magnesium Alloys: *K. Xia*[1]; J. T. Wang[2]; X. Wu[1]; G. Chen[2]; [1]University of Melbourne, Dept. of Mechl. & Mfg. Engrg., Parkville, Victoria 3010 Australia; [2]Nanjing University

of Science and Technology, Sch. of Matls. Sci. & Engrg., Nanjing, Jiangsu 210094 China

Equal channel angular pressing (ECAP) was applied to magnesium alloys in order to refine grain structures. ECAP was carried out at various speeds for up to 8 passes at temperatures as low as 100°C with or without an applied back pressure at the exit channel. The application of a back pressure and a slower pressing speed were shown to be critical in deforming Mg alloys at lower temperatures without causing cracking. Room temperature mechanical properties were obtained by tensile and micro- and macro-hardness tests. With increasing ECAP strain, the initial coarse grained structure was transformed gradually into a submicron grained microstructure. The kinetics of this strain driven microstructure transformation was analysed. The approaches to refined microstructures and improved mechanical properties in Mg alloys through severe plastic deformation (SPD) were discussed based on a comprehensive review of previous works in the literature.

11:45 AM

Nanoscaled Structure of a Cu-Fe Composite Processed by High Pressure Torsion: *Xavier Sauvage*[1]; [1]Université de Rouen, GPM - UMR CNRS 6634, Inst. des Matériaux de Rouen, Site du Madrillet, Saint-Etienne-du-Rouvray 76801 France

The microstructure evolution of pure metals under severe plastic deformation has been widely investigated during the past decade. Much less is known and reported about the behaviour of multi-phase materials. In this paper, a Cu-Fe(10%vol.) composite material was processed by High Pressure Torsion. The resulting nanostructure was investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), micro-hardness measurement and 3D atom probe analysis (3D-AP). Experimental data reveal a strong decrease of the grain size down to 50nm, which is much smaller than in pure iron or pure copper processed by HPT. Moreover, a significant interdiffusion of Cu and Fe was pointed out thanks to 3D-AP investigations. The mutual solubility of these two components is very low at room temperature, thus the alloying mechanism will be discuss as a result of the severe plastic deformation. Finally, this nanostructure will be compared with Fe-Cu nanocrystalline powders prepared by ball milling.

12:00 PM

Microstructure and Properties of an Ultrafine-Grained Low Carbon Steel Processed by Equal-Channel Angular Pressing: *Jing Tao Wang*[1]; Chen Xu[2]; Zhong Ze Du[3]; Terence G. Langdon[2]; [1]Nanjing University of Science and Technology, Sch. of Matls. Sci. & Engrg., No. 200 Xiaolingwei, Nanjing 210094 China; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [3]Xi'an University of Architecture & Technology, Sch. of Metallurgl. Engrg., Xi'an 710055 China

Ultrafine-grained low carbon steel (Fe-0.15%C-0.52%Mn) was fabricated by equal channel angular pressing (ECAP) at room temperature through routes Bc and C up to 10 passes. With route C, nearly parallel bands of an elongated substructure pertains in the ferrite microstructure after ECAP from 1 to 11 passes, with a slight decrease of the band width from 0.3-0.4 to 0.2-0.3?n micrometers. With route Bc, an equiaxed grain structure with a grain size of about 0.25 micrometers forms after 4 passes of ECAP. A tensile strength over 1000 MPa was achieved after only 2 passes of ECAP via route Bc with limited uniform tensile elongation. Annealing at temperatures below or above the recrystallization temperature after ECAP changes the tensile behavior significantly.

12:15 PM

Strain Rate Sensitivity of Flow Stress of Ultrafine Grained Cu and Ti: *Yujiao Li*[1]; Xiaohui Zeng[1]; Philip Eisenlohr[1]; *Wolfgang Blum*[1]; [1]Universität Erlangen-Nürnberg, Technische Fakultät, Inst. f. Werkstoffwissenschaften LS 1, Martensstr. 5, Erlangen 91058 Germany

The deformation behaviors of ultrafine-grained (UFG) pure Cu with 350nm grain size and of commercially pure (CP) UFG Ti with 300nm average grain size produced by severe plastic deformation through equal channel angular pressing (ECAP) were investigated as function of temperature and strain rate at low homologous temperatures within the range of grain stability. A distinct increase of strain rate ensitivity was observed in UFG Cu with increasing temperature and decreasing train rate which is much stronger than in coarse-grained (CG) Cu. This causes UFG Cu to become softer than CG Cu as temperature and strain increase. This softening effect is explained by diffusion-controlled annihilation of lattice dislocation at grain boundaries. A simple model is used to demonstrate this behavior. For Ti the corresponding difference between UFG and CG variants is much less pronounced. This is discussed in terms of impurities.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Novel Processes II

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Wednesday PM, February 16, 2005 — Room: 2009 — Location: Moscone West Convention Center

Session Chairs: John E. Smugeresky, Sandia National Laboratories, Dept. 8772, Livermore, CA 945510969 USA; John E. Carsley, General Motors, Matls. & Processes Lab., Warren, MI 48090 USA

2:00 PM

Nano Metal Powders - Real Life Applications: *Alan Rae*[1]; [1]NanoDynamics Inc., 901 Fuhrmann Blvd., Buffalo, NY 02035 USA

Nanotechnology provides us with a toolkit of techniques that allows us to tailor crystallinity, particle size and surface condition of metals. This allows us to modify reactivity as well as chemical, physical and electrical properties. There are challenges however in preventing agglomeration and unwanted reactions, in scaleup, economics, and applications engineering. This paper reviews specific developments in commercial metal production focusing in particular on Cu, Ag, Fe, Ni and Ti and their applications in electronic, medical and engineering applications.

2:20 PM

Improvement of Ductility and Toughness of Cryomilled Nanostructured Al Alloys for Applications: *Bing Q. Han*[1]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Chem. Engrg. & Matls. Sci., Davis, CA 95616 USA

There are a number of synthesis techniques (for instance, cryomilling) that are capable of producing structural materials with grain sizes of the 10-500 nm range in large quantities. Although a higher strength is generally observed in most of nanostructured materials, their fracture toughness is low and their ductility is limited. These characteristics regarding toughness and ductility have an adverse effect on their potential as structural material. In the present study, two approaches based on microstructural control to improve ductility and toughness of cryomilled Al alloys were reported. The first approach involves architectural designs of nanostructured materials that contain multiple length scales by blending two types of (unmilled coarse grained and cryomilled nanostructured) Al powders, followed by consolidation and extrusion. The second viable approach is to perform a controlled heat treatment on cryomilled nanostructured Al alloys to introduce an intrinsic bimodal microstructure.

2:40 PM

Spray Rolling Aluminum Strip for Transportation Applications: *Kevin M. McHugh*[1]; Yaojun Lin[2]; Yizhang Zhou[2]; Enrique J. Lavernia[2]; Jean-Pierre Delplanque[3]; Sam B. Johnson[3]; [1]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., PO Box 1625, MS 2050, Idaho Falls, ID 83415-2050 USA; [2]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA; [3]Colorado School of Mines, Engrg. Div., Golden, CO 80401 USA

Spray rolling is a novel technology in which a molten aluminum alloy is atomized and the resulting spray is deposited on the rolls of a twin-roll caster to produce aluminum strip. A combined experimental/modeling approach has been followed in developing this technology with active participation from industry. The feasibility of this technology has been demonstrated at the laboratory scale and it is currently being scaled-up. This paper provides an overview of the process and compares the microstructure and properties of spray-rolled 2124 and 5083 aluminum alloys with commercial ingot-processed material.

3:00 PM
Advanced Atomization Process for Metal Spray Deposition: *Peter Kelly Sokolowski*[1]; Volker Uhlenwinkel[1]; Yunzhong Liu[1]; Iver E. Anderson[2]; [1]Institut fuer Werkstofftechnik, Verfahrenstechnik, Badgasteiner Str. 3, Bremen 28359 Germany; [2]Ames Laboratory, Matls. Sci. & Engrg. Dept., 222 Metals Dvpt. Bldg., Ames, IA 50011 USA

Spray deposition typically utilizes a free-fall atomizer (FFA) with a well established understanding of its capabilities and disadvantages. With an ever increasing demand for superior products and optimal processing conditions in mass production applications, including automotive parts (e.g., cylinder liners), a close-coupled atomizer (CCA) was studied for spray forming. Compared to the FFA, a CCA requires less gas to produce the same particle velocities with the possibility of increasing these velocities and maintaining gas consumption below that of FFA. By increasing particle velocity, it is believed higher densities can be achieved during colder spray conditions. In this study, three materials were sprayed: Sn, bearing steel AISI 52100, and Cu-6wt%Sn on tube substrates with diameters of 80mm and 100mm. Results include density measurements, gas consumption trends, surface temperatures, and particle velocities and diameters obtained with the use of Phase Doppler Anemometry (PDA).

3:20 PM
Control of Mn-Based Precipitate Distribution and Morphology in Aluminum Alloys for Elevated Temperature Applications: *M. C. Carroll*[1]; D. Shull[2]; E. A. Ott[3]; M. J. Mills[1]; G. S. Daehn[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA; [2]Transmet Corporation, Columbus, OH 43228 USA; [3]GE Aircraft Engines, Cincinnati, OH 45215-1988 USA

Manganese is used in small concentrations (generally 1.2 wt% or less) as an alloying element in a number of aluminum alloys, principally as a grain-refining agent. Often overlooked as an important player in emerging aluminum alloy developments, manganese behavior has not been thoroughly evaluated from a processing standpoint in order to improve final material characteristics and achieve more aggressive performance levels. In this study, a more comprehensive understanding of casting and homogenization treatments was sought in order to specifically evaluate Mn-based eutectic constituent and precipitate morphology, and as a result gain a better understanding of high-temperature microstructural evolution. Casting distributions and additional precipitation behavior were evaluated using conductivity measurements that were correlated with direct microstructural observation and characterization via scanning and transmission electron microscopy. Based on the stability of the Mn-based precipitate substructure, novel te chniques for maintaining control over particle distribution along with the resulting increase in potential applications for high-grade Mn-containing aluminum alloys are discussed.

3:40 PM Break

3:55 PM
Laser Direct Fabrication of a Glass-Forming Fe-Based Alloy: *John E. Smugeresky*[1]; Baolong Zheng[2]; Yizhang Zhou[2]; Enrique J. Lavernia[2]; [1]Sandia National Laboratories, Laser Engineered Matls., MS 9402, Livermore, CA 94551 USA; [2]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

Laser Engineered Net-Shaping (LENS®), a promising direct manufacturing technique for fabricating near-net shape fully dense metallic components, tailors microstructures by controlling the cooling rate. A relatively rapid solidification of molten metal occurs as a matter of course. Many new metallic materials for automotive application with isotropic properties and high structural efficiency are dependent upon rapid solidification to achieve appropriate microstructure. In this paper, the potential of LENS® for the fabrication of net-shape parts using a glass-forming Fe-base alloy is explored. Fe-based alloy powder is used for laser deposition of bulk coupons made over a range of process parameters to determine adequacy of the LENS® process solidification rate. The microstructure evolution, thermal stability, transformation sequence and micro-hardness of the laser deposited parts, as a function of processing parameters, are investigated using XRD, SEM, TEM, and DSC. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

4:15 PM
Segregation Effects During Semi-Solid Forming of Creep Resistant Mg-Al Alloys: *Frank Ajersch*[1]; Faouzi Messaoud[1]; [1]Ecole Polytechnique, Cheml. Engrg., PO Box 6079, Sta. "centre-ville", Montreal, Quebec H3C 3A7 Canada

The forming of Mg-alloy components for structural applications in the transportation industry has gained considerable interest with the development of new creep resistant high strength Mg-Al alloys. The improvement of these properties is based on small additions of structure modifying elements such as Ca and Sr which influence the solidification structure. The properties and microstructure of these alloys formed in the semi-solid state is also highly dependant on the rheological properties. Segregation of the liquid and solid phases are particularly prevalent in slow deformation processes but can be minimized in rapid forming processes such as Thixomolding®. Experiments were carried out using a squeezing flow technique over a range of shear rates in order to quantify the segregation observed for conventional and creep resistant alloys. Correlations between shear stress, shear rates and the microstructure are presented.

4:35 PM
An Investigation of Liquation Formation in the Heat-Affected Zone of Inconel 718 and Inconel 625 Subjected to GTAW: *Shenavia Wilkerson Howell*[1]; Viola L. Acoff[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487 USA

Nickel-based superalloys Inconel 718 and Inconel 625 are widely used in the aerospace industry, particularly in the gas turbine engine components of aircrafts. During repair welding, these aircraft components are often subjected to numerous cycles consisting of welding followed by postweld heat treatment. Nickel-based superalloys are sensitive to liquation cracking in the HAZ upon postweld heat treating. In the present study, bead-on-plate welds were placed on each specimen using gas tungsten arc welding (GTAW). The effect of welding parameters on liquation in the heat-affected zone (HAZ) of Inconel 718 and Inconel 625 were investigated. The welding current, welding speed and welding environment (i.e. air vs. inert atmosphere) were varied. The resulting welds were characterized using scanning electron microscopy, transmission electron microscopy, and mechanical testing. The purpose of this study is to investigate the conditions that promote liquation in the HAZ during the welding of nickel-based superalloys.

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Welding of Single Crystal Nickel-Base Superalloys: *Sizhao Wang*[1]; Ali Merati[1]; Henry Saari; [1]National Research Council, Inst. of Aeros. Rsch., Bldg. M-13, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

Single crystal nickel-base superalloys have been used as gas turbine blade materials for the last few decades due to their excellent high temperature mechanical properties and corrosion resistance. The high costs associated with replacement blades calls for new repair solutions to extend their life cycles, and welding is an efficient process to repair the blades that are worn or damaged during service. Feasibility of both fusion welding and solid state welding is determined by the ability to produce desired microstructures, which yield the required service properties. Therefore, numerous welding trials and mechanical tests must be employed to determine the optimum welding conditions and method. Weldability of single crystal PWA1484 is investigated in this paper in terms of various welding parameters and directions. Pre and post welding heat treatments are applied to the material to optimize the welding conditions. The crack morphology is observed and microstructure development is analyzed using scanning electron microscopy (SEM), optical microscopy (OM) and indexing electron backscattered diffraction (EBSD) patterns. Several potential difficulties of welding single crystal, such as the susceptibility of hot cracking and stray grain formation, which are related to weld pool shape and grain growth orientation, are discussed. Micro-hardness testing is also carried out to characterize the localized microstructural features and mechanical properties of autogenously welded PWA1484.

Alumina and Bauxite: Precipitation

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday PM Room: 2005
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Chris Vernon, CSIRO Minerals, Bentley, Western Australia 6982 Australia

2:00 PM
Soda Incorporation During Hydrate Precipitation: *Chris Vernon*[1]; [1]CSIRO Minerals, PO Box 90, Bentley, WA 6982 Australia

The economic need to achieve high precipitation yields and tough alumina must be balanced against another important quality consideration - occluded soda. Although there are a number of publications that develop hypotheses for the mechanism of soda incorporation and some that give mathematical relationships to describe the rate of soda incorporation, none attempt to do this with any detailed understanding of the hydrate (gibbsite) growth mechanism. The present work describes sodium incorporation as a function of growth rate and of crystal defect generation, and relates the incorporation of sodium ions into gibbsite crystals to the growth mechanism. A mathematical model for incorporation is developed along statistical mechanical principles and the contribution of organic carbon is explored.

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Dynamic Adsorption Isotherms for Some Hydrate Active Organics and Selected Degradation Products With Implications for Gibbsite Precipitation Yields: Nicolas-Alexandre Bouchard[2]; Raymond Breault[1]; Frédéric Picard[1]; Yannick Chouinard[2]; *Hugues Ménard*[2]; [1]Alcan International Ltd., 1955 Mellon Blvd., Jonquière, Québec G7S 4K8 Canada; [2]Université de Sherbrooke, Dépt. de Chimie, 2500 Blvd. Université, Sherbrooke, Québec J1K 2R1 Canada

The negative impact of organic compounds present in Bayer liquor is well known. To date, the industry mostly relies on bulk removal approaches to control the effect of organics in the Bayer process. However the relationship between total organic carbon and precipitation yield suggests the selective interaction between some organics and precipitating gibbsite is important to plant productivity. These specific organics are referred to as Hydrate Active Organics (HAOs). A better understanding of the characteristics and properties of HAOs could open the way to their selective removal. In this study, the focus was on substituted aromatic compounds that were either detected in Bayer precipitated hydrate or that were generated by the degradation of lignin in caustic solution. Dynamic adsorption isotherms for these compounds obtained using liquid chromatography will be presented to differentiate their adsorptive behaviour towards hydrate. Results for lignin-derived organics and for those obtained from oxygenated and hydrogenated parent molecules, will assist in understanding their relative impacts on gibbsite precipitation yield. These results may show a possible alternative to the complete removal of organics by selective modification of hydrate active organics.

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Boehmite vs Gibbsite Precipitation: *Joanne Loh*[1]; Chris Vernon[1]; Melissa Loan[1]; Greta Brodie[1]; [1]CSIRO Minerals, PO Box 90, Bentley, Western Australia 6982 Australia

Boehmite (monohydrate) precipitation, in place of gibbsite (trihydrate), has been discussed in the literature as a possible energy-saving step in alumina refining. Indeed, the calcination of monohydrate rather than trihydrate would save approximately 12% of the energy consumed in an alumina refinery. These previous investigations have not focussed on the economic and technical feasibility of the process. The current investigation takes a pragmatic approach, measuring precipitation rates, determining product phase and particle size distribution, and assessing the impact of impurities and probe species in an attempt to determine the viability and possible improvements to such a process. The present work indicates that boehmite precipitation rates are approximately 2 orders of magnitude slower than for gibbsite under identical conditions. Agglomeration seems not to occur. The particle size distribution is too fine and the product is unsuitable for calcination. However, the work has further improved the fundamental understanding of gibbsite precipitation and a hypothesis for the mechanism will be presented.

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The Effects of Changes in Solids Concentration on Seed Balance Parameters in Alumina Refinery Seed Classification Systems: *Walter Mason Bounds*[1]; [1]2583 Woodland Ridge Blvd., Baton Rouge, LA 70816 USA

An important consideration in controlling alumina refinery seed classification systems is ensuring that seed produced matches quantity and particle size distribution with seed charged. When this condition is not satisfied, the system may not be stable, resulting in changing seed inventory quantities and particle size distribution. Previous papers have described methods for evaluating operating parameters for individual classifiers as well as seed balance parameters for alumina refinery seed classification systems. In this paper, information from previous papers is utilized to simulate a classification system and determine the effects of changes in solids concentration on seed balance parameters, including mass balance and particle size distribution. Solids concentration effects on out-of-balance cases are also established to aid in illustrating consequences.

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Study of Agglomeration During the Precipitation of Sodium Aluminate Solution: Jiangfeng Zhang[1]; Zhoulan Yin[1]; Qiyuan Chen[1]; [1]Central South University, Physl.-Chmst. Inst., Changsha, Hunan 0086 China

Agglomeration of gibbsite crystals is an important stage in the Bayer process. In the present study, the effect of the crystallization temperature, seed mass, stirring rate, caustic concentration of liquor, molecular rate were investigated. And interpretation of the results was proposed.

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Analysis on the Particle Size Information in Sodium Aluminate Solution During Seeded Agglomeration: Wangxing Li[1]; Jianguo Yin[2]; Qiyuan Chen[2]; Zhoulan Yin[2]; [1]Zhengzhou Research Institute of Chalco, Zhengzhou, Henan 450041 China; [2]Central South University, Physl.-Chmst. Inst., Changsha, Hunan 410083 China

Seeded agglomeration was studied in sodium aluminate solution (Na2Ok=140g?l-1,?¿k=1.37) at 75°.Conclusions were made from the particle size information that agglomeration takes place mainly for particles with the sizes of 0~10?Êm at first and then the larger ones. For particles with certain size, peaks and troughs appear alternately on the curves of the volume percentage to time. The appearance time is basically the same and moves backward when particle size is larger. The kinetic parameters can be further calculated based on the information.

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Study on Negative Effect of K2O on Precipitation Procedure: *Qun Zhao*[1]; *Yanli Xie*[2]; [1]CHALCO, Zhengzhou Rsch. Inst. of Light Metals, No.82, Jiyuan Rd., Shengjie Dist., Zhengzhou, Henan 450041 China; [2]Northeastern University, Sch. of Matls. & Metall., No.11, Ln. 3, Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

The content of impurity K2O in Bauxite is only several thousandths, but it can accumulate in the process and over 10g/l (sometimes ~40g/l), which brings serious negative effect on precipitation, results in fine particles and low strength hydrate. It's demonstrated that the existance of K2O in sodium aluminate liquors can: (1) inhibit the agglomeration between fine particles of hydrates and leads to fine products; (2) change the precipitation mechanism of gibbsite. The products of hydrate grow from sodium aluminate solution is sphere polycrystalline with inlaid structure, which has good ability to resist-attrition. However, the hydrate from potassium aluminate liquor is fragile with needle structure. (3)The existence of K+ in sodium aluminate solution can increase the solubility of SiO2 and then stable the solution, hence decreasing the product yield.

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The Application of Hydrate Seed in Carbonization of Sodium Aluminate Liquors: *Wang Zhi*[1]; Bi Shiwen[2]; Yang Yihong[2]; Yuan Zhangfu[1]; [1]Chinese Academy of Sciences, Inst. of Process Engrg., Zhong Guan Cun, Haidian Dist., Beijing 100080 China; [2]Northeastern University, Sch. of Matls. & Metall, Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

Carbonization was investigated through adding seeds to sodium aluminate liquors in order to improve the particle size and strength of products. All results were explained by analyzing the data obtained and the parameters favoring for improving the product granularity and strength were determined through the orthogonal experiment. By comparison of mean particle size, attrition index and SEM photograph of the products at the conditions with and without seed on operation conditions, it was showed that seeds played important roles in the carbonization process, and the products average particle size was influenced by that of seed, but its effect was related with seed type and seed ratio. In addition, seeds can accelerate the inter-growth of single crystals and gibbsite with inlaid structure had high attrition resistance, as a result the strength of products was increased by 3-25%.

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DFT and Ab Initio Calculation on Thermochemistry of Al6(OH)18(H2O)x(x=0-6), Al(OH)3-3 and Al(OH)4(H2O)2-: Wu Zheng Ping[1]; Chen Qi Yuan[1]; Yin Zhou Lan[1]; [1]Central South University, Coll. of Chmst. & Cheml. Engrg., Changsha City, Hunan Province 410083 China

Enthalpies of formation and Gibbs free energy of formation of Al6(OH)18(H2O)x (x=0-6), Al2O3·2H2O,Al(OH)3-3, Al(OH)4(H2O)2- and Al(OH)4- are calculated at B3LYP/6-31G, 6-31G and B3LYP/6-311++G(3d2p) levels respectively with Dipole & Sphere solvent model by DFT and ab initio methods. Especially, in the process of Al(OH)3 crystals precipitating from supersaturated caustic aluminate solution,

Al6(OH)18(H2O)x(x=0-6) is the favorable growth unit, Al(OH)3-3 is the structure unit of growth units and Al(OH)4(H2O)2- is the minimum growth unit. Using DFT Method with Dipole & Sphere solvent model, ÄfHm(298K)of Al6(OH)18(H2O)x(x=0-6) and Al2O3·2H2O are calculated at B3LYP/6-31G level to be -5419.471013, -5613.439509, -5867.816332, -6018.601893, -6263.675488, -6473.281202, -6698.522508 and -1579.549361 kJ·mol-1, ÄfHm(298K) of Al(OH)6-3, Al(OH)4(H2O)2- and Al(OH)4- are calculated at B3LYP/6-311++G(3d2p) level to be -811.105741, -1993.955225 and -1428.924169 kJ·mol-1. The results are relatively consistent with the available experimental and the theoretical values. ÄrHm(298K)and ÄrGm(298K) of various sorts of hydration reaction of Al6(OH)18(H2O)x (x=0-6) are calculated and the stability of bondage H2O in Al6(OH)18(H2O)x (x=0-6) are discussed. The various combination modes of Al6(OH)18(H2O)6, Al(OH)3-3 and Al(OH)4(H2O)2- are analyzed.

Aluminum Reduction Technology: Cell Stability

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday PM Room: 2002
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Donald P. Ziegler, Alcoa Primary Metals, Alcoa Techl. Ctr., Alcoa Ctr., PA 15069-0001 USA

2:00 PM
The Influence of the Basic Flow and Interface Deformation on Stability of Hall-H´eroult Cells: *Haijun Sun*[1]; Oleg Zikanov[2]; Donald Ziegler[3]; Bruce A. Finlayson[1]; [1]University of Washington, Chem. Engrg. Dept., PO Box 351750, Seattle, WA 98195 USA; [2]University of Michigan, Dept. of Mechl. Engrg., 1334 Engrg. Complex, Dearborn, MI 48128-1491 USA; [3]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA

We use the method of linear stability analysis to study the effect of background melt flows and interface deformation on stability characteristics of Hall-Héroult reduction cells. The linearized perturbation equations are the two-dimensional shallow water model. Two kinds of background state are generated by using some artificial forcing. The critical friction coefficients for these states and no base flow situation are compared. It is found that some background states, for example, mode (3,1) of melt flows and interface deviation, stabilize the cell. Other states, noticeably, modes (1,1) and (2,1) of interface deviation, have strong destabilizing effect.

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Stability of Hall-Heroult Cells: *Kjell Harald Kalgraf*[1]; [1]Elkem Aluminium, Elkem Al Rsch., Box 8040 Vaagsbygd, NO 4675 Kristiansand Norway

The motion and stability of the metal electrolyte interface is determined by Navier-Stokes equation. The equation may be decomposed into two independent equations by taking the divergence and curl of Navier-Stokes equation. The curl determines the flow of metal and bath and is independent of gravitation and hydrostatic pressure. Using the equilibrium velocity from the flow we find that magneto-gravitational waves depend on the sum of hydrostatic and magnetic pressure.From the divergence we can derive an invariant and a criterium of instability that resembles Sele's criterium of stability. Basically, stability depend on the curvature of hydrostatic and magnetic pressure, and this may in turn be expressed as a dependence on current density, ACD, metal height, and density difference between metal and bath.

2:50 PM
Weakly Coupled Thermo-Electric and MHD Mathematical Models of an Aluminium Electrolysis Cell: *Marc Dupuis*[1]; Valdis Bojarevics[2]; [1]GeniSim Inc., 3111 Alger St., Jonquiere, Quebec G7S 2M9 Canada; [2]University of Greenwich, Sch. of Computing & Math., 30 Park Row, Greenwich, London SE10 9LS UK

In the present study, the full 3D thermo-electric model of a 500 kA demonstration cell has been weakly coupled with the non-linear wave MHD model for the full version of the same cell. In the MHD model, the horizontal ledge distribution calculated in the thermo-electric model has been incorporated as part of the model geometry. In the thermo-electric model, the velocity fields calculated by the MHD model in both the bath and the metal have been used to setup the local heat transfer coefficients at the liquids/ledge interface. Both models have been solved alternatively until a convergence for the horizontal ledge distribution has been obtained.

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Wave Mode Coupling and Cell Instability in Aluminum Reduction Cells: *Nobuo Urata*[1]; [1]Consultant, 770 Cascade Dr., Sunnyvale, CA 94087 USA

The interfacial surface waves cause uneven anode-cathode separation in the electrolytic zone and lower the efficiency of electrolysis in the aluminum reduction cells. In the past, the coupled partial differential equations, describing the electromagnetic perturbation in the cell, were formulated and solved with the various mathematical methods. Fourier expansion method is used for understanding the mechanism of destabilization of the two liquid interface as an interaction of the various non-perturbed waves. A proper mathematical treatment of the boundary condition, a critical factor for solving the equations, is presented. The result is summarized as the mode interactions, governed by the symmetry of the vertical magnetic field and the symmetry of the wave modes. The dominant mechanism of the instability is explained and the various practical methods for magnetic field compensation are discussed.

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Determination and Influence of the Ledge Shape on Electrical Potential and Fluid Motions in a Smelter: *Michel V. Romerio*[1]; Michel Flueck[1]; Jacques Rappaz[1]; Yasser Safa[1]; [1]Swiss Federal Institute of Technology, IACS, Lausanne 1015 Switzerland

The temperature field and the ledge shape in a whole smelter are obtained by solving simultaneously the system of equations formed by: a non-linear convection-diffusion heat equation, which can be considered as a Stephan problem in enthalpy and temperature in the domain of the cell occupied by fluids and ledge, Navier-Stokes equations, with a free interface, in the fluid domains and Maxwell equations in the whole space. The source term of the heat equation results from the Joule effect due to the electrical current crossing the cell. We use artificial time dependence of the heat equation to follow the solidification front with a Chernoff scheme. Results of 3-D numerical calculations showing ledge shape temperature and velocity fields as well as the electrical potential for an operating cell are presented.

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Current Field in an Aluminum Electrolysis Cell: Augustin Moraru[1]; *Nobuo Urata*[1]; Aureliu Panaitescu[2]; [1]"Politehnica" University of Bucharest, Electl. Engrg. Dept., Splaiul Independentei No. 313, Bucharest 77206 Romania

The distribution of the current in an aluminium electrolysis cell is discussed, accounting for the shape and position of the anode blocks and the influences of ledges and cathode bars. The effect of the velocity field in the molten aluminium and electrolyte make the object of a second stage analysis. It is shown that the DC current problem in the electrolysis cell may be split into three almost independent problems related to the cell structure-induced features. The aluminium pad is an almost equipotential body, within the limits of a few tens of millivolt, while the overall voltage drops in the cell are within the order of volts. This allows the separation of electrical problem of the cell into upper, lower and middle parts. In the upper part problem the location of the anodes and ledges, the height of the anode blocks and the distances from the anodes to the aluminium pad are given. In the lower part problem the current distribution is determined by the conductive properties of the carbon lining and the cathode collector bars, as well as the contact resistance between the bars and the cathode blocks. In the middle-part problem the surface current distributions are taken from the previous two problems. The impressed currents produced by the motion within the molten media, due to Laplace forces through the interaction of the current density with the magnetic field intervene in the balance equations. The calculation of the velocity field in the molten media implies the solution to a magnetic field problem and a Navier-Stokes problem, coupled by a Laplace body force term. Because the velocity field may modify the current distribution and the magnetic field, we estimated their influence. The real working conditions lead to a non-symmetrical current distribution, therefore the overall problem must be considered, without simplifying, construction symmetry assumptions. The electrokinetic problem is of very great dimension and only the above mentioned splitting allowed its solution within satisfactory limits.

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Modeling Magnetohydrodynamics of Aluminum Electrolysis Cells with ANSYS and CFX: *Dagoberto S. Severo*[1]; Andre F. Schneider[1]; Elton C.V. Pinto[1]; Vanderlei Gusberti[1]; Vinko Potocnik[2]; [1]PCE Engenharia S/S Ltda, Rua Felix da Cunha, 322, Porto Alegre, RS 90570.000 Brazil; [2]Vinko Potocnik Consultant, Jonquière, Québec G7S 3C7 Canada

Modern aluminum industry is studying ways to increase the efficiency of reduction cells in new and retrofit smelters. Numerical simulation has become a very effective tool for analyzing such complex processes. This paper presents magnetohydrodynamics (MHD) simulations of a new, study case cell technology. A 3-D model was developed by coupling commercial codes ANSYS and CFX, via in-house programs and customization subroutines. A detailed electric-magnetic model was built in ANSYS, which uses Finite Element Method. Steady state and transient MHD flows in the cell were calculated with CFX, which uses Finite Volume Method. Metal and bath were treated as multiphase flow. The homogeneous VOF (Volume of Fluid) model, available in CFX, was used to calculate bath-metal interface in steady state and transient regimes. The transient simulation of the bath-metal interface was used for the study of cell stability.

5:10 PM
Compensation for the Magnetic Field of the End Reduction Cells in Potlines: *Alexander Gusev*[1]; Leonid Krylov[2]; Vitaliy Platonov[1]; Petr Vabishchevich[3]; [1]Engineering-Technological Center Ltd, RUSAL, Pogranichnikov 37, Krasnoyarsk 660111 Russia; [2]RUSAL - Management Company, Nikoloyamskaya 13/1, Moscow Russia; [3]Russian Academy of Science, Inst. for Mathematical Modlg., Miusskaya pl. 4, Moscow Russia

The end reduction cells in potlines at aluminum smelters operate in the conditions, which substantially differ from those of the series reduction cells. This is caused, first of all, by the fact that the magnetic field in the potline substantially changes in going from the series reduction cells to the end ones. To minimize the negative consequences of this effect, nonstandard busbars are frequently used for the extreme reduction cells. In the work, another possibility is discussed, which is characterized by the compensation of the magnetic field by means of the development of special busbar configurations for connecting the end reduction cells, arranged side-by-side, of the adjacent rows in a potline. The efficiency of this technical solution was estimated on the basis of mathematical simulation. The proposed compensation scheme is realized, and its operational capability is proved at the Sayanogorsk aluminum smelter of the Russian Aluminum Company.

WEDNESDAY PM

Aluminum Reduction Technology: Emerging Technologies

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday PM Room: 2003
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: John J. Chen, University of Auckland, Dept. of Cheml. & Matls. Engrg., Auckland 00160 New Zealand; Jomar Thonstad, Norwegian University of Science and Technology, Dept. of Matls. Tech., 9471 Trondheim Norway

2:00 PM
Evaluating and Funding New Technologies to Support the U.S. Aluminum Industry: *Thomas Robinson*[1]; [1]U.S. Department of Energy, Industl. Technologies Prog., 1000 Independence Ave. SW (EE-2F), Washington, DC 20585-0001 USA

Proposals for the research and development of new technologies that reduce energy consumption in the U.S. aluminum industry are evaluated and funded by The U.S. Department of Energy, Energy Efficiency and Renewable Energy (EERE) Office's Industrial Technologies Program (ITP). These proposals offer real opportunities to encourage and accelerate the development of new technologies that improve energy efficiency while strengthening the U.S. aluminum industry. ITP's Aluminum Industries of the Future (IOF) is currently partnering with more than 70 firms in over 35 R&D projects. The IOF portfolio of projects ranges from high-risk reduction technologies to high-value melting technologies. This paper reports on how the Aluminum IOF portfolio is performing and how proposals are evaluated, selected and funded. The authors provide researchers with working knowledge of the baselines, benchmarks and bandwidths used to evaluate new technologies and with the proposal merit review selection process.

2:20 PM
Will New Technologies Sustain the U.S. Primary Aluminum Industry?: *William T. Choate*[1]; Majeed Aziz[1]; Rennie Friedman[1]; [1]BCS, Incorporated, 5550 Sterrett Place, Ste. 306, Columbia, MD 21044 USA

The high cost of electricity in the Northwest has resulted in the "moth balling" of nearly forty percent of the U.S. capacity to produce primary aluminum. The very large economies-of-scale needed for Hall-Heroult facilities, coupled with their dependence on a constant low-cost supply of electric power severely limits any potential of locating new primary production facilities in the United States. However, advances in modeling, new materials and novel engineering approaches applied to non traditional primary processes show promise for changing the industry and boosting U.S. primary capacity. This paper speculates on the industrial structure and economic impacts of inert electrodes, multi-polar cells, carbothermic, aluminum chloride and ionic liquid primary production technologies. The projected benefits include: lower electrical use, economies-of-scale at significantly lower volumes, end-user co-location, ability to use off-peak low-cost power, and more... If successfully developed, these technologies could revolutionize the U.S. primary aluminum industry.

2:40 PM
Thermal Stabilities and Viscosities of Low Temperature Aluminum Electrorefining Electrolytes: Di-Alkyl Imidazolium Chloride Ionic Liquids: *Venkat Kamavaram*[1]; R. G. Reddy[1]; [1]University of Alabama, Metallurgl. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Thermal and physical properties of di-alkyl imidazolium chloride ionic liquids were studied for their application in low temperature aluminum electrorefining. The ionic liquids used in this study are 1-butyl-3-methyl imidazolium chloride (C_4mimCl) and 1- hexyl-3-methyl imidazolium chloride (C_6mimCl). Thermal properties of these ionic liquids were studied in the temperature range 150°-200°C under controlled argon atmosphere for 15 hrs. For C_6mimCl, weight loss measured using TG analysis varied from 4 to 85 wt% as the temperature increased from 150° to 200°C. Absolute viscosity of chloroaluminate melts such as C_4mimCl+ $AlCl_3$ and C_6mimCl+$AlCl_3$ were determined as function of temperature and concentration of $AlCl_3$. Non-Arrhenius behavior of absolute viscosities with temperature was observed. Aluminum electrorefining in chloride ionic liquid electrolytes at low temperatures was successfully demonstrated.

3:00 PM
Coprocessing at Cement Plant of Spent Potlining from the Aluminum Industry: *Valerio A. Gomes*[1]; Paulo Z. Drumond[1]; Jose Olimpio P. Neto[2]; Abraão Rodrigues Lira[2]; [1]Consorcio de Aluminio do Maranhao - Alumar, Environml. Safety & Health, Br 135, Km 18 - Distrito Industrial de Pedrinhas, Sao Luis, Maranhão 65095604 Brazil; [2]Cimento Poty S/A, Quality & Environmental, Sítio Santa Helena S/N, Sobral, Ceará 62114-000 Brazil

In line with the concept of Sustainable Development, the Aluminum Consortium of Maranhão [ALUMAR] in partnership with Cimento Poty S/A [Poty Cement Inc.], implemented coprocessing of spent potlining from the aluminum industry, in rotary clinker kilns, on the basis of CONAMA resolution No. 264 of 1999, from the Environmental Council [Conselho Nacional do Meio Ambiente - CONAMA]. Due to its physicochemical characteristics, the addition of Spent Potlining (SPL), as secondary raw material and secondary fuel, entails economic and non-economics benefits for cement production and also safely eliminates a waste considered class I - hazard waste, by Standard ABNT-NBR 10004 of the Brazilian Technical Standards Association. The purpose of the present study is to present the main stages in the process of generation, characterization, and coprocessing of SPL in cement industries, as well as its economic and non-economic benefits and environmental aspects.

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Development of Detoxifying Process for Spent Potliner in CHALCO: *Wangxing Li*[1]; Xiping Chen[1]; [1]Zhengzhou Research Institute of CHALCO, Shangjie, Zhengzhou 450041 China

A novel detoxifying process of spent potliner is described in this paper. Spent potliner is treated in a rotary kiln with special additives, discharge from the kiln reacted with lime solution. Additives are usu-

ally limestone and commercial wastes. The effect of additives, temperature and lime solution on the process is discussed respectively. The result shows that: It is feasible to detoxify SPL by the process, soluble F- and CN- in the spent potliner can be decreased more than 95%, the final solid slag meets the requirement of China national environmental protection standard. There are several advantages of the process: All cuts of SPL can be handled through the process, better utilisation of commercial wastes. The permitting and operating of the process and facilities are verified. A pilot plant is being constructed at a scale of 500-1000 tons per year.

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3:55 PM
Greenhouse Gas Emissions from Aluminum Carbothermic Technology Compared to Hall-Hèroult Technology: Hilde Myklebust[1]; *Pål Runde*[2]; [1]Elkem ASA, Energy Div., PO Box 5211 Majorstuen, N-0303 Oslo Norway; [2]Elkem ASA Research, PO Box 8040 Vaagsbygd, 4675 Kristiansand Norway

Introduction of the Aluminum Carbothermic Technology (ACT) in the aluminum industry will lead to significant reductions in emissions of greenhouse gases compared to the traditional Hall-Hèroult process. This is a fact even though twice as much CO2 will be emitted in the carbothermic process based on the process equations. The main reasons for the total reduction are that the ACT-process eliminates PFC-emissions and significantly reduces the power consumption. Calculation of CO2-emissions from power generation is a complex task, but acceptable estimates can be found by means of simplified calculation principles. The results show that replacing the Hall-Hèroult process by ACT will reduce the total greenhouse gas emissions by at least about 30%.

4:15 PM
Performance of Aluminium Bronze as an Inert Anode: *Mark David Glucina*[1]; [1]University of Auckland, Cheml. & Matls. Engrg., 20 Symonds St., Auckland New Zealand

Aluminium bronze alloys have been investigated as a potential inert anode material for aluminium electrolysis. The goal for such a material is to produce an oxide layer at the electrode/anode interface, which will protect the metal from molten cryolite. A lab-scale electrolysis cell has been used for testing potential anodes. The effects of alloy composition, current density, and electrolysis time have been investigated. The scales formed on the anode, both before and during electrolysis, have been characterised through a combination of XRD, SEM, and optical microscopy techniques.

4:35 PM
On the Corrosion Behaviour of Ni-NiO-NiFe2O4 Cermets as Inert Anodes in Aluminum Electrolysis: Yanqing Lai[1]; *Jie Li*[1]; *Huanan Duan*[1]; Xiaogang Sun[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China

Ni-NiO-NiFe2O4 cermets with different NiO contents were prepared and the corrosion behaviour in Na3AlF6-Al2O3 melts was investigated in laboratory electrolysis tests. The steady-state concentrations of Ni, Fe in these samples were below the corresponding solubility in the similar melts, which implied that the corrosion mechanism in electrolysis might be different from that of unpolarized corrosion. The impurity ions transferred directionally and gathered near cathode, forming a concentration gradient. Such a gradient put the calculation of corrosion rate based on analysis of bath and aluminum into question. Post-electrolysis examination of the anodes showed that Ni metal corroded preferentially at the surface of the anodes, yet the remained ceramic layer prevented the penetration of bath and the loss of the metal phase in the underlying cermets.

4:55 PM
Fabrication of Al-Ni-Cu-O Cermets Inert Anodes and Electrolysis Testing: *Wang Zhao Wen*[1]; Luo Tao[1]; Gao Bing Liang[1]; Qiu Zhu Xian[1]; [1]Northeastern University, Coll. of Matls. & Metall., 3-11 Wenhua Rd., Heping Dist., MB 117, Shenyang, Liaoning 110004 China

An idea about the cermet materials is tested. Al-Ni-Cu-O was applied for the inert anode materials for aluminium electrolysis. The density of samples, fabricated by the hot pressing, is close to the theoretic density. At 900 centigrades the electric conductivity of the samples is >80scm-1. The anode (150×12 mm) was steadily run for 24h at a current of 150A. The deterioration of the anode was analysed. According to the XRD of the electrolysed anode, the NiO content is increasing, and the new phase (NiO) created in the anode, resulted in swelling and delamination. A theory is that a more uniform metal distribution from improving the hot pressing process, gives increased corrosion resistance of the anode.

5:15 PM
Preparation of the Cermet Inert Anode Based on Ferrous Nickel and Electrolysis Study: Luo Tao[1]; *Wang Zhao Wen*[1]; Gao Bing Liang[1]; Qiu Zhu Xian[1]; [1]Northeastern University, Coll. of Matls. & Metall., 3-11 Wenhua Rd., Heping Dist., MB 117, Shenyang, Liaoning 110004 China

Hot pressing-sintering was adopted to fabricate the inert cermets anodes based on the nickel ferrite in aluminum electrolysis. The electrical conductivity of the anodes, with relative density of more than 98%, increases with the increasing of temperature, ranging from $40\Omega^{-1} \cdot cm^{-1}$ to $60\Omega^{-1} \cdot$ cm-1 at 900°C. The electrolysis with the cermet anodes were run at the different anodic current density. The contaminations amount in the aluminium production is increasing with the increasing of anodic current density, and the contaminations (Fe, Ni and Cu) amount is <0.257wt% at 0.8A/cm^2. The main reason of anode corrosion is the ascending activity of Fe^{3+} ions and rich in the outer anode due to the increasing of anodic current density.

5:35 PM
300 A Bench-Scale Aluminum Electrolysis Cell with Fe-Ni-Al2O3 Composite Anode: *Zhong-ning Shi*[1]; Jun-li Xu[1]; Zhu-xian Qiu[1]; Zhao-wen Wang[1]; Bing-liang Gao[1]; [1]Northeastern University, Coll. of Matls. & Metall., PO Box 117, Shenyang 110004 China

A new Fe-Ni-Al2O3 metal matrix composite material was developed for aluminum electrolysis cells. A 300 A experiment for these anodes was operating with wettable cathodes (TiB2) at 960°C for 10 h. The electrolyte consisted of 51NaF-39AlF3-3CaF2-7Al2O3(wt%). The potential from the anode to the aluminum reference electrode was 2.25V. The purity of the produced aluminum was <99% with 75% current efficiency. After electrolysis, the SEM analysis indicated that there was a layer consisting of Al2O3 at the anode surface. To some extent, the layer can prevent the attacks from the melt and oxygen.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Monitor and Control of Nanostructure Synthesis

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Wednesday PM Room: 3018
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Jud Ready, Georgia Tech, GTRI-EOEML, Atlanta, GA 30332-0826 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany; Lourdes G. Salamanca-Riba, University of Maryland, Coll. Park, MD 20742-2115 USA

2:00 PM Opening Remarks

2:05 PM Invited
Optical and Photoelectron Spectroscopy of Carbon Nanotubes: *Tobias Hertel*[1]; [1]Vanderbilt University, Dept. of Physics & Astron., 1807 Sta. B, Nashville, TN 37235 USA

Carbon nanotubes (CNTs) hold promise for use in a variety of applications owing to a combination of extremely high aspect ratios with unique mechanical and electronic properties of graphene - the building material for CNTs. This presentation will focus on the optical and dynamical properties of CNTs. Specifically, the dynamics of optically excited single- and double-wall carbon nanotubes (SWNTs and DWNTs) will be discussed in some detail. They are investigated in quasi-crystalline form - as so called nanotube ropes - or as individual entities in aqueous solution using CW absorption-, femtosecond time-resolved photoemission- and time-resolved photoluminescence spectroscopy. We discuss the importance of carrier-carrier and carrier-phonon scattering as well as of radiative and nonradiative decay processes for the dynamics of optically excited CNTs.

2:35 PM
Synthesis Optimization and Characterization of Multi-Walled Carbon Nanotubes: *Christian Deck*[1]; Erik McKee[1]; Kenneth S. Vecchio[1]; [1]University of California, Dept. of MAE, Matls. Sci. &

Engrg. Grp., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

The unique properties of carbon nanotubes have suggested a myriad of applications in a variety of fields. However, consistent and optimal growth of high purity nanotubes has proven to be a challenge. We report on the synthesis optimization of multi-walled carbon nanotubes through the pyrolytic decomposition of benzene and a metal-organic catalyst precursor in a chemical vapor deposition furnace and the subsequent characterization thereof. Variables such as catalyst type, pressure, carrier gas flow rate, gas turbulence, carbon source feed rate, and temperature are optimized so as to produce well aligned multi-walled carbon nanotubes of lengths between a few microns to greater than one millimeter. We also discuss the characterization of our nanotube products through SEM, TEM, XRD, and EDS, focusing on the structure, composition, and purity of the results. Additionally, thermogravimetric analysis was performed to determine characteristic temperatures at which carbon nanotubes and byproducts oxidize in order to find appropriate ranges within which to purify carbon nanotubes. Results show the synthesis of high purity nanotubes of several millimeters in length with a thermal stability to near or above 500°C. As most synthesis byproducts burn off in the proximity of 300°C, a temperature range for nanotube purification is isolated.

3:05 PM
Synthesis and Characterisation of Carbon Nano-Glassy Films: *Marco Giuseppe Beghi*[1]; Antonio Miotello[2]; Paolo Mosaner[2]; Paolo Maria Ossi[1]; [1]Politecnico di Milano, Dip. Ingegneria Nucleare & Ctr. of Excellence Nano Engineered Matls. & Surfaces (NEMAS), Via Ponzio, 34-3, Milano 20133 Italy; [2]University of Trento, INFM-Dip. di Fisica, Povo, TN 38050 Italy

Carbon films were deposited at room temperature onto (100) Si substrates by Pulsed Laser Deposition (PLD) in the UV of highly oriented pyrolitic graphite. By changing deposition conditions (neon and argon atmospheres, at pressures from 0.6 Pa to 2 kPa; laser power density, from 8.5 to 19 MW mm-2) nanosized cluster-assembled films were obtained. SEM determined film morphology, evolves from dense columns to node-like, to an open dendritic structure. The size distribution and relative abundancies of carbon clusters were evaluated by AFM and correlated with film roughness. Visible Raman spectroscopy shows that all films are sp2 coordinated and structurally disordered and belong to the family of nano-glassy carbons. The calculated coherence length is correlated with the size of carbon clusters agglomerated on the surface of the growing film. The number of atoms per cluster, as calculated by a model of aggregation in the plume, depends sensitively on ambient gas pressure.

3:35 PM Break

4:00 PM
In-Situ Monitoring of the Nucleation and Growth Process of High Aspect Ratio Corundum Structures: *Joris Proost*[1]; Elzbieta Koza[1]; [1]University of Louvain, Div. of Matls. & Process Engrg. (IMAP), Place Sainte-Barbe 2, Louvain-la-Neuve B-1348 Belgium

One-dimensional nanostructures of alpha-Al2O3 were successfully synthesized by simple evaporation of the commercial high-purity oxide powders, followed by vapour condensation onto a sapphire substrate under controlled conditions of supersaturation without the presence of a catalyst. Synthesis was carried out in a specially designed reactor equipped with a laser setup that allows to monitor the nucleation and growth process in situ. Detailed in situ interferometric and high-resolution substrate curvature measurements revealed, for the first time, that the growth of these one-dimensional structures is driven by a mechanical stress, which develops in an initially continuous, 2-dimensional layer. This interaction between chemical activity and mechanical stress is shown to provide an important additional parameter to actively control the large-scale synthesis of high aspect ratio oxide nanostructures.

4:30 PM
Effect of Cationic Substituents on Controlling the Aspect Ratio of Goethite and the Magnetic Properties of G-Fe2O3 Nanoparticles Derived from Substituted Goethite: *Sudakar Chandran*[1]; [1]Royal Institute of Technology, Dept. of Matls. Sci., Tmfy-MSE, Brinellvagen 23, Rm. 224, Stockholm SE 100 44 Sweden

The effect of different substitutional impurities such as Al3+, Cr3+, Co2+ or Ni2+ on the particle morphology, shape control and phase stability of goethite and further the understanding of the effect of particle size, shape and compositional variability on magnetic properties of the nanoparticular g-Fe2O3 system are presented. g-Fe2O3 of desired shape and size are obtained by topotactic reaction from the precursor material goethite and magnetic properties are tuned by controlling these parameters. Acicular goethite particles are prepared using morphology controlling cationic additives.[1] The goethite structure is stable in spite of the iso- or aliovalent substitutions. The individual additives have divergent influence on the particle morphology; Al3+ and Cr3+ decreases the particle size to <50 nm and aspect ratio (AR) <2. Co2+ substitution produces slender particles with AR as high as 25. Whereas, Ni2+ does not have any influence on the particle morphology. The attributable factors in morphology control are the increased nucleation rate, restricted growth along needle axes, and the strain induced in the goethite lattice as a result of differences in ionic radii. Maghemite, g-Fe2O3, particles are obtained from goethite wherein the topotactic conversion renders the retention of the particle morphology of the precursor. Maghemite with substituted impurities showed substantial differences in magnetic properties. Saturation magnetization (ss) and coercivity (Hc) go down to very low values due to relaxation of spins on the surface atoms as revealed by Mössbauer spectroscopy. Decrease in coercivity is by way of the presence of diamagnetic ion (Al3+). Whereas, Co-substituted maghemite has enhanced Hc as a result of high magnetocrystalline anisotropy accompanied by the shape anisotropy. [1]C. Sudakar, G.N. Subbanna and T.R.N. Kutty, J.Mater. Chem., 12 (2002) 107-116.

Automotive Alloys 2005: Session III

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Wednesday PM Room: 2006
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Lexington, KY 40511 USA; Gyan Jha, ARCO Aluminum Inc., Louisville, KY 40223-4032 USA

2:00 PM
Microstructure and Mechanical Properties of Continuous Cast 5754 Alloy Sheet for Automotive Application: *Zhong Li*[1]; Steve Kirkland[2]; Shixi Ding[2]; Paul Platek[2]; [1]Commonwealth Aluminum, 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]Commonwealth Aluminum, 7319 Newport Rd., SE, Uhrichsville, OH 44683 USA

Commonwealth Aluminum has been developing continuous cast 5754 aluminum alloy sheet for automotive structural part applications over the last several years. The results showed that the Commonwealth Aluminum produced continuous cast (twin belt casting technology) 5754 aluminum alloy sheets can satisfy the formability during the forming process and the strength required by automotive industry. Automotive vehicle internal structural parts have been successfully stamped from CC material manufactured by Commonwealth Aluminum and passed the quality inspection. Commonwealth Aluminum, Newport Rolling Mill, has been approved as a raw material supplier for Ford Motor Company and General Motors Corporation.

2:25 PM
Ultrasonic Fatigue Behavior of E319 Aluminum Alloy at Elevated Temperature: *Xiaoxia Zhu*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Matls. Sci. & Engrg., 3062 HH Dow Bldg., 2300 Hayward Rd., Ann Arbor, MI 48105 USA; [2]Ford Motor Company, Ford Rsch. Lab., Dearborn, MI 48124-2053 USA

The effect of microstructure, temperature and load ratio on the fatigue behavior of an Al-Si-Cu cast alloy was investigated for lifetimes as long as 10^9 cycles using ultrasonic fatigue instrumentation operating at 20 kHz. Fatigue testing was conducted at 20, 150 and 250°C. There was a modest decrease in fatigue resistance from 20 to 150°C, while a significant decrease in fatigue resistance was observed from 150 to 250°C at low stress levels. Fractographic studies indicated that most fatigue cracks initiated from microshrinkage pores at the specimen surface, while a much smaller number initiated from interior pores, twin boundaries and oxides. Microstructure has been quantified and correlated with fatigue behavior. Fatigue lifetime was found to depend primarily on early crack growth behavior, rather than crack initiation. This knowledge has been used to extend a model for fatigue life prediction, based on small crack growth behavior. The effect of frequency and load ratio will also be discussed.

2:50 PM
Deformation Characteristics of AZ31 Magnesium Alloy Under Various Forming Temperatures and Strain Rates: Fadi K. Abu-Farha[1]; *Marwan K. Khraisheh*[1]; [1]University of Kentucky, Ctr. for Mfg., 210 CRMS Bldg., Lexington, KY 40506-0108 USA

The increasing demand for lower fuel consumption vehicles and the associated environmental issues induced the automotive industry to make the commitment to reduce fuel consumption levels, mainly by reducing vehicles' weight. As the lightest constructional metal on earth, Magnesium (and its alloys) has already started to occupy an increasingly big space in the different research fields as an important potential material for various automotive components. Many automotive components have been already produced from different magnesium alloys, but they are mainly cast components. Production of magnesium outer body components is still hindered by the metal's inferior ductility at room temperature; that's why magnesium alloys are usually formed at high temperatures. In this work, the deformation behaviour of Magnesium Alloy AZ31 is investigated under various forming temperatures and strain rates. Tests were conducted in the temperature range between 73 and 932°F and the strain rate range between 1x10-5 to 3x10-2 s-1. Results show the enhanced ductility and strain rate sensitivity of the alloy at higher temperatures. The alloy even exhibits a superplastic-like behaviour within a certain range of strain rates and temperatures. The results of these and other tests will be used to develop a constitutive model that predicts the deformation behaviour of the alloy.

3:15 PM

Quantitative Characterization of AM50 and AS21X Magnesium Alloys to Correlate the Variability in Fracture Properties with Microstructure: *Arun Sreeranganathan*[1]; Soon Gi Lee[1]; Gautam R. Patel[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30332 USA

Fracture related properties of high-pressure die cast AM50 and AS21X magnesium alloys are studied at six different temperatures ranging from room temperature to 394K. The properties are correlated with the microstructure through quantitative analysis of bulk and fracture surface microstructures. There is a significant variability in the tensile ductility at all temperatures for AM50 specimens and the variability decreases with increase in temperature. The porosity at the fracture surface is found to correlate well with the ductility of AM50 samples suggesting a preferential fracture path through the regions of highly localized clusters of large pores. No such correlation is observed for AS21X where the variability in properties is seen to increase with increase in temperature. Other factors such as the effect of intermetallic compounds like Mg2Si on the fracture properties of AS21X are investigated.

3:40 PM

On the Creep of Die-Cast Magnesium Alloys: *Bimal Kad*[1]; Qingyou Han[2]; Srinath Viswanathan[3]; [1]University of California, 409 Univ. Ctr., La Jolla, CA 92093 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Sandia National Laboratories, Albuquerque, NM 87185 USA

Die cast magnesium alloy design for elevated temperature creep is evaluated from the perspective of strengthening contributions of alloying additions and the microstructure constituents. A new approach[1] to alloy design incorporating Si, Ca, Ce, Zn ternary additions takes into account the inherently non-uniform die-cast microsturture. This leads to a non-uniform distribution of the solidus/homologous temperature in the α(Mg) phase and a non-uniform distribution of deformation stresses and strains in the specimen under creep conditions. The mechanical response of such inherently non-uniform microstructures is evaluated experimentally and via computational means. Both observations suggest that significant creep deformation occurs in the α(Mg) phase in and adjacent to the eutectic regions with the low solidus constituent while deformation in the primary α(Mg) dendrites is less pronounced. Microstructural design efforts that increase the homologous temperature, reduce eutectic volume or reinforce the eutectic α(Mg) phase hold significant promise towards increasing the creep resistance of magnesium alloys. This approach is then applied to predict and validate a ranking of the creep performance of current die-cast alloys under developement. [1]Qingyou Han, Bimal Kad and Srinath Viswanathan (2004) Philosophical Magazine, in press.

4:05 PM

The Role of Microstructural Variability on the Ultra-High Cycle Fatigue Behavior of SiCp-Reinforced Aluminum Composites: *James Huang*[1]; Jonathan E. Spowart[2]; J. Wayne Jones[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., HH Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA; [2]AF Research Laboratory, Matls. & Mfg. Direct., Bldg. 655, 2230 10th St., Ste. 1, Wright Patterson AFB, Dayton, OH 45433 USA

Compared to their unreinforced counterparts, discontinuously-reinforced metal matrix composites (DRC) generally display improvements in properties such as modulus, wear and creep resistance, and high-cycle fatigue. DRC can also be processed by fairly conventional means, making these attributes increasingly appealing in high-performance structural applications. It has therefore become more important to understand its ultra-high cycle fatigue behavior in order to maximize performance and minimize service costs. The role of microstructural variability on the fatigue behavior of 2124/SiC/15p-T4 MMC was investigated in this study. Ultrasonic fatigue methods were employed in order to specifically explore the ultra-high cycle fatigue regime (10^7 ¡ÜN_f¡Ü10^9 cycles). Although the spatial distribution of SiC particles is known to play a key role in the fatigue behavior of DRC, our data indicate that at these long lifetimes, fatigue life is determined by critical inclusions and not the heterogeneity of the particle distribution. Correlation of fatigue data with inclusion probability studies incorporating serial sectioning and 3-d microstructural characterization is used to examine the role of microstructure on fatigue life and the variability of fatigue life.

4:30 PM

The Effect of Ti Content on Microstructure, Castability and Properties of Foundry Alloy A356.2: *Malcolm J. Couper*[1]; Catherine L. Smith[1]; Mark Easton[2]; Joseph Barresi[1]; [1]Comalco Aluminium Ltd., Comalco Rsch. & Techl. Support, 15 Edgars Rd., Thomastown, VIC 3074 Australia; [2]Monash University, CRC for Cast Metals Mfg., Sch. of Physics & Matls. Engrg., VIC 3800 Australia

The effect of titanium additions on the casting and performance of an Al7Si0.35Mg alloy has been studied. The study investigated various combinations of titanium and grain refiner TiB2 additions and considered grain size, intermetallic phases, age hardening response, tensile properties and fluidity and porosity. Low titanium consisted of 0.01wt% whilst high titanium refers to typically used levels of 0.12-0.14wt%. The results show that high levels of titanium are not required to ensure good performance. In particular, the high titanium levels result in coarse Al-Ti-Si particles, linked to a reduction in tensile elongation in mechanical testing. The fluidity of the alloy with high titanium was reduced. Furthermore, an interaction with hydrogen level in the melt was observed such that this reduction in fluidity was greater in non-degassed melts.

4:55 PM

Development of a Low Carbon Al-Killed/Ti Stabilized Steel for Automotive Applications: *Julio Alberto Juarez-Islas*[1]; Rafael Mendoza[2]; [1]Instituto de Investigaciones en Materiales_UNAM, Materiales Metalicos y Ceramicos, Circuito Exterior S/N, Ciudad Universitaria, Mexico, D.F 04510 Mexico; [2]Ispat Mexicana S.A. de C.V., Aseguramiento de la Calidad, Francisco J Mujica 1B, Lazaro Cardenas, Michoacan 60950 Mexico

An Al-Killed/Ti-Stabilized low carbon steel was produced using the electric arc furnace, vacuum degassing, ladle treatment and continuous casting route. The resulting slabs were then hot rolled at 1100ºC, coiled at 600ºC, cold rolled and annealed at 850ºC. After evaluation of the microstructure, texture and mechanical properties, the fully recrystallized coils fullfilled the target properties stablished by the automotive industry.

5:20 PM

Microstructural Influence on High-Cycle Fatigue Limit of High Carbon Pearlitic Steel Filaments Used for Tires: *Yo Sep Yang*[1]; Seong Yong Park[1]; Chan Gyung Park[1]; Seung Ho Lim[2]; Deok Young Ban[2]; [1]Pohang University of Science and Technology(POSTECH), Matls. Sci. & Engrg., San 31, Hyoja-Dong, Nam-Gu, Pohang, Kyungbuk 790-784 Korea; [2]Trefilarbed Korea, 134, Yusan-dong, Yangsan, Kyungnam 626-230 Korea

Influences of microstructure on high cycle fatigue limit of high carbon steel filaments have been investigated. Fatigue tests were carried out by using Hunter-type tester at a frequency of 60 Hz at bending stress of 900 to 1500 MPa. It was found that fatigue limit and tensile strength were improved with increasing carbon content, which was mainly attributed to decreased lamellar spacing and cementite thickness. However, the fatigue ratio, which is defined as the ratio of the fatigue limit to the tensile strength, was reduced in a higher carbon range of 0.8 to 0.9 wt.%, while the fatigue ratio was nearly constant in a lower carbon range of 0.7 to 0.8 wt.%. Mechanical properties of the filaments have been discussed in terms of the microstructural parameter change of lamellar spacing and cementite thickness. In addition, the variation of cementite morphology on the fatigue crack propagation will be discussed.

5:40 PM

Influence of Additional Mn and Cu Elements on Mechanical Properties of Super High Strength Aluminum Alloy: *Kozo Osamura*[1]; Hiroki Adachi[1]; Tmohiro Sorano[1]; Hidenori Nako[1]; Akihei Tanaka[2]; Ken Kikuchi[2]; Shigeru Okaniwa[3]; [1]Kyoto University, Dept.

of Matls. Sci. & Engrg., Sakyo-ku, Kyoto 6068501 Japan; [2]Toyo Aluminum K.K., Hino 529016 Shiga; [3]Nippon Light Metal K.K., Kanbara 4213203 Shizuoka

One of key technologies for automotive industry is the development of light material with high specific strength. The super high strength Al-Zn-Mg-Cu-Mn-Ag based alloy (Mesoalite20â) includes two type of precipitates of Q and Al6Mn phases. The Q precipitates have a fiber-like shape, but Al6Mn has round and irregular shape. By hot extrusion at 773K, their precipitates flowed along extrusion direction and only Q precipitates aligned well. When increasing Mn content with constant of 1.5mass% Cu, the amount of Al6Mn increased remarkably. The compressive strength decreased with increasing Mn content. When increasing Cu content from 0.5 to 2.5 mass% with constant of 4 mass%Mn, the amount of Q phase increased and their mechanical property improved. Therefore the present study has proposed a new alloy with higher Cu content, which gives higher strength than that of commercial Mesoalite 20, which has tensile strength of 910 MPa.

Beta Titanium Alloys of the 00's: Mechanical Response

Sponsored by: Structural Materials Division, SMD-Titanium Committee

Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Wednesday PM
February 16, 2005

Room: Salon 10/11
Location: San Francisco Marriott

Session Chairs: Robert F. Denkenberger, Ladish Co. Inc., Cudahy, WI 53110-8902 USA; Brian Marquardt, Zimmer, Metals Rsch., Warsaw, IN 46581-0708 USA

WEDNESDAY PM

2:00 PM Invited

Fatigue Performance of Metastable Beta Titanium Alloys: Effects of Microstructure and Surface Finish: *L. Wagner*[1]; A. Boettcher[1]; M. Kocan[1]; H. J. Rack[2]; [1]TU Clausthal, Inst. of Matls. Sci. & Engrg. Germany; [2]Clemson University, Sch. of Matls. Sci. & Engrg., Clemson, SC 29634 USA

Metastable beta titanium alloys may be subjected to a wide variety of thermomechical treatments. For example these materials can be rolled or swaging and statically recrystallization to establish a wide variations in grain size and/or primary alpha size, morphology and volume fraction. Subsequent aging can also result in variations in tensile properties. Finally surface treatment by shot peening or roller burnishing can also be used to alter surface properties. This presentation will review the combined effects of these various treatments on the high cycle fatigue performance of metastable beta alloys either in fully reversed loading(R=-1) or tension-tension loading(R=0.1)of three commercially available metastable beta alloys, TIMET LCB, Beta 21S and Beta C. It is indended to provide a generalized framework of the effects of microstructure, surface roughness, dislocation density and residual stress on the high cycle fatigue crack initiation and propagation in the class of increasingly important titanium alloys.

2:40 PM

High Cycle Fatigue Crack Initiation and Growth in TIMETAL LCB: *B. Yazgan*[1]; Y. Kosaka[2]; H. J. Rack[1]; [1]Clemson University, Sch. of Matls. Sci. & Engrg., Clemson, SC 29634-0971 USA; [2]TIMET, Henderson, NV 89009 USA

This investigation has examined the influence of microstructure variations on the high cycle fatigue behavior of aged TIMETAL LCB, the fatigue performance being evaluated under tension-tension loading conditions at R=0.1 in laboratory air and 25 Hz. Fractographic analysis indicated that fatigue initiation, independent of processing history, involved sub-surface crack formation. Serial sections studies also indicated that crack initiation occurred at the interface between grain boundary alpha and the aged beta matrix. Further BSEI examination of the aged microstructures indicate that the differences in high cycle fatigue behavior can be understood by considering the effect of processing history on the connectivity of grain boundary alpha, decreased connectivity being associated with enhanced fatigue performance.

3:05 PM

Overview of High-Cycle Fatigue of Beta Titanium Alloys: Role of Microstructure on Crack Initiation, Growth and Fatigue Life: *K. S. Ravi Chandran*[1]; Sushant K. Jha[1]; Shankar Srinivasan[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City 84112 USA

Results of extensive research on the effect of microstructure on the fatigue behavior of beta-titanium alloys, with an emphasis on the metastable Ti-10V-2Fe-3Al alloy, is presented. The study considered several variations in primary-alpha volume fraction and aspect ratio as well as the varied secondary-alpha distributions resulting from different aging treatments. Previous work from literature on several beta-Ti alloys is also included in the review. It is concluded that the low stress and high cycle fatigue behavior is determined solely by the aging condition or the nature of secondary alpha distribution in beta matrix. The results from the Ti-10V-2Fe-3Al alloy indicate that a fine distribution of secondary-alpha is necessary to achieve maximum resistance to high cycle fatigue. Reanalysis of the literature data on this basis confirmed this finding. Fatigue crack growth in these alloys is largely insensitive to microstructure. Fatigue life is mostly determined by fatigue crack initiation. In most of the microstructures, fatigue cracks initiated in subsurface and surface regions at low and high stress regimes, respectively. The implications of these results on microstructure design for better fatigue resistance and the analysis of mechanisms of fatigue in beta-Ti alloys are discussed.

3:30 PM

Designing a Beta Titanium Alloy for Optimum Fatigue Performance: *Sushant K. Jha*[1]; K. S. Ravi Chandran[2]; [1]Universal Technology Corp., 1270 N. Fairfield Rd., Dayton, OH 45459 USA; [2]University of Utah, Metallurgl. Engrg., 135 S, 1460 E, Rm. 412, Salt Lake City, UT 84112 USA

A systematic study was conducted to establish the microstructure-fatigue correlation in the metastable beta titanium alloy, Ti-10V-2Fe-3Al (Ti-10-2-3). The aging condition significantly affected the damage tolerance of long cracks as well as the fatigue limit. The omega-aged condition showed an increased damage tolerance but lower fatigue lives and the fatigue limit when compared to the alpha-aged microstructure. In the alpha-aged condition, variation at the length scales of prior beta grain and the primary alpha particle had limited effect on the fatigue limit or the long crack growth rates. However, variation at the secondary alpha size scale caused a significant effect on the fatigue life behavior with almost no loss of "long crack" damage tolerance.

3:55 PM Break

4:10 PM

Dynamic Response of Beta-Ti Alloys: *Kenneth S. Vecchio*[1]; Uday Deshmukh[1]; Raghavendra R. Adharapurapu[1]; Fengchun Jiang[1]; Adam Crocker[1]; [1]University of California, Dept. of MAE, Matls. Sci. Grp., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

Beta-Ti alloys offer attractive alternatives to alpha and alpha+beta Ti alloys due to their increased strength-to-weight ratio, good formability, and excellent fatigue and fracture resistance. With the growing interest in the use of Ti alloys for various defense platforms, the dynamic mechanical response of beta-Ti alloys must be investigated to determine their applicability under impact loading situations. Composition, thermal-mechanical processing, and prestrain level can alter the constitutive response of beta-Ti alloys. Three metastable-beta alloys: Ti-16V-6Cr-4Al, Ti-15V-3Cr-3Sn-3Al, and Ti-10V-2Fe-3Al, in both the annealed+heat-treated and the cold-rolled+heat-treated conditions were investigated to determine the tensile response of these alloys over a broad range of test temperatures and strain rates from 10^{-3}/s to 10^{3}/s. The effect of orientation with respect to the sheet rolling direction was documented. The effect of annealing versus cold rolling on subsequent aging response is discussed, and the relationship between microstructure and mechanical response is established for the various alloy conditions. In general, the flow curves show strong temperature and strain rate dependencies, with relatively little work hardening, consistent with the behavior of many bcc metals. The applicability of existing phenomenological bcc models, such as: Johnson-Cook and Zerilli-Armstrong to these metastable beta-Ti alloys is investigated.

4:35 PM

Quick Reference Guide for Beta Titanium Alloys: *Stacey Nyakana*[1]; John C. Fanning[1]; Rodney R. Boyer[2]; [1]TIMET, PO Box 2128, Henderson, NV 89009 USA; [2]The Boeing Co., PO Box 3707, Seattle, WA 98124 USA

Beta alloys provide useful combinations of physical and mechanical properties as well as a wide range of processing and corrosion properties. Throughout the past several decades, many beta alloys have been researched, yet only a handful of these have currently enjoy

commercial production. While the primary applications continue to be in the aerospace market, beta alloys have demonstrated usefulness in other arenas. This section provides a quick reference guide for a large variety of common and not-so-common beta titanium alloys. Information regarding chemical composition, mechanical properties, physical properties, heat treatment, fabrication, applications, and production status is included.

5:00 PM
Panel Discussion: Beta Titanium Alloys: Future Needs and Opportunities

Biological Materials Science and Engineering: Biological Materials Characterization and Biomimetics II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM Room: 3009
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Larry Hench, Imperial College London, Tissue Engrg. & Regenerative Medicine Ctr., Ashford, Kent TN25 4AH UK; Andrea M. Hodge, Lawerence Livermore National Laboratory, Matls. Dept., Livermore, CA 94550-9234 USA

2:00 PM Invited
Rapid In Vitro Testing of Cell-Biomaterial Interactions With Bio-Photonics: *Larry Hench*[1]; [1]Imperial College London, Tissue Engrg. & Regenerative Medicine Ctr., Wye Campus, Ashford, Kent TN25 5AH UK

Obtaining quantitative biological data on the effect of materials on cells and cells on materials is essential in developing new biomaterials and quality assurance testing of commercial biomaterials. Standard biological assays are time consuming and expensive. There is considerable societal pressure to minimize animal testing. However, a limitation of typical in vitro cell culture based tests is monitoring and maintaining a mature cell phenotype during the experiment. A newly developed integrated living cell and bio-photonics system provides rapid in vitro testing of biomaterials using human primary cell cultures with in situ spectroscopic monitoring of the cell phenotype. This bio-photonic based system also can be used for evaluating potentially toxic chemicals, environmental or chemical and biological warfare agents. Advantages of the system include: rapid throughput, non-invasive, real-time monitoring of individual cells, clusters of cells or 3-D assemblages of cells (organoids). Quantitative Raman spectroscopic data are used to follow cell viability, onset of cell death, mechanism of cell death, cell division, cellular differentiation and de-differentiation and cell phenotype when exposed to control environments, biomaterials or chemical, biological, pharmaceutical or environmental elements or compounds. Applications to 10 different types of living cells are presented, including differentiation of embryonic stem cells. Results of using the system on testing the effects of bioterrorism agents, ricin and sulfur mustard, on human lung-derived cells are reviewed.

2:30 PM Invited
Bioactive Ceramic-Tetrahedral Amorphous Carbon Nanocomposites: *Roger Jagdish Narayan*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr. NW, Rm. 361, Atlanta, GA 30332-0245 USA

We have developed a hydroxyapatite-tetrahedral amorphous carbon nanolayered composite using pulsed laser deposition. The hydroxyapatite surface layer possesses bioactive properties that mimic the properties of natural bone, and the tetrahedral amorphous carbon interlayer possesses biocompatibility, wear resistance, and corrosion resistance properties that hermetically seal the bulk biomaterial. Unfortunately, tetrahedral amorphous carbon thin films exhibit poor adhesion due to internal compressive stress and poor interfacial bonding with the substrate. To solve this problem, we have created multilayered composites using a pulsed laser deposition. These films exhibit significantly higher hardness values than the underlying Ti-Al-V alloy. We envisage numerous applications for these hydroxyapatite/tetrahedral amorphous carbon multilayer coatings, including use in orthopedic and dental implants.

3:00 PM
Net Shape Processing of Functionally Graded Materials by Field Activated Sintering: Vladimir Y. Kodash[1]; *Joanna R. Groza*[1]; Jing Zhang[2]; Antonios Zavaliangos[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Drexel University, Dept. of Matls. Sci. & Engrg., Philadelphia, PA USA

Field Activated Sintering Technique (FAST) is widely used for sintering ceramic and metal products. Processing of the powder is usually conducted in graphite dies and punches and results in simple flat shapes, mainly discs. However, the manufacture of curved surfaces presents some difficulties. A new technique has been developed to produce a non-flat shape made of several layers (functionally graded material, FGM). The FGM was processed by powder cold spraying of several ceramic-metal layers starting with pure materials at each surface and increments of 25% compositions. The final shape was pre-pressed by cold isostatic pressing and then sintered by FAST sintering. The simulation of cold compaction and FAST sintering of non-flat surfaces has been conducted on metal powders. It was found that the temperature homogeneity can be improved by applying thermal insulating coating at the interface between the specimen and the die. The FGM materials may be used for medical applications.

3:20 PM
In Situ Characterization of Ti-Peroxy Gel During Formation on Titanium Surfaces in Hydrogen Peroxide Containing Solutions: *Julie J. Muyco*[1]; Jeremy J. Gray[1]; Timothy V. Ratto[1]; Christine A. Orme[1]; Joanna McKittrick[2]; John Frangos[3]; [1]Lawrence Livermore National Laboratory, Chmst. & Matl. Sci., 7000 East Ave., L-350, Livermore, CA 94550 USA; [2]University of California, Mechl. & Aeros. Engrg., Matl. Sci. & Engrg. Grad. Prog., 9500 Gilman Dr., La Jolla, CA 92093 USA; [3]La Jolla Bioengineering Institute, 505 Coast Blvd. S., Ste. #405, La Jolla, CA 92037 USA

Three possible functions of Ti-peroxy gel are: reduction of the inflammatory response through the reduction of hydrogen peroxide and other reactive oxygen species; creation of a favorable surface for calcium phosphate nucleation; and as a transitional layer between the soft tissue and the stiff titanium. Traditional surface characterization techniques operate in high vacuum environments that alter the actual sample-solution interface. Our studies used techniques that allowed samples to remain in solution and be observed over time. Atomic force microscopy (AFM) force-distance curves, electrochemical impedance spectroscopy (EIS), Raman spectroscopy, and ellipsometry were each used in situ to define kinetic and mechanical properties of Ti-peroxy gel as it formed over time on titanium during exposure to hydrogen peroxide. Our studies enabled us to monitor real-time changes in the native oxide layer on titanium in hydrogen peroxide containing solution, including the formation of a Ti-peroxy gel layer above the native oxide. Peaks attributed to Ti-peroxy gel were seen to emerge over the course of several hours using in situ Raman spectroscopy. Force-distance curves suggest a layer that thickens with time on the titanium sample surface. EIS data showed that changes in the surface layers could be monitored in solution over time. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

3:40 PM
Effect of Post Vacuum Heating on the Microstructural Feature and Bonding Strength of Plasma-Sprayed Hydroxyapatite Coatings: *Chung Wei Yang*[1]; Tzer Min Lee[2]; Truan Sheng Lui[1]; Edward Cheng[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., No. 1, Univ., Rd., Tainan 701 Taiwan; [2]National Cheng Kung University, Inst. of Oral Medicine, Coll. of Medicine, No. 1, Univ., Rd., Tainan 701 Taiwan

Plasma-sprayed HA coatings (HACs) on Ti-6Al-4V substrate with post vacuum heating treatment were employed to improve the properties of HACs. The heating temperatures are 400ºC, 500ºC, 600ºC, 700ºC and 800ºC. According to the experimental results, post vacuum heating treatment could improve the average bonding strength and the minimum life of HACs by using Weibull's reliability analysis. The HACs with 600ºC vacuum heating shows the highest strength value of all. On the other hand, the index of crystallinity (IOC) of heat-treated HACs increases and impurity phase content decreases with increasing heating temperature. The crystallization during vacuum heating also leads the variation in thermal expansion coefficient and the volume

contraction of heat-treated HACs, especially higher than that of 600ºC vacuum heating. Therefore, the volume contraction induces apparent and concentric cracks and reduces the bonding strength of heat-treated HACs.

4:00 PM Break

4:15 PM
Mechanical and Radiographic Properties of a Shape Memory Polymer Composite for Intracranial Aneurysm Coils: *Janet M. Hampikian*[1]; Brian C. Heaton[2]; Frank C. Tong[3]; Zhuqing Zhang[2]; C. P. Wong[2]; [1]Boise State University, Matls. Sci. & Engrg., 1910 Univ. Dr., Boise, ID 83725-2075 USA; [2]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA; [3]Emory University Hospital, Dept. of Radiology, 1364 Clifton Rd. NE, Ste. A121, Atlanta, GA 30322 USA

An intracranial aneurysm can be a serious condition which may go undetected until the aneurysm ruptures causing hemorrhaging within the subarachnoid space surrounding the brain. An increasingly common treatment method is by embolization using platinum coils. However, in about 15% of the cases treated by platinum coils, the aneurysm eventually re-opens as a result of compaction of the platinum coil ball, incomplete occlusion/thrombosis of the aneurysm, or the inability of tissue to grow on platinum. One solution to this is to develop bio-responsive materials to use as coil implants. In this research, a thermoplastic shape memory polymer (SMP), CalomerTM, produced by The Polymer Technology Group, Inc., was investigated as a potential candidate for aneurysm coils. Small diameter fibers produced from SMPs can be fed through a micro-catheter to adopt a preprogrammed three-dimensional configuration when heated to body temperature. The SMP was tested to determine modulus behavior and shape recovery force. In addition, the polymer was made radio-opaque by adding tantalum filler at 3% volume fraction. Thermo-mechanical testing shows that the material exhibits a shape recovery force near 37° C. Coils produced from the material deployed inside a simulated aneurysm model demonstrate that typical hemodynamic forces do not hinder the shape recovery process. Fluoroscopic imaging of the composite coils in a clinical setting verifies the radio-opacity of the composite material.

4:35 PM Cancelled
Rate- and Cycle Dependence of Deformation of Small-Scaled Materials

4:55 PM
The Recrystallization and Thermal Oxidation Behavior of CP-Titanium: *Fatih Mehmet Güçlü*[1]; *Hüseyin Çimenoðlu*[1]; *Eyüp Sabri Kayalý*[1]; [1]Istanbul Technical University, Cheml. & Metall. Faculty, Metall. & Matls. Engrg. Div.- Maslak, Ýstanbul 34469 Turkey

Titanium and its alloys are used in biomedical industry due to their low weight, excellent corrosion resistance and biocompability. However, wear of these alloys causes adverse tissue reactions in the human body and needs to be replaced short time after implantation. Recently some surface modification techniques are utilized to improve the wear properties of titanium. Thermal oxidation is one these processes which is a low cost and an effective way to form a hard surface coating with an oxygen diffusion zone beneath it. The studies of the thermal oxidation titanium revealed that thermally oxidized samples showed 25 times resistance to wear than non-oxidized samples. Thermal oxidation, which is essentially carried out temperatures about 600°C, may associate with recrystallization of the bulk structure. In this study the recrystallization behavior of cold worked CP titanium are investigated and the relation between thermal oxidation and cold working of the CP titanium is investigated.

5:15 PM
Mechanical Characterization of Coronary Stents Using Nanoindentation and Nanoscratch Testing Techniques: *David J. Vodnick*[1]; Richard J. Nay[1]; Dehua Yang[1]; Thomas J. Wyrobek[1]; [1]Hysitron Inc., Nanomech. Rsch. Lab., 10025 Valley View Rd., Minneapolis, MN 55344 USA

Many generations of coronary stents have been developed, with each succeeding design being more flexible and easier to deliver to the occluded artery. The newest generation contains a drug-eluting coating designed to interfere with restenosis, thrombosis, and scarring. In addition to other biomaterial properties, the mechanical properties of stent material and coatings are of vital importance to product performance and reliability. However, the stent shape, size, and coating thickness have exposed great challenges to existing mechanical testing instruments. Therefore, the nanomechanical properties of several commercially available coronary stents were investigated using nanoindentation and nanoscratch testing techniques for the first time. Hardness and reduced modulus values were utilized to characterize the mechanical properties of various stents and their coatings. Scratching techniques were developed to quantify the interfacial adhesion of thin ceramic and polymeric coated stents. This study demonstrates the capability of nanoindentation and nanoscratch to quantify the mechanical properties and interfacial adhesion strength of coronary stenting systems.

5:35 PM
Effect of Firing Time on the Bonding of Porcelain Fused to Titanium With and Without a Plasma-Sprayed Zirconia Bond Coat: *Tsung Nan Lo*[1]; Truan Sheng Lui[1]; Edward Chang[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., No. 1, Ta Hsueh Rd., Tainan 701 Taiwan

Porcelain and titanium are applied in fixed prosthodontics by porcelain-fused-to-metal (PFM) recently. During firing of porcelain fused to titanium,it was observed that the oxidation of titanium becomes severe under high temperature and longer duration time. The results show that the bonding strength of porcelain fused to titanium decrease from 23.0 MPa to 18.4 MPa with increasing firing time. It is speculated that the titanium might be deteriorated by oxygen dissolution during oxidation process and enable the fracture to occur slightly into the upmost titanium near the porcelain/titanium interface. To alleviate the deterioration of titanium, a plasma-sprayed zirconia layer was introduced to serve as an oxygen diffusion barrier on the titanium prior to fusing of porcelain. Experimental results reveal that the bonding strength of porcelain/zirconia/titanium composite specimen increases (35.8 MPa) and there is no obvious degradation of bonding strength (34.1 MPa) after longer firing time.

Bulk Metallic Glasses: Mechanical Behavior

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Wednesday PM Room: 3006
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Katharine M. Flores, Ohio State University, Matls. Sci. & Engrg., Columbus, OH 43210 USA; John J. Lewandowski, Case Western Reserve University, Matls. Sci. & Engrg., Cleveland, OH 44106 USA

2:00 PM
Ductile vs Brittle Behavior of Metallic Glasses: *J. J. Lewandowski*[1]; W. H. Wang[2]; A. L. Greer[3]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106-7204 USA; [2]Chinese Academy of Sciences, Inst. of Physics, PO Box 603(42-1), Beijing 100080 China; [3]University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK

Notch toughness experiments have been conducted on a variety of bulk metallic glasses in both the fully amorphous condition as well as after annealing. The magnitude of the notch toughness is very dependent on the extent of shear deformation emanating from the notch. The presentation will summarize recent observations of notch toughness in metallic glasses as well as a review of the literature where similar experiments have been conducted. A number of the bulk metallic glasses exhibit exceptionally high values for notch toughness (e.g. > 60 MPa-m1/2) accompanied by extensive shear banding and fracture surface roughness. However, other bulk metallic glasses exhibit much lower notch toughness (e.g. < 10 MPa-m1/2), a lack of extensive shear banding, and fracture surfaces which are very brittle in appearance. The effects of annealing experiments on the change in notch toughness will also be presented, as it is possible to embrittle some, but not all, of the metallic glasses by annealing at temperatures near and above the glass transition temperature. The results will be rationalized by examining the changes to various elastic constants that accompany annealing within one metallic glass system. This approach will also be used to rationalize the difference in notch toughness between the metallic glasses exhibiting high values for notch toughness from those that are brittle. The implications of these correlations will be discussed.

2:20 PM
Structural Changes Associated with Plastic Deformation in Bulk Metallic Glasses: Matthew Lambert[1]; *Katharine M. Flores*[1]; [1]Ohio State University, Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210-1178 USA

It is well known that at low temperatures and high stresses, plastic deformation in metallic glasses is highly localized in shear bands, while at temperatures near the glass transition, deformation becomes homogeneous. Free volume theory indicates that the average free volume increases during flow, and prior studies of free volume distribution suggest that the sizes of free volume sites range from inherent interstitial-like defects to larger defects capable of flow. However, the precise flow mechanism and the evolution of the free volume distribution during flow remain unclear. In this study, the flow behavior of a Zr-based bulk metallic glass was investigated over a range of temperatures and strain rates. The average free volume before and after deformation was quantified using DSC. More detailed information about the evolution of the free volume distribution was obtained using positron annihilation spectroscopy, where an increase in the concentration of flow defects was observed following deformation.

2:40 PM
Cast Bulk Metallic Glass Alloys: Prospects as Wear Materials: *Jeffrey A. Hawk*[1]; Omer N. Dogan[1]; Gary J. Shiflet[2]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., Sch. of Engrg. & Applied Sci., 116 Engr.'s Way, Charlottesville, VA 22904-4745 USA

Bulk metallic glasses are single phase materials with unusual physical and mechanical properties. One intriguing area of possible use is as a wear material. Usually, pure metals and single phase dilute alloys do not perform well in tribological conditions. When the metal or alloy is lightweight, it is usually soft leading to galling in sliding situations. For the harder metals and alloys, their density is usually high, so there is an energy penalty when using these materials in wear situations. However, bulk metallic glasses at the same density are usually harder than corresponding metals and dilute single phase alloys, and so could offer better wear resistance. This work will discuss preliminary wear results for metallic glasses with densities in the range of 4.5 to 7.9 g/cc. The wear behavior of these materials will be compared to similar metals and alloys.

3:00 PM
Effects of Changes in Notch Radius on Deformation and Fracture of a Bulk Metallic Glass: *T. Jacobs*[1]; J. J. Lewandowski[2]; A. L. Greer[1]; S. Tin[1]; [1]University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK; [2]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106-7204 USA

Notched bend bars have been tested with different root radii in order to determine the effects of changes in stress triaxiality on the deformation and fracture of a bulk metallic glass. The evolution of shear bands at the notch surfaces was monitored via sequential loading experiments, while fracture surfaces were analyzed via scanning electron microscopy. FEM analyses were conducted on the bend bars using DEFORM in order to determine the evolution of stress and strain with increased loading, and the results were compared to existing stress analyses of similar specimens. Possible changes in temperature accompanying the deformation at the notches was initially modeled using DEFORM. The effects of changes in notch radius on the extent and magnitude of shear banding and likely locations of fracture initiation will be covered, while estimates of temperature rise predicted by DEFORM will also be presented.

3:20 PM
Effects of Changes in Specimen Geometry and Loading Rate on a Bulk Metallic Glass: *G. Sunny*[1]; A. S. Nouri[2]; J. J. Lewandowski[2]; V. Prakash[1]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Engrg., Cleveland, OH 44106-7204 USA; [2]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106-7204 USA

The effects of changes in specimen geometry and loading rate have been determined for a Zr-based bulk metallic glass. Right circular cylindrical specimens with length to diameter ratios varying from 0.5 to 2.0 were tested under various strain rates ranging from quasi-static to in excess of 1000/sec. Quasi-static tests were conducted using servo-hydraulic testing machines, while the high strain rate tests utilized both split Hopkinson pressure bar (SHPB) and gas guns. The effects of changes in specimen geometry and loading rate on the deformation and fracture behavior will be presented.

3:40 PM Break

4:00 PM
Effects of Superimposed Pressure on Flow and Fracture of BMGs and Devitrified Amorphous Aluminum Alloys: *P. Wesseling*[1]; L. O. Vatamanu[1]; J. J. Lewandowski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA

Compression tests have been performed with high alignment fixtures on 4 different bulk metallic glass specimens at atmospheric pressure as well as with levels of superimposed hydrostatic pressure up to 700 MPa. The results show essentially no difference in applied flow stress or fracture stress over the range of pressures tested. However, fracture did not occur on the plane of maximum shear, suggesting a normal stress effect on the flow and fracture behavior of the bulk metallic glass specimens. The data is compared to various flow and fracture theories. A Mohr-Coulomb flow theory appears to best describe the data. In contrast, a BMG composite and a devitrified aluminum alloy were tested with different pressures and temperatures. These composite materials fail via ductile fracture under tensile loading at ambient conditions. Initial investigations reveal large increases in ductility due to both increases in pressure and temperature.

4:20 PM
As-Cast Bulk Metallic Glasses Containing Nanocrystalline Particles: *Cang Fan*[1]; Hahn Choo[1]; Peter K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Engrg. Dept., 434 Dougherty Engrg. Bldg., Knoxville, TN 37996 USA

Bulk metallic glasses (BMG) exhibit heterogeneous deformation under loading at room temperature due to the formation of highly localized shear bands, which leads to a catastrophic failure. Lately, in an attempt to improve ductility, various BMG composites (BMGC) containing crystalline phases have been developed. In this talk, we will present recent advances in the processing technique, and in the understanding of the relationship between the microstructure and mechanical behavior of a Zr-based BMGC. By adding Nb to Zr-Ni-Cu-Al, nanocrystalline-particle-containing BMGCs were synthesized in an as-cast condition without subsequent heat treatments. The HRTEM results show that the addition of 2 at.% Nb introduces nanocrystals with an average size of 3 nm. The x-ray diffraction and DSC results also indicate that the presence of the as-cast nanocrystalline particles strongly influence the crystallization process. The heat flow of the nano BMGCS before reaching supercooled liquid region is different from monolithic BMG. Furthermore, compressive ductility of the Nb-containing BMGC at room temperature is significantly improved (about 3.5%) compared to the monolithic Zr55Ni5Cu30Al10 BMG (about 0.1~0.3%), together with a small increase in the yield strength from 1750 to 1800 MPa.

4:40 PM
High Temperature Hardness Indentation Studies on Metallic Glasses: *P. Wesseling*[1]; J. J. Lewandowski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA

High temperature microhardness indentations were conducted on a variety of metallic glasses, e.g. Al-, Fe-, Cu-, Hf-, Mg-, Ce- and Zr-based metallic glass, using a Nikon QM high temperature hardness indenter. Microhardness indentations were conducted while increasing the temperature up to the glass transition temperature of the material and subsequently during cooling to room temperature. Significant softening is observed as the temperature approaches the glass transition temperature. The softening behavior is further investigated by determining the activation energies for high-temperature flow, which can be derived from the slopes of the hardness versus temperature curves in the high temperature regime. After cooling to room temperature the microhardness of the materials has increased or was unchanged. AFM and SEM studies have been conducted to examine the surface features around the indentations conducted close to or at the glass transition temperature. Comparisons will be made amongst the various systems investigated.

Carbon Technology: Cathode Materials and Corrosion I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland

Wednesday PM Room: 2007
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Harald A. Oye, University of Science and Technology, Dept. of Matls. Tech., Trondheim N-7491 Norway

2:00 PM
Revisiting Sodium and Bath Penetration in the Carbon Lining of Aluminium Electrolysis Cell - Part I: *Pierre-Yves Brisson*[1]; Gervais Soucy[1]; Mario Fafard[2]; Guillaume Servant[3]; Hans Darmstadt[3]; Jean Camiré[3]; [1]Université de Sherbrooke, Génie chimique, 2500 boul. de l'Université, Sherbrooke, Québec J1K 2R1 Canada; [2]Université Laval, Génie Civil, Pavillon Adiren-Pouliot, Québec, Québec G1K 7P4 Canada; [3]Alcan Métal Primaire Inc., Arvida R&D Ctr., 1955 Blvd. Mellon, CP 1250, Jonquière, Québec G7S 4K8 Canada

Sodium and bath penetration in the carbon cathode blocks for aluminium electrolysis are known to be amongst the most detrimental phenomena for the cell lifespan. Although much work has been done over the years, some important questions still remain to be answered. In an attempt to adress this problem, we present an ongoing project dedicated to answer specific questions: How the sodium is carried inside the blocks? Which compounds are formed between sodium and carbon? How the bath penetrates the block and how can we model it? What is the connection between sodium, bath penetration and the augmentation of the cathodic voltage drop? This first part will present the background for each question in the form of a literature review. Then we will present the strategies used to try to give some answers to the previously asked questions along with the results obtained so far.

2:25 PM
Development and Validation of a Thermo-Chemo-Mechanical Model of the Baking of Ramming Paste: *Daniel Richard*[2]; Guillaume D'Amours[3]; Mario Fafard[1]; Martin Désilets[4]; [1]Laval University, Civil Engrg. Dept., Pouliot Bldg., Quebec, Quebec G1K 7P4 Canada; [2]Université du Québec, Applied Sci. Dept., 555 boul. de l'Université, Chicoutimi, Québec G7H 2B1 Canada; [3]Aluminium Technology Centre, Natl. Rsch. Council Canada, 501 boul. de l'Université E, Chicoutimi, Québec G7H 8C3 Canada; [4]Alcan International Limited, Arvida R&D Ctr., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

During preheating and the early operation stages of a Hall-Héroult cell, the green ramming paste undergoes several transformations. This cohesive porous granular material will first swell and will lose mass and then, it will develop more porosity and shrink. Moreover, it is also mechanically compressed by the thermal expansion of the cathode blocks. In the on-going project START-Cuve, a new thermo-chemo-mechanical constitutive law has been developed to model the behaviour of the ramming paste. Stresses and strains generated in the rammed seam during baking, can now be accurately predicted. This paper presents some aspects of the mechanical model, which was largely based on extensive experimental observations, both at ambient and at high temperatures. The model was implemented in the finite element code FESh++. To illustrate the complex behaviour of the ramming paste and the capabilities of the model, simulations have been performed using representative temperature evolution scenarios and mechanical loading paths.

2:50 PM
A Laboratory Evaluation of Ramming Paste for Aluminium Electrolysis Cells: *Flemming Bay Andersen*[1]; [1]Corus Research, Development & Technology, Ceram. Rsch. Ctr., PO Box 10000, Bldg. 3J22, IJmuiden 1970 CA The Netherlands

At the primary aluminium smelters of Corus a number of ramming pastes have been used in the pots over the years. Furthermore a lot of new paste qualities are available on the market. The information provided by the suppliers on paste properties is limited and this makes it difficult to compare products. In order to gain knowledge on paste properties and in order to approve ramming paste qualities for use in Corus electrolysis cells we have tested a number of ramming paste qualities. In this paper the results of these tests are presented together with a discussion on expected performance in electrolysis cells. Of special interest are the test results from the so-called "eco-friendly" qualities and how they compare to the standard qualities. Based on the testing results product specifications are proposed to be used for approving ramming pastes from new and existing suppliers.

3:15 PM
Surface Exchange of Sodium, Anisotropy of Diffusion and Diffusional Creep in Carbon Cathode Materials: *Alexander Zolochevsky*[1]; Jørund G. Hop[2]; Trygve Foosnæs[1]; Harald A. Øye[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Sem Sælandsvei 12, Trondheim N-7491 Norway; [2]Hydro Aluminium, Tech. & Operational Support, Øvre Årdal N-6884 Norway

Experimental data for the expansion of a semigraphitic cylinder due to sodium diffusion in the axial and radial directions are explained by anisotropic diffusion coefficients and surface exchange coefficients. Compressive creep deformation due to sodium diffusion into the binder phase (short-term creep) produces reduction of the sodium expansion when an external pressure is applied to the material. A constitutive model describes the diffusional short-term creep of an anthracitic material. The long-term creep deformation is considered related to sodium diffusion into grains. Creep measurements have been made for a semigraphitic material under axial loading applied after the maximum value of free sodium expansion. A new constitutive model considering the surface boundary conditions with the exchange rate of sodium describes the carbon swelling and short-term creep. Modeling of the diffusional long-term creep in grains under loading after binder phase sodium saturation is also described. Validation of the proposed model through correlation of theoretical results and experimental data for the sodium concentration, sodium expansion and creep strain is performed. Practical recommendations on how to extend the model to full-scale reduction cells are formulated.

3:40 PM Break

3:55 PM
Influence of Internal Cathode Structure on Behavior During Electrolysis. Part I: Properties of Graphitic and Graphitized Materials: *Frank Hiltmann*[1]; Pretesh M. Patel[2]; Margaret Hyland[2]; [1]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, 65933 Frankfurt Germany; [2]University of Auckland, Dept. of Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand

The internal structure of cathode blocks used in modern aluminium smelting cells will determine their physical, mechanical and operating characteristics. A comprehensive study was undertaken to determine the effect changes in internal structure have on graphitized and graphitic cathodes. Pronounced differences in internal structure were achieved by extreme variation of cathode formulations in lab scale thus allowing characteristics such as porosity and grain orientation to be manipulated. Standard properties such as Young's modulus, electrical resistivity, coefficient of thermal expansion, flexural and compressive strength were measured and the variables most affecting these properties evaluated. Results showed that - for the applied range of formulations - graphitic cathode specimens had superior mechanical properties to graphitized ones, with grain orientation having a significant effect on properties. This is attributed to greater packing efficiency and preferred grain orientation, thus reducing porosity and subsequently improving properties.

4:20 PM
Influence of Internal Cathode Structure on Behavior During Electrolysis. Part II: Porosity and Wear Mechanisms in Graphitized Cathode Materials: *Pretesh M. Patel*[1]; Margaret Hyland[1]; Frank Hiltmann[2]; [1]University of Auckland, Dept. of Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand; [2]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, 65933 Frankfurt Germany

Overall cathode wear in aluminium reduction cells is due to a combination of physical, chemical and electrochemical processes acting simultaneously on cathode blocks during operation. Due to advances in technology graphitized cathode blocks have been more extensively used in the aluminium smelting industry. However, technology progress has also seen the use of high excess AlF3 concentrations and high current densities during electrolysis. These two conditions have been linked to an increase in electrochemical attack on cathode blocks. Research conducted has looked at the chemical and electrochemical wear occurring during lab-scale electrolysis on graphitized cathode samples. Emphasis was placed on the wear mechanisms such as Al4C3 formation within the pores of the cathode sample. During electrolysis pores within the cathode will fill with bath and therefore promote electrolytic sub-surface wear of cathodes. Total porosity and pore-size distributions of cathode blocks will therefore play an important role in cathode wear.

4:45 PM
A Study on the Fundamentals of Pothole Formation: *Eng Fui Siew*[1]; Mark P. Taylor[3]; Toni Ireland-Hay[2]; Gretta Theobald Stephens[2]; John J.J. Chen[1]; [1]University of Auckland, Cheml. & Matls. Engrg. Dept., PB 92019, Auckland New Zealand; [2]Comalco Research and Technical Support, 15 Edgars Rd., Thomastown, VIC 3074 Australia; [3]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand

Potholing is a localised and rapid type of erosion through the carbon cathode. It only takes a single pothole to cause a reduction cell failure if the eroded hole is deep enough for the molten aluminium to come in contact with a collector bar. The contact between the molten metal and collector bar allows iron to be dissolved into the molten aluminium, thus contaminating the metal produced. This form of cathode wear is one of the predominant failure modes for aluminium reduction cells. If a better understanding of the contributing factors to the initiation and propagation of potholes can be achieved, then actions may be taken to prevent or reduce their occurrence. This paper discusses the presently held beliefs and hypotheses behind the formation of potholes, compares them to plant data and introduces other possible pothole formation theories.

Cast Shop Technology: DC Casting: Microstructure and Hot Tearing

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday PM Room: 2001
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Laurens Katgerman, Delft University, Netherlands Inst. for Metals Rsch., Delft 2628 AL The Netherlands; Bjørn-Rune Henriksen, Elkem Aluminium, Rsch., Kristiansand 4675 Norway

2:00 PM
Solidification Studies of 6XXX Alloys With Different Mg and Si Contents: Hu Jin[1]; Majed Jaradeh[1]; *Torbjörn Carlberg*[1]; [1]Mid Sweden University, Engrg., Physics & Math., Holmgatan 10, Sundsvall 85170 Sweden

In choosing alloys for structural applications, a trade off between strength of the material and extrudability is made. A trend is that higher contents of Si than of Mg becomes more common, as this type of alloys tend to give an optimum combination of the mentioned properties. However, in the cast house the experience is that such alloys are more sensitive to surface defects, and thus more difficult to cast than alloys with balanced content of Si and Mg. It is therefore of interest to increase the knowledge of the solidification behaviour of these type of alloys. Directional solidification studies, simulating billet casting, have been made off alloys with balanced and not balanced Mg and Si additions, and with different alloying levels ranging from 6063 to 6082. Effects of growth rate, grain refinement, temperature gradient and composition on structure formation have been investigated.

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Thermo-Inelastic Simulation of Butt Curl Phenomena During Aluminum DC Casting: *Nobuhito Ishikawa*[1]; [1]Furukawa-Sky Aluminum Corp., Melting & Casting Tech. Sec., Techl. Rsch. Div., 21-1 Kurome, Mikuni-cho, Sakai-gun, Fukui-pref. 913-8588 Japan

In an aluminum DC casting, butt curl phenomena are recognized at the short side of its bottom region and they are prone to decrease the productivity through fatal defects such as hot cracking or metal bleeding. In order to evaluate the mechanism of butt curl growth, a thermo-mechanical finite element model has been developed, in which thermally induced strains and stresses associated with solidification are simulated using an elasto-plastic constitutive equation based on an isotropic hardening rule and the Mises yield condition. Comparison of butt curls shows a good agreement between calculations and measurements within tolerance of?\@?}20%. As a driving force of butt curl, a torque moment due to tensile stresses distribution along a mushy region makes the solidified bottom shell bent rapidly when the whole slab surface is covered with secondary coolant.

2:50 PM
The Investigation into Kinked Sheet Ingot Butts at an Alcan Casthouse: Clark Weaver[1]; Yves Caron[2]; Joseph Langlais[2]; Marco Biagioni[3]; *Jean-Claude Pomerleau*[3]; [1]Alcan Primary Metal Group, 1955 Mellon Blvd., Jonquière, QC G7S 4K8 Canada; [2]International Limited, 1955 Mellon Blvd., Jonquière, QC G7S 4K8 Canada; [3]Alcan Primary Metal Group, 1 Smelter Site Rd., Kitimat, BC V8C 2H2 Canada

Sheet ingots that are subsequently to be hot rolled into coils have to meet rather stringent specifications regarding their cross sectional shape and their straightness. The cross sectional shape is fixed by the mould contour and the casting speed for any given alloy and this subject has been the object of many papers in the past. The ingot straightness (i.e. the absence of longitudinal or lateral bow and twist) is dependant on several factors such as casting machine alignment, bottom block condition and starting practices, just to name a few. A rather unique problem of occasional kinked sheet ingot butts was identified in one of Alcan's sheet ingot casting centers about a year ago. This paper describes the methodology that was used to eliminate possible causes as well as the surprising results obtained via a series of short test casts done on the production machine. Upon further investigation, the mechanism causing the kinked ingots was found to exist at many of the other Alcan sheet casting centers. A simple solution was proposed, tested and confirmed as a way to prevent kinking in the bottom butt region of sheet ingots.

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Integrated 3D Model to Simulate Solidification and Predict Hot Cracking: *Zhengdong Long*[1]; Qingyou Han[2]; Srinath Viswanathan[3]; Shridas Ningileri[4]; Subodh Das[4]; Kazunori Kuwana[5]; Mohamed Hassan[5]; Marwan Khraisheh[5]; Adrian S. Sabau[2]; Kozo Saito[5]; [1]University of Kentucky, Ctr. for Al Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA; [3]Sandia National Laboratories, Matls. & Process Scis. Ctr., Albuquerque, NM 87185-0889 USA; [4]Secat, Inc., 1505 Bull Lea Rd., Lexington, KY 40511 USA; [5]University of Kentucky, Dept. of Mechl. Engrg., Lexington, KY 40506-0108 USA

An integrated 3D Direct Chill (DC) casting model was used to simulate the heat transfer, fluid flow, solidification, and thermal stress during casting. Temperature measurements were performed in an industrial casting facility to setup and validate the model. The key features such as heat transfer between cooling water and the ingot surface as a function of surface temperature, cooling water flow rate, air gaps caused by mold and bottom block design were also coupled into the model. An elasto-viscoplastic constitutive model, which was determined based on mechanical testing, was used to calculate the evolution of stress during casting. The stress evolution was compared at various locations and correlated with physical phenomena associated with the casting process. An Ingot Cracking Index, which represents the ingot cracking propensity, was established based on the ratio of stress to strength. The Index calculation results were consistent with cracking observations in industrial casting practice.

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Implementation of a Strain Based Hot Tearing Criterion in Direct Chill Cast Aluminum Ingots: *Andre B. Phillion*[1]; Steve L. Cockcroft[1]; Daan M. Maijer[1]; Mary A. Wells[1]; [1]University of British Columbia, Dept. of Matls. Engrg., Frank Forward Bldg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Hot tearing in D.C. ingot castings has been investigated using a strain based hot tearing criterion, implemented into a three dimensional coupled thermal - stress finite element process model. In this presentation, the strain predictions in two different casting simulations will be discussed, as the strain fields from one casting recipe clearly show a high accumulation of tensile strain during solidification on the rolling face, just above the ingot lip. Finally, a comparison of the predicted plastic strain and mushy zone ductility data will be shown. In the critical region just above the ingot lip, the plastic strain accumulated during solidification exceeds the ductility limit at a temperature of about 575°C. Both further up the ingot, and out towards the edge of the rolling face, the accumulated strain was less than the ductility limit.

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Hot Tearing of Aluminium - Copper Alloys: *David Michael Viano*[1]; [1]University of Queensland, Div. of Matls., Sch. of Engrg., St. Lucia, Queensland 4072 Australia

For many aluminium alloys, hot tearing susceptibility follows a lambda curve relationship when hot tearing severity is plotted as a function of solute content. In the past, there has been some difficulty quantifying hot tearing. Traditional methods rely upon measuring electrical resistivity or the number and/or length of cracks in tests such as the ring test. In this experimental program, a hot tear test rig was used. This device measures the load imposed on the mushy zone during

solidification. It was found that the load recorded at completion of solidification (i.e. the solidus) was a good indicator of hot tearing susceptibility. The load measured at the solidus of a range of binary Al-Cu alloys is compared with published results from other researchers. It is found that the results from the hot tear rig are reproducible and correlate well with those obtained by other experimental methods.

Characterization of Minerals, Metals and Materials: Materials Preparation and Characterization

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

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Session Chairs: James J.Y. Hwang, Michigan Technological University, Inst. of Matls. Procg., Houghton, MI 49931-1295 USA; Ann M. Hagni, Construction Technology Laboratories Inc., Microscopy Grp., Skokie, IL 60077 USA

2:00 PM
Elastic Properties of FeGa and FeGaAl Alloys with Resonance Ultrasound Spectroscopy: *Pinai Mungsantisuk*[1]; Sivaraman Guruswamy[1]; [1]University of Utah, Metallurgl. Engrg., 135 S., 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Resonance Ultrasound Spectroscopy is a measurement technique using absorption of sound waves at the natural resonance frequencies of a material body to determine elastic properties. In this work, the elastic properties of Fe-x at.% Ga and Fe-(20-x) at.% Ga-x at.% Al polycrystalline alloys were evaluated. The samples used had parallelepiped shape. The absorption spectra of FeGa and FeGaAl alloys obtained using a Dynamic resonance modulus system over a specific frequency range were analyzed assuming the material to be isotropic. The elastic constant values, C11 and C44, were obtained from which Young's modulus and shear modulus values were determined. The Young's modulus obtained from FeGa and FeGaAl alloys were in the range of 110 to 170 GPa are much greater than those obtained from Terfenol-D alloys. The paper examines the trend in elastic property variation with changes in Ga and Al content. Work supported by NSF-DMR Grant #0241603.

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Lattice Parameter Determinations of Potassium Nitrate (KNO_3)-Ammonium Nitrate (NH_4NO_3) Solid Solutions: *Wen-Ming Chien*[1]; Dhanesh Chandra[1]; Jennifer Franklin[1]; Claudia J. Rawn[2]; Abdel K. Helmy[3]; [1]University of Nevada, Metallurgl. & Matls. Engrg., MS 388, Reno, NV 89557 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6064 USA; [3]Special Devices Inc., 14370 White Sage Rd., Moorpark, CA 93021 USA

The lattice parameters of the low and high temperature phase of KNO_3-rich (in NH_4NO_3) solid solutions have been determined by the X-ray diffraction method. The low temperature single KNO_3 phase (KN II) are from 92%-100% KNO_3 in NH_4NO_3 composition range and from room temperature to 393 K showing in X-ray diffraction patterns. The high temperature KNO_3 phase (KN I) showed very broad composition range between 20% to 100% KNO_3. The lattice parameters of KNO_3-rich (KN II and KN I) solid solutions have been calculated at different temperature range. The volumes of orthorhombic KN II phase decrease from 0.3218(4) to 0.3195(3) nm^3 at the room temperature as the compositions increase from 92% to 100%KNO_3. The lattice constants of the hexagonal KN I phase show that there is no significant change in a-direction when the temperature increases. Detail X-ray results and lattice expansion equations during heating will be presented.

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An EPR Characterization of Vanadium in CaO and Na2O Based Al2O3-SiO2 Glasses: *Humera Farah*[1]; [1]GIK Institute of Engineering Sciences and Technology, Faculty of Metall. & Matls. Engrg., Topi, District Swabi, N.F.W.P. Pakistan

The relationship between glass composition and local structure was examined by Electron Paramagnetic Resonance (EPR) analysis of vanadium (IV) in CaO and Na_2O based Al_2O_3-SiO_2 glasses. The characteristic EPR specrtrum of vanadium (IV) was affected with change in glass composition, equilibrium oxygen partial pressures and melting temperature. From the available previous reports and current study, it seems that a close relationship exists between octahedral and tetrahedral vanadium, which depends upon temperature. The results are important to understanding of vanadium behavior as a candidate in Al-V-Si BMGs and their nano composites.

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Variation in Poisson's Ratio with Other Elastic Constants: An Attempt Towards Rationalization of Elastic Constants for Isotropic Solid Materials: *Anish Kumar*[1]; T. Jayakumar[1]; Baldev Raj[1]; Kalyan K. Ray[2]; [1]Indira Gandhi Centre for Atomic Research, Div. for PIE & NDT Dvlp., Kalpakkam, TamilNadu 603102 India; [2]Indian Institute of Kharagpur, Dept. of Metallurgl. & Matls. Engrg., Kharagpur, W. Bengal 721302 India

The polycrystalline isotropic materials are characterized by only two independent elastic constants. Hence, identification of any new relationship between two independent elastic constants would reduce the number of required independent elastic constants to one. In this direction, an attempt is made to study the variation in Poisson's ratio with other elastic constants using the experimental data generated by the authors and also that collected from the literature for various isotropic solid materials, such as pure elements, ceramics, polymers and intermetallics. The analysis revealed that Poisson's ratio decreases with increase in other elastic constants and a linear correlation is obtained between Poisson's ratio and ultrasonic shear wave velocity. It is also deduced that shear wave velocity is a better parameter for microstructural characterization as compared to longitudinal wave velocity. These observations reveal the possibility for rationalization of elastic constants at least for a group of alloy systems with different microstructural conditions.

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Thermodynamic Analysis on Synthesis of Fibrous Ni-Co Alloy Powder Precursors with Coprecipitation Process: *Zhan Jing*[1]; *Zhang Chuan Fu*[1]; Wu Jian Hui[1]; Masafumi Maeda[2]; Ryoichi Yamamoto[2]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China; [2]University of Tokyo, Dept. Matls. & Environment, Inst. of Industrial Sci., Tokyo Japan

The mathematics model of Ni(•)-Co(•)-C2O4(2-)-NH3?H2O-H2O of thermodynamic equilibrium system has been developed theoretically according to the law of conservation of mass and the law of simultaneous equilibrium, and the logarithm concentration-pH (log[Me2+]T-pH) diagrams of this system have been made. The effect of ligand and pH value was studied. Two issues in coprecipitation route to synthesize fibrous nickel-cobalt alloy precursor powders, composition and morphology which are a first priority, are addressed based on thermodynamic analysis and experiments. The results show that suitable pH value range of coprecipitation of nickel ions and cobalt ions in theory can be obtained to maintain fibrious, excellent compositional homogeneity and stoichiometry for in coprecipitation products. Experimental demonstration is also carried out.

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Synthesis and Characterization of Fibrous Ni-Co Alloy Precursor Particles by Modified Coprecipitation Method: Zhang Chuan Fu[1]; *Zhan Jing*[1]; Wu Jian Hui[1]; Masafumi Maeda[2]; Ryoichi Yamamoto[2]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China; [2]University of Tokyo, Dept. Matls. & Environment, Inst. of Industrial Sci., Tokyo, Tokyo Japan

Homogeneous solid-solution oxalates of Ni2+ and Co2+ ions were synthesized by modified co-precipitation method and the composition and morphology of fibrous solid-solution oxalates precursor were characterized by XRD, IR, DTA/TGA and SEM analysis. The effects of feeding methods, pH values, precipitation temperature, reactant concentration, addition surfactant, alcohol washing and drying process on the morphology and dispersion of the precursor were investigated in detail. The experimental results showed that the co-precipitated powders have characteristics that are different from those of the mechanical mixtures with the same stoichiometry. Based on the observations of precursor growth and SEM morphology of the precursor, the oriented attachment was proposed for the well-aligned growth of the fibrous Ni-Co alloy precursor particles.

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Preparation and Characterization of Fibrous Ni-Co Alloy Powders by Thermal Decomposition of Solid Solutions of Complex Ni-Co Oxalates: Zhang Chuan Fu[1]; *Zhan Jing*[1]; Wu Jian Hui[1]; Masafumi

Maeda[2]; Ryoichi Yamamoto[2]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China; [2]University of Tokyo, Dept. Matls. & Environment, Inst. of Industl. Sci., Tokyo Japan

Synthesis of fibrous Ni-Co alloy powders was achieved by thermal decomposition of solid solutions of complex Ni-Co oxalates precursor obtained via modified coprecipitation method among intimate mixing solution of NiCl2.6H2O, CoCl2.6H2O and H2C2O4.2H2O with surfactant PVP and organic solvent A. Scan electron microscopy(SEM), X-ray diffraction(XRD), X-Ray photoelectron spectroscopy (XPS), energy X-ray analyses(EDX) were used to characterize the structure features and chemical compositions of the as-prepared fibres. The influences of pyrolytic conditions, including temperature, time, the flow rate of N2+H2, and the heating rate, on the morphology, average size and specific surface area of the Ni-Co alloy powders were investigated. It is found that a trend of the mutual alloying of nickel and cobalt: the formation of perfect solid solution for any compositions NixCo100-x was indicated and corresponding NixCo100-x alloys with fcc phase can be obtained under the optimal conditions.

Computational Thermodynamics and Phase Transformations: Thermodynamic Models and Databases

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Wednesday PM Room: 3005
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Moneesh Upmanyu, Colorado School of Mines, Matls. Sci. Prog., Div. of Engrg., Golden, CO 80401 USA

2:00 PM
Some Issues on Integration of First-Principles Calculations and CALPHAD Modeling: *Zi-Kui Liu*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

CALPHAD modeling has become a mature technique in developing thermodynamic databases of multicomponent systems. It has also been extended to the development of other databases such as atomic mobility and lattice parameters. In the CALPHAD modeling of thermodynamics, the modeling of Gibbs energy of individual phases and the coupling of phase equilibria and thermochemistry are the key in developing internally consistent thermodynamic descriptions of multicomponent materials with sound fundamentals and predictive power because these two sets of data are deduced from the Gibbs energy of individual phases under given constraints. One issue in CALPHAD modeling is the lack of experimental data, particularly for new materials. Experimentally, it is relatively easier to obtain phase equilibrium data in comparison with thermochemical data. On the other hand, first-principles calculations predict thermochemical data better than phase equilibrium data. More and more researchers are using enthalpy of formation from first-principles calculations. In this presentation, the recent research activities at our Phases Research Lab at Penn State will be presented with the following issues addressed. [1]Calculations of enthalpy of mixing in phases with solubility ranges using the special quasirandom structures (SQS). [2]Structure stability related to the SQS calculations. [3]Efficient route in obtaining entropy of formation and entropy of mixing.

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Utilizing Crystal Structure Correlations for Structure Prediction: Kevin Tibbetts[1]; *Chris Fischer*[1]; Dane Morgan[1]; Gerbrand Ceder[2]; [1]Massachusetts Institute of Technology, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]University of Wisconsin, 1509 Univ. Ave., Madison, WI 53706 USA

When faced with the task of determining the stable structure types of a new alloy system, scientists have relied on physical intuition, quantum mechanics, model hamiltonians, and usually a healthy dose of past experience. This talk will present an approach that can combine the guidance of previous data and the accuracy of quantum mechanics. In this approach, the information from past experience, either experimental or computed, is captured in a mathematical framework of correlations, which then in turn can be used to direct accurate first principles computations. As a result, our model is independent of any specific hamiltonian or sophistication of physical understanding. When applied to data taken from a database of experimental information, the extracted structure-structure correlations can be used in a generic manner to predict the stable structure types for new alloy systems. The predictive capability and quality of our model will be shown to expedite the search for stable phases in binary alloy systems. These predictions are applicable to both computational and experimental communities investigating new or partially observed alloy systems. The information content of our model will be compared with the basic behavior of data contained within the experimental database used in this study.

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Modelling Precipitate Evolution in Aluminium Alloys During Complex Processing: *Joseph Douglas Robson*[1]; Nicolas Kamp[1]; [1]UMIST, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK

A coupled microstructural modelling approach is described to predict the precipitate evolution in multicomponent industrial aluminium alloys during complex, non-isothermal, processing. The thermodynamic properties of the precipitate phases are calculated using JMatPro, a software package based on Calphad methods. This thermodynamic data is used as inputs to a numerical model for phase transformation kinetics based on classical theory and the Kampmann and Wagner methodology. This framework has been extended to include multiple precipitate phases, as well as both heterogeneous and homogeneous precipitation under non-isothermal conditions. This is demonstrated by predicting the microstructural evolution during welding of a 7xxx series aluminium alloy and comparing with experiment.

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Simulation of the Kinetics of Precipitation Reactions in Ferritic Steels: *Andre Schneider*[1]; Gerhard Inden[1]; [1]Max-Planck-Institut fuer Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, Duesseldorf 41199 Germany

Computer simulations of diffusion controlled phase transformations in model alloys of Fe-Cr-C, Fe-Cr-W-C, Fe-Cr-Si-C, and Fe-Cr-Co-V-C are presented. The compositions considered are typical for ferritic steels. The simulations are performed using the software DICTRA and the thermodynamic calculations of phase equilibria are performed using Thermo-Calc. The thermodynamic driving forces and the kinetics of diffusion controlled precipitation reactions of M23C6, M7C3, cementite and Laves-phase (Fe, Cr)2W are discussed. The simultaneous growth of stable and metastable phases is treated in a multi-cell approach. The results show remarkable effects on the growth kinetics due to the competition during simultaneous growth.

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Phase Equlibria Predictions in Nb-Silicide Based Composites: *Ying Yang*[1]; Bernard P. Bewlay[2]; M. R. Jackson[2]; Y. Austin Chang[1]; [1]University of Wisconsin, Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [2]General Electric Global Research, Schenectady, NY 12301 USA

Nb-silicide based in-situ composites are promising materials for future high-temperature structural applications. Nb-Si alloys are typically alloyed with Hf, Ti, Cr, and Al to provide a balance of mechanical and environmental properties. In order to develop an improved understanding of phase equilibria in Nb-Hf-Ti-Si quaternary system, a methodology coupling the CALPHAD-type computational thermodynamics with experimental measurement of phase equilibria was used in this study. This paper first describes phase equilibria in the Hf-Ti-Si ternary system determined by experiments. This data was then used to develop a thermodynamic description of the Hf-Ti-Si system. A thermodynamic description of the Nb-Ti-Hf-Si quaternary system was then obtained by extrapolating the thermodynamic descriptions of Hf-Ti-Si, Nb-Hf-Ti, Nb-Ti-Si and Nb-Hf-Si into the quaternary space. The phase equilibria and solidification paths predicted from the currently obtained Nb-Ti-Hf-Si quaternary thermodynamic description are compared with experimental results.

3:40 PM
Incorporating Volume in Thermodynamic Databases: *Suzana G. Fries*[1]; Alan T. Dinsdale[2]; Bo Sundman[3]; [1]ACCESS e. V., Intzestrasse 5, Aachen D-52072 Germany; [2]NPL, Teddington, Middlesex TW11 0LW UK; [3]MSE, KTH, Div. of Computatl. Thermodynamics, Stockholm SE-10044 Sweden

Powerful multicomponent thermodynamic databases based on semi-empirical modelling of the Gibbs energies are able to describe other thermodynamic functions which are related to the Gibbs energy through its derivatives. Such properties include the Enthalpy, Entropy, Heat Capacity etc, as functions of composition and temperature for each phase present. In addition the same properties can be calculated for complex multiphase stable and metastable equilibria. One of the reasons for the success of the use of these databases lies in the standards adopted by Scientific Group Thermodata Europe (SGTE) for the temperature dependence of the Gibbs energy for the pure elements in their stable and metastable states. These values are used internationally and this ensures that the effort of the whole thermodynamic community in the production of data is universally consistent. Calculated results from use of these databases are also being provided as input to other applications involving kinetics eg phase transformation and microstructure simulations require information about other derivatives of the Gibbs energies such as Thermal Expansion and Bulk Modulus as functions of composition. A prerequisite for the incorporation of these and other derivatives of the Gibbs energy in the database is the definition of data for the molar volumes of the pure elements and other end-members and their variation with temperature and pressure. The use of experimental and theoretically calculated information as input for the construction of this set of standard reference data for the molar volumes of phases for a ternary system will be presented.

4:00 PM Break

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Comparison Between Calculations and Measurements of Precipitate Phases and Their Compositions in Cr-Mo Alloys: *J. M. Vitek*[1]; R. L. Klueh[1]; P. J. Maziasz[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Bldg. 4508; MS 6096, Oak Ridge, TN 37831-6096 USA

Calculations of equilibrium phases and their compositions were made for several different Cr-Mo steel alloys that are of interest in high temperature applications. The predicted results were compared with experimental data for extremely long aging times (up to 75,000 h). The experimental data were based on extensive analytical electron microscopy results of extractions made from the aged materials. The comparisons were specifically made with respect to the types of precipitates found, their amounts, and their compositions. The predictions showed reasonably good agreement with experimental results, including the compositions of the precipitates. However, some discrepancies were noted and these will be described in detail. This research was sponsored by the Division of Materials Sciences and Engineering, and the Office of Science, Laboratory Technology Research Program, U. S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:35 PM
Phase Diagram Calculations of the NH4NO3-KNO3 Binary System: *Raja Chellappa*[1]; Dhanesh Chandra[1]; [1]University of Nevada, Metallurgl. & Matls. Engrg., MS 388, Reno, NV 89557 USA

The study of ammonium nitrate [AN: NH4NO3]-potassium nitrate [KN: KNO3] binary system is very important for practical applications such as automobile air bag gas generators. In this work, we will present the results of our phase diagram computations of the AN-KN system. The thermodynamic optimization was carried out using our experimental phase diagram as the basis utilizing the PARROT module of the Thermo-CalcTM software. The AN-KN binary system exhibits complex behavior with seven invariant equilibria: three peritectoids, two eutectoids, one congruent, and one eutectic transformation. Interaction parameters were considered only for the high temperature KN-rich phase (subregular solution) and liquid phase (regular solution). All the other phases were assumed to be ideal solutions. The optimized phase diagram under these assumptions is in good agreement with the experimentally determined phase diagram.

4:55 PM
A Thermodynamic Database for Thermal Energy Storage Materials: *Raja Chellappa*[1]; Dhanesh Chandra[1]; [1]University of Nevada, Metallurgl. & Matls. Engrg., MS 388, Reno, NV 89557 USA

Alcohol and amine derivatives of neopentane are potential candidates for thermal energy storage. Examples include Pentaerythritol [PE:C(CH2OH4)], 2-amino-2-methyl-1,3-propanediol [AMPL: (NH2)(CH3)C(CH2OH)2], Pentaglycerine [PG:(CH3)C(CH2OH)3], Neopentylglycol [NPG:(CH3)2C(CH2OH)2], and Tris(hydroxymethyl) aminomethane [TRIS:(NH2)C(CH2OH)3]. In recent times, we have published calculated phase diagrams of NPG-AMPL and PE-AMPL under the CALPHAD framework, based on our experimental phase diagram studies. Work is under progress on other binary systems such as PE-NPG, and TRIS-NPG. We will present the results of our phase diagram computations and discuss the evolution of a thermodynamic database for these organic compounds.

5:15 PM
Experimental Investigation and Thermodynamic Description of the Quaternary Organic Alloy System AMPD -DC -NPG -SCN: *Victor T. Witusiewicz*[1]; Laszlo Sturz[1]; Ulrike Hecht[1]; Stephan Rex[1]; [1]ACCESS e.V., Matls. & Processes, Intzestr. 5, Aachen D-52072 Germany

Transparent organic substances with low entropy of fusion (plastic crystals) for the past seventy years have been used as analogues to metals, because they allow for in situ observation of the microstructure evolving at the solid/liquid interface. Unfortunately, the number of such organic alloy systems is quite limited and experimental information concerning the phase diagrams and related thermodynamic data is scarce. Furthermore, no hints in literature on the existence of ternary or quaternary eutectic alloys formed by plastic crystals have been found. The search for new suitable organic alloys gained new drive within the frame of solidification studies addressing pattern formation in multicomponent, multiphase alloys. We have found experimentally that AMPD-DC-NPG-SCN system is suitable for such purposes. The temperature and enthalpy of phase transformations of organic alloys of the constituent binary and ternary systems of the quaternary AMPD-DC-NPG-SCN system were measured by means of differential scanning calorimetry. The quaternary phase diagram was assessed via the CALPHAD approach using Thermo-Calc by simultaneously optimizing the thermodynamic and phase equilibrium data measured in the present work. A good agreement between the experimental and calculated data for the phase diagram as well as for the thermochemical properties was achieved. Additionally unidirectional solidification of several eutectic alloys was performed in order to verify the nature of the eutectics. We have found the composition region for eutectic alloys that grow with three nonfacetted phases and result in very regular lamellar or rod like patterns. Note: AMPD is 2-amino-2-methyl-1,3-propanediol, CAS No [115-69-5]; DC is (D)camphor or (1R)-1,7,7-trimethylbiciclo[2,2,1]heptan-2-one, CAS No [464-49-3]; NPG is 2,2-dimethyl-1,3-propandiol, CAS No [126-30-7]; SCN is succinonitrile, CAS No [110-61-2].

Friction Stir Welding and Processing III: Microstructure and Texture

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday PM Room: Nob Hill C/D
February 16, 2005 Location: San Francisco Marriott

Session Chair: Rajiv S. Mishra, University of Missouri, Rolla, MO 65409-0340 USA

2:00 PM Invited
A Preliminary Investigation on the Microstructure and Properties of Friction Stir Welds in an Al7075/Al2O3/10% Reinforced Alloy: Luisa Marzoli[1]; *Jorge F. dos Santos*[1]; Rudolf Zettler[1]; Mario Volpone[2]; Enrico Rizzuto[3]; [1]GKSS Forschungszentrum, Inst. for Matls. Rsch., Joining Tech., Max-Planck-Str. 1, Geesthacht D-21502 Germany; [2]Fincantieri Cantieri Navali S.p.A, via Cipro, 11, Genova 16129 Italy; [3]University of Genova, DINAV, Ship Structl. Design, Via Montallegro, 1, Genova 16145 Italy

The main objectives of this study were to establish a friction stir welding (FSW) process parameters envelope for an Al7005 alloy reinforced with 10% of Al2O3 and to determine the microstructural features and mechanical properties of the welded joints produced with optimised process technology. Weld thermal cycles recorded during welding have shown that thermal stability conditions could be reached in all investigated welding conditions. Substantial differences have been observed between the microstructures in base material and stir zone. In the latter, a large amount of very small (4 μm and less), rounded particles, totally absent in the base material, has been observed. The tensile testing revealed joint efficiencies of 100% for the yield strength and of around 95% for the UTS. Failure in tensile testing always took place outside the stir zone. The fact that high quality joints could be

obtained in a wide range of parameter combinations indicates that the FSW process has the required robustness for industrial application to the investigated alloys.

2:20 PM
Microstructure Evolution During Friction Stir Welding of Commercial Aluminum Alloys: *Bertrand Huneau*[1]; Xavier Sauvage[2]; Surendar Marya[1]; Arnaud Poitou[1]; [1]Ecole Centrale de Nantes, GeM UMR CNRS 6183, 1, rue de la Noë; BP 92101, Nantes 44321 France; [2]Université de Rouen, Inst. des Matériaux de Rouen GPM UMR CNRS 6634, Ave. de l'univ., BP 12, Saint Etienne du Rouvray 76801 France

The friction stir welds result from frictional heating combined with intense plastic deformation of the material induced by the tool in the processed zone. The joints are free of defects and usually exhibit a fine recrystallised microstructure which make them strong and ductile. However, this microstructure dramatically depends on the rotation and translation speed of the tool, and on the diameter of the pin and of the shoulder. The aim of this work was to investigate thanks to scanning electron microscopy (SEM) and transmission electron microscopy (TEM) the microstructure evolution during friction stir welding of commercial aluminum alloys (cold rolled 1050 and Al-Sc alloys). Grain morphology and size together with precipitates distributions through the welds provide new information about the material flow in the process zone. The influence of the Al2O3 oxide layer prior to the welding process was also investigated.

2:40 PM
Microstructural Details of Friction-Stir Weld Interfaces: *A. C. Somasekharan*[1]; L. E. Murr[1]; [1]University of Texas, Dept. of Metallurgl. & Matls. Engrg., 500 W. Univ. Ave., El Paso, TX 79968-0001 USA

This study dealt with the unique interfaces within the flow regimes observed in the friction-stir welding (FSW) of dissimilar magnesium (Mg) and aluminum (Al) alloys. Mg alloys AZ31B-H24 (hot-rolled), and AZ91D (semi-solid cast, primary solid fractions of ~3% and ~20%) were friction-stir welded to Al wrought alloys 6061-T6, and 5052-H34. Dissimilar Mg alloy welds included the FSW of AZ31B-H24 with AZ91D, and the dissimilar Al alloy welds included the FSW of 6061-T6 and 5052-H34. Optical microscopy, scanning electron microscopy, and transmission electron microscopy were some of the microscopy techniques used in the analysis of the welds and the weld-related interfaces. In the case of dissimilar systems A and B, the transition or interfacial regime from the base metal to the weld zone is characterized by a narrow recrystallized band of A grains, a mixture of A and B, and B grains making up the interfacial regime, especially on the advancing side. The transition from the interface regime to the base material is characterized by a zone of extreme plastic deformation where the grains are distorted, and this distortion decreases into the base material. The complex intercalated mixture of materials within the weld zone of Mg-Al welds is characterized by lamellar and intercalated recrystallized bands of either material, along with swirls and vortexes unique to dissimilar welds. The weld zones of dissimilar Mg alloy and Al alloy welds also show an intercalated mixing of materials, with the interfacial regime from the base material to the weld zone being a sharply transitioning zone of recrystallized grains on the advancing side and a rather diffuse region on the retreating side. An attempt has been made to understand how these interfacial or transition regimes accommodate solid-state flow of the welded materials.

3:00 PM
Microstructure Characteristics of Friction Stir Processed A1 7075 via Macroscopic Approach: *Bin Cai*[1]; Dongfang Huang[1]; Brent L. Adams[1]; Tracy W. Nelson[1]; [1]Brigham Young University, Mechl. Engrg., 435 CTB, Provo, UT 84602 USA

Friction stir welding (FSW) is evolving rapidly as a viable method for joining metals. Developing a deeper understanding of the affects of microstructure on post-stirring mechanical behavior is an important step towards controlling process parameters for optimal performance. However, the traditional vantage point of microscopic observation is on the transverse cross section which is incomplete. Here a new methodology, called the macroscopic or 3-dimensional approach, couples observations of microstructural characteristics with their corresponding geometrical locations. Data is recovered from the plan view in the microstructure, with spatial specificity in the transverse plane. Orientation imaging microscopy (OIM) is extensively employed; and the results via OIM are presented and discussed. Grain sizes at the center line decrease from top to bottom. Very fine grains were found in the weld nugget with prominent alignment. This observation indicates that grains in the nugget are not 3-dimensional equiaxial, but 2-dimensional.

3:20 PM Break

3:40 PM Invited
Analysis of Local Texture and Grain Boundary Structure in Friction Stir Welds of Magnesium Die Castings: *David P. Field*[1]; Naiyi Li[2]; [1]Washington State University, Mechl. & Matls. Engrg., Box 642920, Pullman, WA 99164-2920 USA; [2]Ford Motor Company, 2101 Village Rd., PO Box 2053, MD 3135, Dearborn, MI 48121 USA

Friction stir welding produces severe textural and microstructural gradients in metals. This structural inhomogeneity dominates local properties of the welds that are anisotropic in nature. Because of the limited number of slip systems available in Mg, these include virtually all mechanical and electrical properties of interest for use of a material in structural applications. Friction stir welds of AM60 Mg casting alloys were produced and structural gradients through the weld regions were characterized using electron backscatter diffraction, electron and optical microscopy. The gradients are shown to reflect the metal flow history occurred during welding to some extent. These inhomogeneous structures are discussed in relation to mechanical properties measured on both a local and bulk scale.

4:00 PM
TEM Analysis of Annealed and Thermo-Mechanically Processed Friction Stir Welds in Al-2195: Glen A. Stone[1]; *Anand B. Kaligotla*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA

The objective of this work was to investigate the underlying substructural cause of abnormal grain growth (AGG) resulting from the friction stir welding (FSW) of Al-2195. Preliminary investigation has shown that the AGG is eliminated when the FSW Al-2195 is cold-rolled to at least 20%. This investigation continues the earlier work by comparing the substructures of as-FSW, FSW and initiated, and the 20% cold-rolled samples. Factors of interest included dislocation densities, second phase particles, grain substructure, and grain orientation. Samples for analysis were cut parallel to the rolling direction.

4:20 PM
Effect of FSW Parameters on the Evolution of Swirl Zone Grain Structure During Post-Weld Heat Treatment of Commercial Purity Aluminum: *Bala Radhakrishnan*[1]; Gorti Sarma[1]; Oleg Barabash[1]; Zhili Feng[1]; Stan A. David[1]; [1]Oak Ridge National Laboratory, Computer Sci. & Math., Bldg. 5600, MS 6008, Oak Ridge, TN 37831-6008 USA

The deformation substructures in the swirl zone of commercial purity aluminum as a function welding parameters are analyzed using orientation imaging microscopy to determine the microtexture, grain size distribution and grain misorientation distribution. The orientation scan data consisting of the Euler angle set for each site are used as input to a mesoscale substructure evolution model based on a Monte Carlo technique. The relative subgrain energy and subgrain mobility are expressed as a function of the boundary misorientation. The Monte Carlo simulations are used to predict whether normal or abnormal grin growth will occur during post-weld heat treatment of the substructure. The model predictions are compared with experimental measurements of grain structures obtained after annealing.

4:40 PM
Effect of Process Parameters on Mechanical Properties and Microstructure in Friction Stir Welded Thin Sheet 2024-T3: *Alpesh Khushalchand Shukla*[1]; William A. Baeslack[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., 110 8th St., Troy, NY 12180 USA; [2]Ohio State University, Coll. of Engrg., 142 Hitchcock Hall, 2070 Neil Ave., Columbus, OH 43210-1275 USA

Optimum welding parameters, viz. tool rotation, welding speed, plunge depth and tool design are established for friction stir welding 1mm thick 2024-T3 aluminum alloy by using design of experiments and the effect of these parameters on mechanical properties (hardness and tensile strength) and microstructure is studied. Hardness profiles indicate presence of two minima, one at the TMAZ/HAZ boundary and the other farther away in the HAZ. Average hardness at the TMAZ/HAZ boundary decreases with increase in heat input. Various regions of the welds are characterized by using Transmission Electron Microscopy and the presence, density and coarsening of S type precipitates are related to the changes in hardness across the weld. The hardness minima at the TMAZ/HAZ boundary and farther in the HAZ are found due to presence of overaged S precipitates and dissolution of GPB zones respectively. The hardness peak in the HAZ is found to be due to the presence of GBPII zones.

5:00 PM
Root Flaws of Friction Stir Welds - An Electron Microscopy Study: *Tamara Vugrin*[1]; Martin Schmücker[1]; Günter Staniek[1]; Claudio Dalle Donne[1]; [1]German Aerospace Center, Mech. of Matls. & Joining Tech., Linder Höhe, 51145 Köln Germany

Friction stir welding usually is a stable process that only sporadically produces flaws. In this work flaws in the root region of the weld were examined. These root flaws cause a only partial bonding of the welded material. The origin of the root flaws is not fully understood yet, but it has often been associated with oxides, that are always present on aluminium surfaces. In the present work the root flaws in aluminium friction stir welds were created intentionally by a preoxidation treatment. The surface oxide layer on the aluminium alloys was examined prior to friction stir welding by transmission electron microscopy. The friction stir welds were inspected non destructively by ultrasound. The root flaws were examined by optical-, scanning electron- and transmission electron microscopy. It was possible to trace the oxides that are forming the root flaws.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Advanced Technology and Applications II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Wednesday PM Room: 3020
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Nuggehalli (Ravi) M. Ravindra, New Jersey Institute of Technology, Physics, Newark, NJ 07102 USA; John T. Prater, Army Research Office, Research Triangle Park, NC 27709-2211 USA

2:00 PM Invited
Analysis of Defects and Interfaces with Single Atom Sensitivity Through Aberration-Corrected Scanning Transmission Electron Microscopy: *Stephen J. Pennycook*[1]; Matthew F. Chisholm[1]; Andrew R. Lupini[1]; Albina Y. Borisevich[1]; Maria Varela[1]; Yiping Peng[1]; Klaus Van Benthem[1]; Naoya Shibata[2]; [1]Oak Ridge National Laboratory, Condensed Matter Scis. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6030 USA; [2]Universtiy of Tokyo, Sch. of Engrg., 2-11-16, Yayoi, Bunkyo-ku, Tokyo Japan

The ability to correct the major aberrations of the electron microscope is bringing enormous improvements not only in resolution but also in sensitivity. We have recently achieved a resolution of 0.06 nm in a Z-contrast image using a VG Microscopes 300 kV scanning transmission electron microscope (STEM) equipped with a Nion aberration corrector. The small beam size allows oxygen columns to be imaged within perovskite materials, and individual high atomic number dopant or impurity atoms to be imaged on and within specific columns of a crystal. In addition, a simultaneous, aberration corrected, conventional phase contrast image is available which shows oxygen columns with high contrast. Local electronic structure can be studied with electron energy loss spectroscopy, and single atom spectroscopy has been recently been achieved. Applications will be presented on the study of defects and interfaces in perovskite-based oxide materials, structural ceramics, nanomaterials and catalysts, showing how single atom sensitivity and the ability to image dislocation core structures leads to new insights into structure-property relations and has solved some longstanding problems. A previously unanticipated advantage of aberration correction is the reduced depth of focus. It is now possible to perform optical sectioning in the STEM simply be changing the focus of the beam. Single Hf atoms have been imaged in 3D in a Si/SiO2/HfO2 gate dielectric structure; they are not attached to the Si implying good passivation may be possible. In future generations of aberration-corrected STEM, 3D atomic resolution imaging may become routine.

2:30 PM Invited
Bonding Parameters of Cu Wafer Bonding for 3D Integration: *K. N. Chen*[1]; A. Fan[1]; C. S. Tan[1]; R. Reif[1]; [1]Massachusetts Institute of Technology, Microsys. Tech. Labs., 60 Vassar St., Rm. 39-623, Cambridge, MA 02139 USA

A reliable copper wafer bonding process condition, which provides strong bond at low bonding temperature with a short bonding duration, and does not affect the device structure, is desirable for future three-dimensional (3-D) applications. In this work, the effects of different process parameters on the quality of copper wafer bonding are summarized. Various parameters such as bonding pressure and Cu film thickness were studied to investigate the bonding strength. These bonding results were compared with other reported data. Thus, an overall view of Cu wafer bonding for different bonding parameters, including pressure, temperature, duration, copper thickness, clean techniques and anneal option, can be established. In addition, by meeting the process requirement of future IC process, the best bonding condition for three-dimensional (3-D) integration can be decided.

3:00 PM
Nanoengineering Approaches to Self-Assembled InAs Quantum Dot Laser Medium: *Serge Oktyabrsky*[1]; Vadim Tokranov[1]; Mathew Lamberti[1]; Gabriel Agnello[1]; Jobert Van Eisden[1]; Michael Yakimov[1]; [1]University at Albany - SUNY, Coll. of Nanoscale Sci. & Engrg., 251 Fuller Rd., Albany, NY 12203 USA

Interconnect 'bottleneck' in emerging IC's generated a need for alternative signal transmission solutions, such as optical technologies, in chip-level applications. To enhance performance parameters of chip-level active III-V photonic components, several emerging technologies are being developed: shape-engineered InAs quantum dot (QD) laser gain medium, digital alloys for precise control of semiconductor properties, novel "oxidation lift-off technology" for transfer of an optical device layer onto Si substrate. Technological issues and physics of self-assembled QDs with high gain, strong electron-hole coupling and high uniformity will be discussed. Beneficial properties of the developed QD medium are demonstrated by evaluation of laser diodes with unsurpassed thermal stability with a characteristic temperature of 380 K, high modal gain up to 31 cm^{-1} and by implementation of all-epitaxial QD vertical cavity laser. The QD structures are shown to withstand over two orders of magnitude higher defect density than quantum wells typically used in lasers.

3:30 PM Break

3:45 PM
Nanotechnology Related Materials Processing and Future Generation of Computing Systems: *Rajendra Singh*[1]; [1]Clemson University, Ctr. for Silicon Nanoelect. & Holcombe, Dept. of Electl. & Computer Engrg., Clemson, SC 29634 USA

The term nanotechnology has different meanings and expectations to different peoples. In this paper we have defined and examined what really is nanotechnology. Based on the use of fundamental knowledge of science and system level engineering and business knowledge that we know as of today, the role of materials processing of nanotechnology in the development of future generations of computing systems is described. In terms of materials processing techniques, the "top down" approach is already being used in the manufacturing of 90 nm feature size computing systems. The most advanced photomask manufacturing today is already exploiting the ability to manipulate material at atomic level and generate virtually defect free photomasks. However, there is no "bottom up" technique invented as of today that can be used directly in the manufacturing of future generation of computing systems. Future nanotechnology research should be directed in the directions that can lead to materials with very low defect random and systematic defects with lower manufacturing cost that is possible in current methods.

4:15 PM
Pulsed Laser Deposition for Novel Materials: Theory and Experiment: *Rajiv K. Singh*[1]; [1]University of Texas, Microelect. Rsch. Ctr., Austin, TX 78758 USA

Ever since the first successful application of the pulsed laser ablation technique in the deposition of high Tc superconductors in 1987, this method has become the technique of choice for the deposition of other materials such as oxide semiconductors, ferroelectrics, phosphors, colossal magnetoresistance (CMR), high dielectric constant and energy storage compounds. More recently, this method has been found to have excellent potential for the deposition of polymeric thin films for novel applications such a targeted drug delivery systems. The key factors which have led to the widespread use is its ability to maintain chemical stoichiometry, layer by layer thickness control, and relative simplicity of this method. The non-equilibrium nature of species gen-

erated during the laser irradiation process also plays a significant role in low temperature deposition and growth. This talk will focus on the unique fundamental aspects of the PLD process and its applicability to the next generation of oxide thin films and nanoscale materials.

4:45 PM
Recent Trends in Thin Films and Nanostructured Materials: *Ashutosh Tiwari*[1]; [1]North Carolina State University, Matls. Sci. & Engrg., Raleigh, NC 27695-7916 USA

In this talk we will present a brief overview of various research activities and projects currently undergoing in NSF Center of Advanced Materials & Smart Structures (NSF-CAMSS) at North Carolina State University under the director-ship of Prof. Jagdish Naryan. Before starting this talk I would first like to take an opportunity to say few words about Prof. Narayan. In his famous lecture William Arthur Ward said "The mediocre teacher tells. The good teacher explains. The superior teacher demonstrates. The great teacher inspires." Professor narayan belongs to the last category, he indeed inspires. I have had an opportunity to work under his supervision for several years, first as a research associate and then as a senior researcher. I have closely watched his dedication for research, extraordinary scientific vision and a desire to provide exceptional leadership to scientific community. Prof. Narayan has mentored hundreds of students, postdoctoral fellows and scientists, who are spread all over the world and are providing their valuable contribution to science. Prof Jagdish Narayan, known as "Jay" in scientific community, is an exceptional educationalist, outstanding researcher and visionary mentor. In his more than a quarter century long scientific career, he has made numerous outstanding contributions to almost every branch of Materials Science, Physics and Engineering. Prof Narayan has more than 800 papers and patents to his credit and he has written 12 books. His recent inventions in the field of nanostructured materials and Domain matching epitaxy-a new paradigm are attracting tremendous amount of scientific and technological attention. Currently under his direction we are working on several topics. However, the major focus of our research at NSF-CAMSS is on the novel methods of thin film processing, atomic scale structural characterization, physical property measurements and structure property correlation. We study variety of advanced materials and structures including nanostructured magnetic materials, thin film sensors, optically active wide bandgap materials, diluted magnetic semiconductors, perovskite oxides, ultraviolet/infrared detectors, novel diffusion barriers, magnetic superlattices exhibiting interlayer exchange coupling and low-field-magnetoresistance. Recently we have invented a new superlattice structure which exhibits highest magnetoresistance ever observed in any magnetic systems at low magnetic fields. I will discuss all of above mentioned topics in detail and at the end of the talk I will show some of our groundbreaking results related to nanostructured materials for which we have been granted the most recent United States Patent. Ref: J. Narayan and Ashutosh Tiwari "Methods of Forming Three-Dimensional Nanodot Arrays in a Matrix" United States Patent (June 24, 2004).

5:15 PM
Macro Self-Assembly Techniques: *Sudhakar Shet*[1]; Vishal R. Mehta[1]; Anthony T. Fiory[1]; Martin P. Lepselter[2]; Nuggehalli M. Ravindra[1]; [1]New Jersey Institute of Technology, Dept. of Physics, 161 Warren St., Univ. Hgts., Newark, NJ 07102 USA; [2]BTL Fellows Inc., 25 Sweetbriar RD., Summit, NJ 07901 USA

A review of various macro self-assembly techniques for integration of components to create powerful and complex microsystems is presented here. The current status, comparisons and limitations of the approaches to self-assembly methods are described. Applications of macro self-assembly techniques in defense, bio and aerospace industries are summarized.

5:45 PM
Bio-Inspired Designing and Fabrication of Advanced Coating Using Nanomaterials: *Wenping Jiang*[1]; Ajay P. Malshe[1]; [1]University of Arkansas, MMRL, Dept. of Mechl. Engrg., Fayetteville, AR 72701 USA

Nature is the master of organization as evidenced by the complex designs easily seen on the simplest structures and systems with priceless functionality, i.e., the unique nano materials and micro structures giving rise to the color of butterfly wings, super hydrophobic character to lotus leaf and tireless motion to flagella motors in E. Coli cells. The subject addressed in this manuscript is the realization of such structures and systems paralleling to Nature by understanding and controlling the size, shape, and position of nano materials based micro structures, while learning how to tailor nanomaterials with specific performance. We have used electrostatic spray process for self-assembly of nanoparticles to demonstrate surface morphology resembling lotus leaf. This morphology along with combination of hard and soft material phases make such coating an idea candidate for wear and advanced tribological applications.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Mechanical Properties of Lead-Free Solder Alloys and Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Wednesday PM Room: 3014
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Nik Chawla, Arizona State University, Dept. of Cheml. & Matls. Engrg., Tempe, AZ 85287-6006 USA; Paul T. Vianco, Sandia National Laboratories, Albuquerque, NM 87185-0889 USA

2:00 PM Invited
Creep Behavior of Sn-Rich Solders at Bulk and Small-Length Scales: *Nik Chawla*[1]; Rajen S. Sidhu[1]; Matthew Kerr[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA

The creep behavior and microstructure of bulk solder and solder spheres will be reviewed in this talk. Results on Sn-Cu, Sn-Ag, Sn-Ag-Cu, and pure Sn, will be presented. It will be shown that the creep behavior of Sn-rich solders is very much dependent on: (a) nature and interface between second phase particles and the pure Sn- matrix, (b) Sn matrix microstructure, and (c) temperature. Changes in the creep stress exponent with increasing stress were observed and will be explained in terms of a threshold stress for creep. The activation energy for creep was also found to be temperature dependent. Scanning and transmission electron microcopy were used to characterize the as-reflowed microstructure, as well as to understand creep deformation mechanisms in the solder. Creep mechanisms will be presented and discussed in terms of the creep stress exponents and the solder microstructure.

2:30 PM
Creep Properties of Pb-Bearing and Pb-Free Composite Solder Joints Reinforced with Nano or Micron-Sized Ag Particles: *Fu Guo*[1]; Feng Tai[1]; Yanfu Yan[1]; Jianping Liu[1]; Yaowu Shi[1]; [1]Beijing University of Technology, Key Lab. of Advd. Functional Matls., Ministry of Educ. P.R.C., Coll. of Matls. Sci. & Engrg., 100 Ping Le Yuan, Chaoyang Dist., Beijing 100022 China

Solder alloys are typically subjected to harsh environments and are used at temperatures well above half of their melting points in degrees absolute. Microstructural evolution, recrystallization, superplasticity, creep, and creep-fatigue are operative under normal service conditions, among which creep is the most common and important micromechnical deformation phenomena. Composite approach has been used in an effort to improve the service performance of solder joint including service temperature capability through enhanced creep properties. Systematic creep study was carried out on Pb-bearing (Sn-37Pb) and Pb-free (Sn-3.5Ag and Sn-0.7Cu) solder joints reinforced with nano or micron-sized Ag particles at different temperature and stress levels. Various creep parameters and deformation mechanisms were overviewed and compared with non-composite solder joints. The effects of particle size on the creep properties of different base solder joint materials were also analyzed and extensively discussed.

2:50 PM
Using Different Test Techniques to Investigate the Bond Strength of Cu Wafer Bonding: *K. N. Chen*[1]; S. M. Chang[2]; L. C. Shen[2]; R. Reif[1]; [1]Massachusetts Institute of Technology, Microsys. Tech. Labs., 60 Vassar St., Rm. 39-623, Cambridge, MA 02139 USA; [2]Industrial Technology Research Institute, Elect. Rsch. & Service Organization, Pkgg. Process Tech. Div., APC, Chutung, Hsinchu 310 Taiwan

Direct Cu-to-Cu wafer bonding has been proposed as an attractive method for various applications such as three-dimensional (3-D) IC and package, opto-electronic integration, and micro-electro-mechanical system (MEMS). In order to successfully apply Cu-Cu bonding to these applications, strong bonding strength to support the structure and low bonding temperature to avoid the destruction of the device structure are both crucial. In this paper, to understand more details of the bonding strength of Cu wafer bonding with respect to different bonding temperature and bonding duration, both quantitative and qualitative approaches are proposed, respectively. These investigations include the tensile (or pull) test, the tape test, and the mechanical dicing test.

3:10 PM
High Temperature Hardness of Lead Free Solder: *Joo Won Lee*[1]; Zin H. Lee[1]; Hyuck M. Lee[1]; Sung K. Kang[2]; D. Y. Shih[2]; Paul Lauro[2]; [1]KAIST, Dept. of Matls. Sci. & Engrg., 373-1 Guseong-dong, Youseong-gu, Daejeon 305-338 Korea; [2]IBM T.J. Watson Reaserch Center, Yorktown Hgts., NY 10598 USA

Solder joints are occasionally operated at an elevated temperature in service. They also experience plastic deformation caused by temperature excursion and differences in thermal expansion coefficient. Deformed solders can go through a recovery and recrystallization process at an elevated temperature, which would alter their microstructure and mechanical properties. In this study, to predict the changes in mechanical properties of Pb-free solder joints at high temperatures, the micro-hardness of several Pb-free solders and a composite solder was measured as a function of temperature, deformation, and annealing condition. Solder alloys investigated include pure Sn, Sn-0.7%Cu, Sn-3.5%Ag, Sn-3.8%Ag-0.7%Cu, and Sn-Ag-Cu-Ni (composite). Solder pellets were cast at two cooling rates (0.4 and 7°C/s). The pellets were compressively deformed by 30% and annealed at 150°C for 2 days. The micro-hardness was measured as a function of temperature from 25 to 130°C.

3:30 PM Break

3:40 PM
Isothermal Fatigue of 95.5Sn-3.9Ag-0.6Cu Pb-Free Solder: *Paul T. Vianco*[1]; Mark Grazier[1]; Robert Wright[1]; Eric J. Cotts[2]; [1]Sandia National Laboratories, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA; [2]SUNY Binghamton, Physics Dept, PO Box 6016, Binghamton, NY 13902-6016 USA

Isothermal fatigue behavior was investigated of the 95.5Sn-3.9Ag-0.6Cu (wt.%) solder, using shear strain controlled tests on 100 micron solder joints. The samples were tested at 25°C, 100°C, and 160°C. The shear strain ranges were 2.5, 5.0 and 10%. A six minute hold time was introduced at the stain limits and the extent of stress relaxation was documented. The apparent shear moduli were monitored at the loading and unloading terminal points of the cycle. Load drop was and strain energy (area in the hysteresis loop) were computed at benchmarked cycles. Fatigue crack growth occurred along the latter grain boundaries. In some cases, a path was generated in-situ for crack propagation by localized recrystallization of the Sn-rich matrix. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

4:00 PM
Bending Fatigue Test as a Reliability Evaluation Method for Solder Joints: *Choong-Un Kim*[1]; Jae-Yong Park[1]; Rajendra Kabade[1]; Viswanadam Puligandla[2]; Ted Carper[2]; [1]University of Texas, Matls. Sci. & Engrg., Arlington, TX 76019 USA; [2]Nokia Mobile Phone, Inc., Irving, TX USA

Concerns on the mechanical impact on the chip and package are growing as trends in the electronic industry are heading toward mobile environment. Frequent key pressing, bending due to the bolt fastening, and unexpected drop of the entire device can cause immediate and irrecoverable damage to the device. Consequently, reliability of the electronic device due to the mechanical impact is becoming critical and various test methods such as vibration, bending, and drop test are gaining importance. Among them, bending fatigue test is a promising candidate which provides controlled test environment and also enables us to extract meaningful material properties. This paper introduces the bending fatigue test as a test methodology of solder joint reliability against cyclic mechanical stress. Using a simplified package design for an idealistic test environment, the effectiveness of this methodology and considerations are discussed through the failure analysis.

4:20 PM
Optimization of Pb-Free Ball Grid Array Solder Sphere Reliability: *Daniel Cavasin*[1]; Arthur Huang[2]; [1]Advanced Micro Devices, PCSG, 5204 E. Ben White Blvd., MS: PCS-3, Austin, TX 78741 USA; [2]Advanced Semiconductor Engineering, Inc, 26 Chin 3rd Rd., Kaohsiung 811 Taiwan

A series of statistically-designed experiments were conducted to maximize the long-term reliability of Pb-free Plastic Ball Grid Array (PBGA) components. The primary focus of the experimentation was optimization of the solder sphere/substrate joint integrity. Both Sn-3.5Ag and Sn-4.0Ag-0.5Cu solder alloys were investigated, using substrates with electrolytic Ni/autocatalytic Au plated pads. Components were subject to JEDEC moisture sensitivity level 3 (MSL3)/260C preconditioning, followed by high temperature storage and temperature cycling stresses. Simulated electrical test (test socket insertion/removal) was conducted at each phase of the experimentation, followed by 100% inspection of the solder spheres. Primary response variables included shear strength and presence/absence of solder spheres. Results indicated that the thickness of the plated Au layer on the substrate surface played a key role in determining the components' ability to survive reliability stresses. The formation of complex ternary and quaternary intermetallic compounds in the area of the joint interface was characterized, and the package susceptibility to solder sphere separation due to brittle fracture was defined as a function of process conditions and materials.

4:40 PM
Effects of Strain Rates and Bi-Axial Stress Conditions on Plastic Yielding and Flow Stress of Sn3.8Ag0.7Cu Lead-Free Alloy: *Jin Liang*[1]; Nader Dariavach[1]; Gordon Barr[1]; [1]EMC, 176 South St., Hopkinton, MA 01748 USA

This study systemically investigates the rate-dependent mechanical properties of Sn3.8Ag0.7Cu lead-free alloy and Sn-Pb eutectic alloy under pure shearing and bi-axial stress conditions with thin-walled specimens using a servo-controlled tension-torsion material testing system. The pure shearing tests were conducted at strain rates between 10-6 /sec to 10-1 /sec. In additions, axial tensile and compressive stresses were superimposed onto the shearing samples to examine the effects of bi-axial stress conditions on the yielding and on post-yielding plastic flow of the solder alloys. Strain hardening is observed for the lead-free alloy under all the tested strain rates, while strain softening happens with Sn-Pb eutectic solder under low to moderate strain rates. Special tests were also conducted for sudden strain rates change, load drop and stress relaxation for the purpose to develop a visco-plasticity model to simulate time-depended multi-axial deformation and for damage and fatigue life prediction of real world solder interconnections.

5:00 PM
Evaluation of Solder-to-Chip Attachment, Wafer-Level Bumping Architectures: II. Performance in BHT, Cyclic Mechanical Bending and Temperature Cycling: Ian R. Harvey[1]; *Mark R. Larsen*[1]; David Turner[2]; Ian Doyle[3]; Jim Somers[3]; Jim Ortowski[4]; [1]University of Utah, Coll. of Engrg., 1495 E. 100 S., Kennecott Bldg., Rm. 101, Salt Lake City, UT 84112 USA; [2]Inovar, Inc., 1073 W. 1700 N., Logan, Utah 84321 USA; [3]Bourns Electronics, Microelect. Div., Mahon Industl. Park, Blackrock, Cork Ireland; [4]EDO Ceramics, 2645 S. 300 W., S. Salt Lake City, Utah 84115 USA

We have created a test chip to evaluate the relative tendency for alloy migration under BHT testing, as well as enable comparative thermomechanical performance and induced parasitic effects in standard TC testing and a customized cyclic bending fatigue test with a demonstrated history of reproducing cell phone field failure mechanisms. In this paper, we describe the BHT and failure analysis results indicating robust performance of the NEMI-std lead-free alloy, comparable to the eutectic alloy reference, and demonstrate thermomechanical reliability advantages of two system architectural design features, applicable to both eutectic and lead-free alloys: (1) use of narrow via architecture which acts as a compliant "cushion" under the bump, and (2) use of microvia-in-pad PCB designs.

5:20 PM
The Effect of Aging and Reflow on Shear Behavior of Sn-3.5Ag Lead Free Solder/Cu Joints: *R. S. Sidhu*[1]; X. Deng[1]; P. Johnson[1]; N. Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA

Mechanical behavior of lead free solder/Cu joints is sensitive to the reflow and aging process due to the relatively low melting tempera-

tures of solders and the high growth rate of intermetallics (Cu6Sn5 and Cu3Sn) that form between the solder and Cu substrate. In this study, the effects of reflow time, aging time, and aging temperature on the shear behavior of the Sn-3.5Ag solder/Cu system were investigated. Increasing reflow time changed the intermetallic thickness but not the solder microstructure, since the latter is controlled by cooling rate after reflow. Aging, on the other time, affected both the microstructure of solder and the thickness of the intermetallic layer. These important differences in microstructure significantly affected the strength dependence of the joints. The underlying fracture mechanisms will be discussed and coupled with microstructure-based finite element analysis.

Magnesium Technology 2005: Creep Resistant Magnesium Alloys and Welding-Joining

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Wednesday PM Room: 2004
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Mihriban O. Pekguleryuz, McGill University, Montreal, Quebec H3A 2B2 Canada; Zi Ki Liu, Pennsylvania State University, State College, PA 16802-5006 USA

2:00 PM
Friction Stir Spot Welding of Mg-Alloys for Automotive Applications: Peter Su[1]; *Adrian Gerlich*[1]; Tom North[1]; Gabor Bendzsak[1]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Wallberg Bldg., Toronto, Ontario M5S3E4 Canada

This paper presents the detailed produced during friction stir spot welding of AZ91D, AM60 and AZ31 Mg alloys. The features of the friction stir spot welding process were delineated using a combination of data acquisition outputs and metallographic examination of completed joints. The peak temperature attained during FSW spot welding of AZ91D, AM60 and AZ91 base materials was very close to the melting temperature of the Mg-Al solid solution/Al12Mg17 eutectic (437ºC), and higher than the reported incipient melting temperature (421°C). Evidence of local liquid film formation is shown in FSW spot welded AZ31 material. Factors determining the tensile shear loads of FSW spot welds in AZ91D and AM60 material were investigated. Higher failure loads during overlap shear testing properties were produced when the projected bonded area immediately adjacent to the keyhole and the amount of mechanical energy delivered during the FSW spot welding operation increased.

2:20 PM
Surface Contamination on Magnesium Ingots: *Anne Kvithyld*[1]; Magdalena Zadrozna[2]; Sean Gaal[1]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Alfred Getz vei 2, Trondheim 7491 Norway; [2]Warsaw University of Technology, Faculty of Matls. Sci. & Engrg., Woloska 141, Warsaw 02-507 Poland

Surface contamination present on magnesium ingots results in increased dross formation. The surface of the ingots primarily reacts with water and carbon dioxide, depending mainly on the humidity, temperature, cooling technique and the alloy of magnesium. Investigations were conducted on shavings from magnesium ingots. Impurities on the surface were studied in a thermo-gravimetric/differential thermal analysis (TG/DTA) furnace, and the evolved gases were measured using quadrupole mass spectrometer (MS). Mass loss curves show two peaks, at low and high temperatures, for pure Mg and AZ91D alloy. For AM20HR alloy only one peak at the higher temperature appears. The lowest total mass loss is obtained for AM20HR, while the highest was measured for pure Mg. The intensity of mass 44 was measured for carbon dioxide. This allowed the mass of the carbon dioxide produced during heating of the magnesium to be calculated, and by difference the quantity of water to be determined.

2:40 PM
Metallurgical Background to the Development of Creep Resistant Gravity Casting Alloys: *Boris Bronfin*[1]; Mark Katsir[1]; Oren Bar-Yosef[1]; Florian Moll[2]; Soehnke Schumann[2]; [1]Dead Sea Magnesium Ltd, Rsch. Div., PO Box 1195, Beer-Sheva 84111 Israel; [2]Volkswagen AG, Rsch., PO Box 1511, Wolfsburg D-38436 Germany

Magnesium alloys being the lightest structural materials, are very attractive in automotive and aerospace industries. New alloys are required that would resist the increasingly onerous operating environment and that would provide more complex components with increased lifetime and reduced maintenance cost. The desire to achive weight reduction of large powertrain components used by the automotive and aerospace industries has resulted in the development of new gravity casting alloys that provide a good combination of service properties and cost. The present paper addresses physical metallurgy principles that enabled to develop new alloys designated MRI201S, MRI202S and MRI203S. New alloys are designed for applications up to 250-300°C and have excelent mechanical properties, corrosion behavior and creep resistance in T6 condition. In addition, the paper will also highlight some practical applications that illustrate the capabilities of newly developed alloys.

3:00 PM
Solid Solution Effects on the Tensile Behaviour of Concentrated Mg-Zn Alloys: *Andrew Blake*[1]; Carlos H. Caceres[1]; [1]University of Queensland, Matls. Engrg., Sch. of Engrg., Brisbane, QLD 4072 Australia

Previous work has suggested that short-range order is responsible for the strengthening in concentrated Mg-Zn alloys. This added strengthening, above the random solid solution hardening observed in dilute alloys, is expected to affect the tensile behaviour of the concentrated alloys through its effects on secondary (prismatic and pyramidal) slip. Solid solution effects on the flow stress, strain hardening rate and ductility of polycrystalline magnesium zinc alloys, with zinc contents between 0 and 3 at.% have been studied. A constant grain size was obtained in all alloys by adding a small amount of Zr. The results are discussed in terms of possible solid solution softening and hardening effects on the different slip systems.

3:20 PM Break

3:35 PM
On the Microstructure and Properties of Friction Stir Welds in AZ31 and AZ61 Alloys: *Jorge F. dos Santos*[1]; Antonio C. Blanco[1]; Rudolf Zettler[1]; Surendar K. Marya[2]; [1]GKSS Forschungszentrum, Inst. for Matls. Rsch., Max-Planck-Str. 1, Joining Tech., Geesthacht D-21502 Germany; [2]Ecole Central Nantes, Welding Joining & Forming Processes, Nantes France

The increased use of Mg in the car manufacturing industry has raised questions on weldability aspects of Mg and its alloys. The Friction Stir Welding (FSW) has the advantage of achieving metallic bonding below the melting point of the base material avoiding metallurgical problems associated with the solidification process. The present study presents the results of a development programme carried out to investigate the response of alloys AZ31 and AZ61 to different FSW tool geometries and process parameters. Temperature development across the weld zone was monitored and the produced welds have been subjected to microstructural analysis and mechanical testing. Defect free welds have been produced with the optimised FSW-tool and parameters. The microstructure of the welded joint resulted in similar ductility and hardness levels as compared to the base material. The results also showed that tool geometry plays a fundamental role on the response of the investigated alloys of the FSW process.

3:55 PM
Effects of Alloying Element on Microstructure and High-Temperature Mechanical Properties of Mg-Al Alloys: *Kwang Seon Shin*[1]; Yeon Jun Chung[1]; [1]Seoul National University, Sch. of Matls. Sci. & Engrg., San 56-1 Shinrim-dong Kwanak-gu, Seoul 151-742 Korea

Magnesium alloys exhibit excellent properties such as high specific strength at room temperature, good damping characteristics and castability. Mg-Al alloys such as AM50, AM60 and AZ91 have been used extensively since these alloys exhibit superior die castability and a good balance of strength and ductility. However, the application temperature of these alloys is generally limited to about 100°C, above which a rapid degradation in mechanical properties was observed due to the presence of Mg17Al12. The poor high-temperature mechanical properties of the commercially available magnesium alloys have prevented their applications at elevated temperatures. In the present study, the effects of alloying elements on microstructure and high-temperature mechanical properties of Mg-Al alloys were examined. The specimens used in this study were die-cast on a 320ton cold chamber high-pressure die casting (HPDC) machine. Tensile and creep tests were performed and the microstructure was examined. It was found that the addition of alloying elements modified the precipitation behavior and improved the high-temperature mechanical properties of Mg-Al alloys.

4:15 PM
DaimlerChrysler Corporation High Temperature (Creep Resistant) Magnesium Alloy Development: *Randy S. Beals*[1]; [1]DaimlerChrysler Corp., 800 Chrysler Dr., Auburn Hills, MI 48326 USA

In the past few years several new magnesium alloys for high temperature applications have been developed. These alloys are typically based on rare earth and alkaline earth element additions to magnesium. Unfortunately, it is very difficult to achieve an adequate combination of properties (die castability, creep resistance, mechanical properties, fatigue resistance, corrosion performance and affordable cost) that allows an alloy to have cost effective performance. In previous studies it has been reported that current alloy systems either achieve a high level of creep resistance or acceptable die castability but never both. This study describes a research and development effort by DaimlerChrysler Corporation's Materials Engineering to arrive at a magnesium alloying system that delivers excellent creep resistance as well as excellent die castability at an affordable cost. This study compares the new alloy with an overview of the various magnesium alloy systems for use in elevated temperature applications (150-175°C).

4:35 PM
Creep Studies of MRI153 Magnesium Alloy Castings: *S. M. Zhu*[1]; B. L. Mordike[2]; J. F. Nie[1]; [1]Monash University, Sch. of Physics & Matls. Engrg., Victoria 3800 Australia; [2]Technical University of Clausthal, Dept. of Matls. Engrg. & Tech., Agricolastrasse 6, D-38678, Clausthal-Zellerfeld Germany

The microstructures and creep properties of three MRI153 alloy castings, die casting, squeeze casting and ingot casting, have been investigated and compared. The stress and temperature dependence of the minimum creep rate suggested that similar deformation mechanisms hold for the three castings. The stress dependence exhibited a transition from power-law creep at low stresses to power-law breakdown at high stresses. In the power-law regime, stress exponents of ~6 and activation energies close to that for lattice self-diffusion in magnesium were obtained. The squeeze-cast alloy showed the highest creep resistance in terms of minimum creep rate or rupture time, while the poorest creep resistance was observed in the die-cast alloy. The creep resistance of the three castings are discussed in terms of grain size, casting porosity and degree of solute supersaturation.

4:55 PM
An Electron Microscope Study of Intermetallic Phases in AZ91 Alloy Variants: *Takanori Sato*[1]; Barry L. Mordike[2]; Jian-Feng Nie[3]; Milo V. Kral[1]; [1]University of Canterbury, Mechl. Engrg., PO Box 4800, Christchurch New Zealand; [2]TU Clausthal, Inst. für Werkstoffkunde & Werkstoftechnik, Agricolastrasse 6, D-38678, Clausthal-Zellerfeld Germany; [3]Monash University, Sch. of Physics & Matls. Engrg., PO Box 69M, Clayton, Victoria 3800 Australia

The magnesium alloy AZ91 has been employed extensively due to its superior castability and mechanical properties at room temperature. New alloys based on AZ91 have been developed to improve creep properties by forming grain boundary and intragranular intermetallic phases via additions of elements such as Ca and Sr. Intermetallic phase stability and distribution are not well understood in these new alloys. In the present work, the various intermetallic phases in two high pressure die cast AZ91-type alloys were characterized using a combination of transmission electron microscopy, selected area and microbeam electron diffraction, scanning electron microscopy, electron backscatter diffraction pattern analysis and energy dispersive x-ray spectroscopy. The crystallographic identification, relative amounts and distributions of phases will be presented.

5:15 PM
Nd:YAG Laser Welding of Magnesium Alloy Castings: *Xinjin Cao*[1]; Min Xiao[1]; Mohammad Jahazi[1]; [1]Institute for Aerospace Research, Aeros. Mfg. Tech. Ctr., 5145 Decelles Ave., Montreal, Quebec H3T 2B2 Canada

Laser welding will become an important joining technique for magnesium alloys with their increasing applications in aerospace, aircraft, automotive, electronics and other industries due to low heat input, small heat affected zone, narrow fusion zone, high welding speed, low residual stress and distortion. The ongoing project on laser welding investigates the laser weldability of cast magnesium alloys and aims to develop a reliable welding and repair process for sand castings. This presentation will report on the progress made in the Aerospace Manufacturing Technology Center of the NRC Institute for Aerospace Research, in studies involving laser welding of Mg-4.2Zn-1.2Ce-0.7Zr (ZE41A-T5) alloy butt joints. The influence of some important processing parameters such as surface condition, defocusing distance, laser power, welding speed, joint gap and filler metal on the welding mode, macrostructure, microstructure, defect and mechanical property of the weld joints will be presented and discussed.

Materials Issues for Advanced Nuclear Systems: Materials for Nuclear Waste Storage

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Robert J. Hanrahan, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA

Wednesday PM Room: 3012
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Raul B. Rebak, Lawrence Livermore National Laboratory, Matls., Livermore, CA 94550 USA; Robert J. Hanrahan, Los Alamos National Laboratory, ADWP, Los Alamos, NM 87544 USA

2:00 PM
Localized Corrosion Behavior of Alloy 22 Solution Annealed Welded Plates: *Robert A. Etien*[1]; Steven R. Gordon[1]; Gabriel O. Ilevbare[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

Alloy 22 (N06022) may be used in industrial service in the as-welded condition, that is, without prior solution annealing. In some instances, welded Alloy 22 parts may be solution annealed to relieve residual tensile stresses introduced during the welding process. After annealing, it might be necessary to remove the oxide film formed during the heat treatment, depending on the application. Therefore, it is important to determine the corrosion resistance of Alloy 22 in the as-welded condition compared with the annealed condition with and without the annealed oxide film. The crevice corrosion resistance of Alloy 22 (N06022) in these three metallurgical conditions was measured using a number of electrochemical techniques. Studied variables included electrolyte composition (chloride and nitrate ratio), temperature and applied potential.

2:25 PM
Corrosion Behavior of Welded Plates of Alloy 22 With Varying Heat Composition: David V. Fix[1]; Kenneth J. King[1]; John C. Estill[1]; *Raul B. Rebak*[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

The ASTM standard B 575 provides the requirements for the chemical composition of Nickel-Chromium-Molybdenum (Ni-Cr-Mo) alloys such as Alloy 22 (N06022) and Alloy 686 (N06686). The compositions of each element are given in a range; for example, the content of Mo is specified from 12.5 to 14.5 weight percent for Alloy 22 and from 15.0 to 17.0 weight percent for Alloy 686. It was important to determine how the corrosion rate of welded plates of Alloy 22 using Alloy 686 weld filler metal would change if heats of these alloys were prepared using several variations in the composition of the elements even though still in the range specified in B 575. Especially prepared seven heats of plate were welded with seven heats of wire. Immersion corrosion tests were conducted in a boiling solution of sulfuric acid plus ferric sulfate (ASTM G 28 A). The study also covers the effect of solution annealing on the corrosion rate of welded plates.

2:50 PM
Stress Corrosion Cracking of Cladding Materials: Ajit K. Roy[1]; *Unnikrishnan Valliyil*[2]; *Elumalai Govindaraj*[2]; [1]University of Nevada, Dept. of Mechl. Engrg., 4505 Maryland Pkwy., Box 454009, Las Vegas, NV 89154-4009 USA; [2]University of Nevada, Dept of Mechl. Engrg., 4248 Claymont St., Apt #1, Las Vegas, NV 89119 USA

Cladding is the primary structural barrier that prevents the release of radionuclides contained in the spent nuclear fuel (SNF). SNF cladding used in most commercial reactors in the United States is made of zirconium (Zr) alloys such as Zircaloy-2 (Zr-2) and Zircaloy-4 (Zr-4). This paper is focused on the evaluation of stress corrosion cracking (SCC), hydrogen embrittlement (HE) and localized corrosion susceptibility of Zr-2 and Zr-4 in neutral and acidic solutions at 30, 60 and 90°C. Constant-load and slow-strain-rate (SSR) testing methods were used to evaluate the SCC and HE behavior of both alloys by using smooth and notched tensile specimens. Cyclic potentiodynamic polarization (CPP) technique was used to determine the localized corrosion susceptibility. The morphology of failure was analyzed by optical microscopy and scanning electron microscopy (SEM), respectively.

The SSR testing revealed enhanced ductility and reduced failure stresses with increasing temperature. Fractographic evaluations by SEM revealed dimpled microstructure.

3:15 PM Break

3:25 PM
Effect of Stress Mitigation on the Corrosion Behavior of Alloy 22: *Ahmet Yilmaz*[1]; David V. Fix[1]; John C. Estill[1]; Lana L. Wong[1]; Raul B. Rebak[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

Welding processes may introduce residual stresses in the parts that are joined together. A full solution annealing could eliminate these residual stresses. When annealing is not possible, stress minimization may be applied locally to reduce the level of tensile stresses in the outer layers. Tensile stress minimization techniques include low plasticity burnishing (LPB) and laser shock peening (LSP). It is important to determine if the applied stress mitigation process affects the corrosion resistance of the welded structure. Studies were carried out using welded plates of Alloy 22 (N06022). The corrosion behavior was compared using as welded plates with plates that were treated using LPB and LSP. Immersion tests were carried out in a boiling solution of sulfuric acid and ferric sulfate (ASTM G 28A). Also, cyclic potentiodynamic polarization tests (ASTM G 61) were used to determine the susceptibility to crevice corrosion in brines containing chloride and nitrate ions. Results show negligible effect of the surface treatment on the corrosion resistance of Alloy 22.

3:50 PM
Effect of Stress Mitigation on Precipitation Kinetics in Alloy 22 Welds: *Bassem S. El-Dasher*[1]; Sharon G. Torres[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

Understanding the phase stability of Alloy 22 (N06022) is important since the precipitation of tetrahedrally close-packed (TCP) phases over time has been known to adversely affect corrosion and mechanical properties. Prior observations have shown that these phases precipitate during the welding process. After welding, residual stresses due to the solidification and cooling from temperature remain. When the weld cannot be stress-relieved by solution annealing, the application of commercially available stress-mitigation processes such as low plasticity burnishing (LPB) and laser shock peening (LSP) may be used to produce near-surface compressive stresses. This study involved examination of cross-sectional samples of aged 1.25" thick welds of Alloy 22 plates using electron backscatter diffraction (EBSD) for TCP identification, and micrograph analysis for TCP quantification. Precipitation in the LSP treated weld was observed primarily in inter-dendritic regions, similar to that in the as-welded material. Precipitation in the LPB treated weld however was observed in both inter-dendritic as well as intra-dendritic regions.

4:15 PM
Effect of Heat Treated Oxide Film on the Corrosion Behavior of Ti Gr 7 in Fluoride Containing Solutions: *Tiangan Lian*[1]; Michael T. Whalen[1]; Lana L. Wong[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-631, Livermore, CA 94550 USA

Titanium Grade 7 (R52400) is a highly corrosion resistant alloy, mainly due to the spontaneous formation of a stable, protective and strongly adherent passive oxide film in presence of water and oxygen. One of the few environments that may hamper the stability of this oxide film are solutions containing fluoride ions. Welded components of titanium alloys may be used in the as fabricated conditions or either after stress relieving or solution annealing and air-cooling. The stress relief is generally performed at 1000°F (538°C) and the solution annealing at 1300°F (704°C), both for approximately 45 min. It was important to characterize the effect of the high temperature air formed oxide film on the corrosion resistance of R52400 in fluoride containing solutions and to compare the results with the behavior of non-heat treated material. Studied environmental variables included solution composition, temperature and applied potential.

Mechanical Behavior of Thin Films and Small Structures: Advanced Characterization Techniques

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Wednesday PM Room: 2024
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Andrew M. Minor, Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA 94720 USA; Kevin J. Hemker, Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA

2:00 PM Invited
Characterisation of the Mechanical Properties of MEMS Devices Using Nanoscale Techniques: *Nicholas X. Randall*[1]; [1]CSM Instruments Inc., 197 First Ave., Needham, MA 02494 USA

This presentation will focus on recent developments in the localised characterisation of the mechanical properties of Microsystems and MEMS devices and structures. Conventional indentation techniques provide a highly powerful method for measuring the load and depth response of bulk and coated materials, but can also be used to measure the mechanical properties of very small micro-machined silicon structures. Beam structures, such as are used for accelerometers, need to be characterised in terms of the number of cycles to failure, the spring constant or the energy required to bend the beam by a required amount. Such localised testing needs to be adapted to work at various distances from the origin of the beam with a positioning accuracy of less than a micron. Initial studies have proved to be highly repeatable. A range of examples is presented which covers a range of application areas, including accelerometer beam structures, microswitches and printer head structures. The basic instrumental concepts are explai ned together with the modifications required for testing small structures in a localised way. In addition, the localised testing of friction and wear in MEMS devices will be covered with some examples of the technology available and how it may be applied to such small contact areas in an accurate and reproducible way.

2:25 PM
In-Situ SEM Observations of Thermal Fatigue Damage Evolution in Cu Interconnects: Effect of Frequency and Overlayers: *Young-Bae Park*[1]; Reiner Moenig[2]; Cynthia A. Volkert[3]; Guangping Zhang[4]; [1]Andong National University, Sch. of Matl. Sci. & Engrg., Andong S. Korea; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA; [3]Institut fuer Materialforschung II, Forschungszentrum Karlsruhe, Karlsruhe Germany; [4]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsch., Shenyang China

It has recently been observed that severe fatigue damage is formed in Cu interconnects due to the cyclic temperatures generated by Joule heating of the metal lines by the passage of alternating currents (AC). However, the effect of the AC frequency on the damage evolution characteristics is not known so far. In this talk, we will summarize our in-situ SEM observations of damage formation during thermal fatigue of polycrystalline sputtered Cu lines (100 to 300 nm thick and 8 um wide) with Ta underlayer on Si. The exact nature of the damage evolution depends on the grain size and orientation, which will be discussed in detail. The higher loading frequency not only accelerated grain growth of damaged grains, but also led to earlier failure. Generally, smaller grained films exhibited longer lifetimes. Finally, it will be shown that the presence of a soft overlayer does not hinder damage formation and fatigue failure. These results imply that thermal fatigue may be a serious reliability threat to Cu interconnects with soft low-k interlevel dielectrics.

2:40 PM
The Nature of Contact Deformation of a Diamond-Like Carbon Coating on Stainless Steel Substrate: *Zonghan Xie*[1]; Mark Hoffman[1]; Singh Rajnish[1]; Avi Bendavid[2]; Phil Martin[2]; [1]University of New South Wales, Sch. of Matls. Sci. & Engrg., Kensington NSW 2052 Australia; [2]CSIRO Division of Telecommunications & Industrial Physics, Lindfield NSW 2070 Australia

Diamond-like carbon films have the unique mechanical properties of high hardness and low friction arising from their amorphous sp3/sp2 structure. For a number of years, these features have drawn considerable interest towards wear applications which seek enhanced surface hardness and abrasion resistance. An understanding of deformation mechanisms of diamond-like carbon coating on ductile substrate systems is therefore critical for design of these coatings to improving their service life. In this work, a diamond-like carbon film was coated onto a stainless steel substrate using a plasma assisted chemical vapour deposition (PACVD) technique. Nanoindentation was undertaken to deform the coating. The indented region was then studied using scanning and focused ion beam (FIB) microscopy of cross-section profiles. Formation and growth of ring cracks in the coating, as well as plastic flow in the ductile substrate, were found to be the predominant deformation processes. Delamination at the interface between the coatin g and substrate occurred upon unloading. No plastic deformation within the coating was observed. Coating deformation was, therefore, controlled by its fracture energy. An indentation-energy based model then allows deconvolution of the coating behavior from that of the substrate, enabling quantification of the fracture toughness of the coating.

2:55 PM Invited
Microsample Tensile Testing and Characterization of Nanocrystalline Aluminum Thin Films: *Kevin J. Hemker*[1]; Daniel Gianola[1]; Derek Warner[1]; En Ma[2]; Jean-Francois Molinari[1]; William N. Sharpe[1]; [1]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA; [2]Johns Hopkins University, Dept. of Matls. Sci. & Engrg., Baltimore, MD 21218 USA

The mechanical performance of nanocrystalline thin films and MEMS or NEMS materials has been shown to be very different from their bulk microcrystalline counterparts. It is widely recognized that reducing the grain size of a material to nanocrystalline dimensions will result in Hall-Petch strengthening and greatly increased strength and hardness. What is not currently well understood is how nanocrystalline metals accommodate plastic deformation. In the present study fully dense nanocrystalline structures with high angle grain boundaries were fabricated by pulse sputtering of submicron thin films. MEMS-inspired handling procedures and a custom designed load frame were developed to test these nanocrystalline thin films. This presentation will present results of thin film tensile experiments conducted to characterize the strength and in situ transient behavior of nanocrystalline thin films. The results of these experiments will be contrasted with TEM observations, rationalized with regard to possible deformation mechanisms and compared with quantitative atomistic/finite element models that allow for intergranular sliding and capture the composite-like nature of a nanocrystalline solid.

3:20 PM
Real-Time Observation of Plastic Yielding in Aluminum Films During Nanoindentation: *D. Ge*[1]; E. A. Stach[1]; A. M. Minor[1]; M. Jin[2]; J. W. Morris[2]; [1]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, MS 72, One Cyclotron Rd., Berkeley, CA 94720 USA; [2]University of California, Dept. of Matls. Sci. & Engrg., Berkeley, CA 94701 USA

An experimental study of the initial stages of plasticity in aluminum thin films has been conducted in order to understand the anomalous yielding behavior in instrumented nanoindentation experiments. Typical load-displacement curves from the nanoindentation of aluminum thin films shows a characteristic yielding composed of discrete displacement excursions separated by an elastic response. To clarify the origins of this unique phenomenon, the technique of in-situ nanoindentation in a transmission electron microscopy (TEM) has been applied to aluminum films of different thickness. Taking advantage of the real-time observation of the microstructural evolution in aluminum films under localized loading, the present study focuses on the effect of grain size, penetration depth and indentation position relative to the grain boundary. Possible mechanisms responsible for discrete plasticity have been rationalized based on the observations of dislocation activities. Additionally, ex-situ TEM characterization of nanoindentations are presented for comparison.

3:35 PM Break

3:50 PM Invited
Micro Instrumentation for Studying Mechanical Properties of Free Standing Films: Aman Haque[1]; *Taher Saif*[1]; [1]University of Illinois, Mechl. & Industl. Engrg., Urbana, IL 61801 USA

Investigation of the mechanical behavior of free standing thin films has always been challenged by instrumentation. Most frequently used methods employed to study thin films include nano indentation, wafer curvature, bulge test, and uniaxial tension, to name a few. Except for uniaxial tension, these methods need a material model to extract mechanical properties beyond elastic limit. Furthermore, none of the above methods allow in-situ observation of the microstructure of the specimen while its stress-strain relation is measured. We have developed a MEMS based micro-instrument that allows uniaxial tension of free standing thin films with thickness 30nm and higher. The method also allows in-situ observation of the micro structure so that the mechanism of deformation can be related to the macroscopic stress-strain response. Here, the sample and the measuring instrument are lithographically patterned and co-fabricated. Each instrument is dedicated to one sample. After test, the instrument is discarded. We demonstrate the method by testing 30-300nm thick free standing films of aluminum in SEM (scanning electron microscope) and TEM (transmission electron microscope). We find that as grain size decreases below 50nm, few dislocations exist in aluminum films. They do not generate under high stresses, and even when a crack grows through small grains and grain boundaries prior to failure. Furthermore, as grain size decreases, elastic modulus decreases by a small percentage, and aluminum shows non-linear elastic response with little plastic deformation. We postulate that soft grain boundaries and boundary generated defects are responsible for both reduced elastic modulus and non-linear elastic response.

4:15 PM
Nano- to Micro-Scale Influence of Coating Mechanical Properties Determined Using 3D Omniprobe: *Tathagata Mitra*[1]; Richard J. Nay[1]; Dehua Yang[1]; Thomas J. Wyrobek[1]; [1]Hysitron Inc., Nanomech. Rsch. Lab., 10025 Valley View Rd., Minneapolis, MN 55344 USA

Mechanical characterization of thick coatings presents challenges to nanoindentation due to the ranges of load and displacement it can offer. Additionally, conventional microindentation is unable to probe nanoscale properties of a material, which requires higher measuring and controlling sensitivity. Attempts to bridge nanoscale characterization to macroscale applications have resulted in the emergence of new instrumentation. In this study, a newly developed load and displacement sensing indenter, 3D Ominprobe, capable of both nano- and micro-scale indentation and scratch is introduced. Load- or displacement-controlled indentations were performed on thick DLC, ZrN, and AlTiN coatings. Results on elastic modulus and hardness show depth variation of the properties of the coatings from nano-scale to micro-scale using one instrument. Fracture toughness and indentation size effects are also investigated. Further, interfacial adhesion strength and tribological properties of the coatings are evaluated using the instrument's scratch capability. This study demonstrates the ability of the instrument to integrate nano- and micro-scale mechanical characterization.

4:30 PM
Three-Dimension Imaging of Deformation Modes in TiN-Based Thin Film Coatings: Lok Wang Ma[1]; Julie Marie Cairney[1]; *Paul Richard Munroe*[1]; Mark Hoffman[1]; [1]University of New South Wales, Matls. Sci. & Engrg., Sydney, NSW 2052 Australia

TiN-based thin film coatings are routinely applied to steel substrates to improve their wear-resistance. One method of assessing the deformation behaviour of these coatings is through nano-indentation. However, this method is limited as it provides little microstructural information about the deformation mechanisms that operate under stress. The authors have previously used focused ion beam milling to generate and observe cross-sections through such coatings, where features such as intergranular cracking, transgranular cracking and shear steps at the coating-substrate interface have been observed. However, this method is also limited as it provides data about the crack shape and path in only two dimensions. In this study, we have used a dual-beam focused ion beam miller to create three-dimensional sections of the deformation zones created during nano-indentation. The three-dimensional sections that will be presented provide highly detailed insights into the mechanical behaviour of these coatings.

4:45 PM Invited
X-Ray Diffraction of Small Volumes - Metal Plasticity Under Confinement: *Ralph Spolenak*[1]; [1]Swiss Federal Institute of Technol-

ogy, Dept. of Matls., Wolfgang-Pauli-Strasse 10, Zurich 8093 Switzerland

X-ray diffraction is an important method to study the mechanical properties of thin films and small structures. In this contribution two novel techniques, Laue microdiffraction and in situ diffraction of ultrathin films will be reviewed. Volumes as small as 1 cubic micron can be probed. The results shed new light on thin film metal plasticity on the nanoscale. Examples will be presented on poly- and single crystalline Au and Cu thin films in the thickness range from 1 micron down to 20 nm. The effect of external and internal interfaces on plasticity will be critically discussed.

5:10 PM
Acoustic Methods for the Measurement of the Elastic Properties of Films: Comparative Assessment of Surface Brillouin Scattering and Laser-Induced Ultrasonics: *Marco G. Beghi*[1]; Andrea C. Ferrari[2]; Dieter Schneider[3]; Pavel V. Zinin[4]; [1]Politecnico di Milano, Nucl. Engrg. Dept., Via Ponzio 34/3, Milano I-20133 Italy; [2]Cambridge University, Engrg. Dept., Cambridge UK; [3]Fraunhofer Institut, Werkstoff- und Strahltechnik, Dresden Germany; [4]University of Hawaii, Sch. of Ocean & Earth Sci. & Tech., Honolulu, HI USA

The measurement of the acoustic properties allows the precise determination of the elastic constants in thin films. The main non-destructive techniques using surface acoustic waves (SAWs) in different frequency ranges are Surface Brillouin Scattering (SBS) and laser-induced SAWs (LISAW). We present a critical assessment of their performances, testing them on different diamond-like carbon films of increasing thickness (from 2 nm to microns). Nanometer thick carbon films are used in magnetic hard disk coatings. Two sets of such films were measured by SBS and LISAW. The results of this round robin test show a good correlation, proving the ability of SAW techniques to assess the Young modulus in a thickness range not attainable by techniques such as nano-indentation. Diamond-like carbon films in the micrometer thickness range are widely used for protective coatings and show high promise as low stiction-high Young moduls material for micro electro mechanical systems. For these films the combination of SBS and LISAW achieves a better precision in the elastic properties measurements.

5:25 PM
3D Strain Mapping of Small Structures by Synchrotron X-Ray Microtomography: *JeongJu Ahn*[1]; Hiroyuki Toda[1]; Mitsuo Niinomi[1]; Toshiro Kobayashi[1]; Kentaro Uesugi[2]; Toshikazu Akahori[1]; [1]Toyohashi University of Technology, Dept. of Production Sys., Toyohashi, Aichi 441-8580 Japan; [2]Japan Synchrotron Radiation Research Institute, Sayo, Hyogo 679-5198 Japan

With miniaturization of the products, it is difficult or impossible to estimate the mechanical properties of the parts, which form the small products, using a conventional mechanical property assessment technique exactly and quantitatively. For adding the reliability to the mechanical properties of small structures, it needs to evaluate three-dimensional mechanical properties as well as 2D mechanical properties. Therefore, synchrotron X-ray microtomograhpy is applied to real-time measurement and evaluation of the 3D strain distribution of the miniature dog-bone sample of a pure Al with/without artificial circumference notch under the several stroke conditions at the BL47XU of the Spring 8, Japan. It is possible to evaluate 3D local strain mapping of the area or part which is considered to be important as well as whole 3D strain mapping in terms of the stroke using the miniature Al through analyzing 3D images reconstructed from X-ray images.

5:40 PM Closing Remarks: Summary

Metallurgical Technology for Waste Minimization: Session III

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Toru Okabe, University of Tokyo, Institute of Industrial Science, Tokyo Japan; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA

Wednesday PM Room: 2012
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Yoshio Nakano, Tokyo Institute of Technology Japan; Norihiro Murayama, Kansai University, Cheml. Engrg., Suita, Osaka 564-8680 Japan

2:00 PM Invited
Precipitation of Hematite from Metallurgical Processing Streams by Hydrolysis in the Presence of Anionic Resins: Salima Djaroudib[1]; *Joris Proost*[1]; André Van Lierde[1]; [1]Université Catholique de Louvain, Div. of Matls. & Process Engrg., Place Sainte-Barbe 2, Louvain-la-Neuve B-1348 Belgium

The precipitation of hematite (Fe2O3) from ferric chloride media at ambient pressure and at temperatures below 100°C was studied as part of a program to recover a marketable iron product form metallurgical processing streams and effluents. Hematite was formed in preference to ferric oxyhydroxides (e.g. ß- FeO-OH) by controlling the precipitation conditions, especially by adding Fe2O3 seeds. The extent of the hydrolysis reaction was found to depend primarily on temperature and on the free-acid concentration. As to the latter, the controlled addition of an adsorbant of free acid, in our case the anionic resin polyvinyl-pyridine (PVP), was shown to allow for the nearly complete elimination of iron as readily filterable Fe2O3. The hematite product typically contains > 63% Fe and < 1% Cl, and its composition does not change appreciably upon repeated cycling using regenerated PVP.

2:30 PM Invited
The Transformation of Crystal Structure of Calcium Aluminate Phase and Ettringite as the Solidification of Heavy Metals: *Ji-Whan Ahn*[1]; Kwang-Suk You[1]; Song-Min Chon[1]; [1]Korea Institute of Geoscience & Mineral Resources, Minls. & Matls. Procg. Div., Daejon 305-350 Korea

Tetracalcium sulphoaluminate clinker composed of CaO, Al2O3, SO3 has been synthesized using reagent of CaCO3, Al2O3 and CaSO4?2H2O. The effect of simultaneous addition of heavy metals (Cu, Cr, Pb, Zn) on varying temperature has been investigated. The synthesized solid solutions have been characterized through X-ray diffraction (XRD) and scanning electron microscope (SEM). And the vaporization of them during sintering process was measured by the liquid analysis of clinker. Through the measurements, the solidification and substitution of heavy metal on the crystal structure of tetracalcium sulphoaluminate and Ettringite, which is hydrates, could be evaluated. The morphology of calcium sulphoaluminate phase deformed with increase of heavy metal concentration and the hydraulic reactivity of clinker was changed with the addition of heavy metal. Especially the addition of Cr ion caused to the deformation of Ettringite in the opposition direction of (010).

3:00 PM Break

3:15 PM
Thermodynamic Evaluation of Electronic Waste Treatment: *Scott A. Shuey*[1]; Patrick R. Taylor[1]; [1]Colorado School of Mines, Dept. Metallurgl. & Matls. Engrg., Kroll Inst. for Extractive Metall., 1500 Illinois St., Golden, CO 80401-1887 USA

Electronics disposal has been discussed in some circles for at least the past 25 years. The recycling of electronic waste tends to be a rather complex undertaking when considering the intimate bonding of materials in the average printed circuit board. The development of a recycling process is further complicated when considering the wide variety of products being generated under the heading of "electronics." Ignoring trace elements, and the various halide species generated, will lead process engineers down a development path that is sure to cause material handling issues. Researchers at Colorado School of Mines are modeling the complex chemistry that exists within the body of e-waste. An understanding of the systems thermodynamic behavior will

result in the development of a more robust, long-term process for the recovery of valuable materials from the e-waste stream.

3:35 PM Cancelled
Environmental Control in Russian Alumina and Smelter Plants: Now and the Future

3:55 PM
The Practice on Refining of Impure Indium in Shaoguan Smelter: *Guo Xueyi*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

The coarse indium ingots produced by Shaoguan smelter contained a lot of impurities. In order to produce the high pure indium with the purity more than 99.99%, the appropriate refining processes were designed and the practice of combined use of Glycerin-KI process and electrolysis in Shaoguan smelter was introduced.

4:15 PM
Recovery of Germanium and Indium from Imperial Smelting Process in Shaoguan Smelter: *Guo Xueyi*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

The distributions of zinc, indium, and germanium in the smelting process of the closed blast furnace in Shaoguan smelter were analyzed, and the recovery methods of indium & germanium from the slag by the combined processes of separate-blaze-furnace, electrical furnace, the vacuum furnace process, and fusion-electrolysis process were studied systematically. The focus study was put on the small-scale and pilot experiments of zinc removal from the hard zinc by vacuum distillation, indium and germanium enrichment, and a new process, i.e. Ball milling - Neural leaching - Oxidation calcinations - Chloridizing distillation - Hydrolysis, was designed to recover Germanium from the vacuum furnace slag. The suitable process and operational parameters were determined for the practical production.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Fatigue, Fracture and Failure

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Wednesday PM Room: 3000
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: S. J. Burns, University of Rochester, Mechl. Engrg. Dept., Rochester, NY 14627 USA; B. A. MacDonald, National Science Foundation, Arlington, VA 22230 USA

2:00 PM Invited
Alloying Effects on the Fracture Toughness of Nb-Based Silicides and Laves Phases: *Kwai S. Chan*[1]; [1]Southwest Research Institute, Matls. Engrg. Dept., 6220 Culebra Rd., San Antonio, TX 78238 USA

Nb-based in-situ composites are characterized by a large volume fraction of silicides and Laves phase particles in an Nb solid solution matrix. The intermetalllic hard particles can affect adversely the fracture resistance of the composites. Recent work has shown that certain alloy additions can improve the fracture resistance of Nb-based silicides, Laves phases, and the Nb solid solution. In this paper, the effects of alloy addition on the fracture toughness of Nb-based silicides, Laves phases, and solid solutions are summarized. Theoretical calculations have been performed to determine the influence of alloy addition on the bond order, unstable stacking energy, and the Peierls-Nabarro energy for selected slip systems in the intermetallics. The theoretical results will be used in conjunction with experimental data to elucidate the possible roles of alloy addition in the slip process and the fracture resistance in Nb-based silicides and Laves phases.

2:20 PM
Determination of Fracture Toughness of Ferritic Steels by Automated Ball Indentation Tests: Fahmy M. Haggag[1]; *M. Ashraf Imam*[2]; Heshmat A. Aglan[3]; Robert L. Bridges[4]; [1]Advanced Technology Corporation, 1066 Commerce Park Dr., Oak Ridge, TN 37830-8026 USA; [2]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6320, Washington, DC 20375-5343 USA; [3]Tuskegee University, Tuskegee, AL USA; [4]Oak Ridge National Laboratory, BWXT Y-12, Oak Ridge, TN USA

The Automated Ball Indentation (ABI) test techniques, invented in 1989, determine key mechanical properties of metallic samples and structures in a nondestructive and localized fashion. The test is based on progressive indentation with or without intermediate partial unloadings until the desired maximum depth (maximum strain) is reached, and then the indenter is fully unloaded. The fracture toughness is calculated from integration of the indentation deformation energy up to the critical depth. The measured key mechanical properties are used with other nondestructive measurements such as crack/defect sizes to determine the safe operating/loading conditions of the Ferritic steel structures or to necessitate certain rehabilitation actions. The new fracture toughness capability is, in practical terms, material thickness independent. Furthermore, its localized nature allows testing welds and heat-affected-zones that cannot be tested destructively because of their irregular shape and small volumes. Applications of ABI test techniques to determine the fracture toughness of steel samples, components, and structures, including welds and heat-affected-zones are discussed.

2:40 PM
In-Situ Investigation on the Mechanical Behaviors of Bulk-Metallic Glasses by Thermography: *Bing Yang*[1]; Peter K. Liaw[1]; Mark Morrison[1]; Gongyao Wang[1]; Chain T. Liu[2]; Raymond A. Buchanan[1]; Y. Yokoyama[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37916 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [3]Himeji Institute of Technology, Matls. Sci. & Engrg., Shosha 2167, Himeji City Japan

Since the discovery in the 1990s, bulk-metallic glasses (BMGs) have aroused intense interests because of their ultra-high strengths, super elasticity, and good fracture toughness. Despite these superior properties, the lack of dislocations and plastic deformations in BMGs limits their ductility and fatigue limit in structural applications, especially under tension-loading conditions. In the current research, the mechanical-damage processes of Zr-based BMGs during both fatigue and tensile tests were investigated in-situ by thermography technologies. The thermoelastic effect was used to analyze the stress variations of the BMG specimens during both tensile and fatigue experiments. Except for the final fracture moment, the mechanical behaviors of BMGs have been observed to be dominated by elastic deformations. Multiple shear bands were observed in-situ and analyzed on BMGs before failures during tensile tests by thermography, while no shear bands were observed during fatigue tests, which indicated that the failure mechanism during fatigue could be different from that during the tensile test. As a new nondestructive-evaluation (NDE) method, thermography could open up wide applications in detecting in-situ mechanical damages of materials and structural components.

3:00 PM
Monitoring Tensile Damage Evolution in Nextel/Blackglas Composites: *Jeongguk Kim*[1]; Peter K. Liaw[2]; [1]Korea Railroad Research Institute, Railroad Safety Rsch. & Testg. Ctr., 360-1 Woulam, Uiwang, Kyunggi 437-757 S. Korea; [2]University of Tennessee, Dept. of Matls. Sci. & Engrg., 427B Dougherty, Knoxville, TN 37996-2200 USA

Tensile damage evolution in Nextel/Blackglas composites was monitored with the aid of nondestructive evaluation (NDE) techniques. Several NDE methods, such as ultrasonic testing (UT), infrared (IR) thermography, and acoustic emission (AE) techniques, were employed to analyze damage evolution during tensile testing. Prior to tensile testing, UT was used to characterize the initial defect distribution of the samples. During tensile testing, AE sensors and an IR camera were used for in-situ monitoring of the progressive damages of the samples. AE provided the amounts of damage evolution in terms of the AE intensity and/or energy, and the IR camera was used to obtain the temperature changes during the test. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate the fracture mechanisms and modes of Nextel/Blackglas samples. Moreover, SEM characterization was used to provide the substantial

evidence of failure behavior, and to show comparable results with NDE signatures.

3:20 PM
Micromechanics of Damage Evolution in Solid Propellants: *Petros Sofronis*[1]; Fengbin Xu[1]; Nikolaos Aravas[2]; [1]University of Illinois, Dept. of Theoretl. & Applied Mech., 216 Talbot Lab, 104 S. Wright St., Urbana, IL 61801 USA; [2]University of Thessaly, Dept. of Mechl. & Industl. Engrg., Pedion Areos, Volos 38334 Greece

Solid propellants are composite materials with complex microstructure. In a generic form, the material consists of polymeric binder, ceramic oxidizer, and fuel particles (e.g. aluminum). Damage induced by severe stress and extreme temperatures is manifested in particle cracking, decohesion along particle/polymer interfaces, void opening or even polymer crazing at low temperatures and inert propellants. In this work, the effect of damage due to void formation on the material macroscopic response is investigated from a solid mechanics perspective. First, issues related with the constitutive behavior of the individual phases in the absence of damage are reviewed. Next, with the use of rigorous composite homogenization theory, a macroscopic constitutive law is proposed that accounts for continuous void nucleation and growth upon straining. Numerical calculations for the uniaxial tension test capture most of the experimentally observed features, namely an initial elastic regime, a viscoplastic regime in which void formation competes with hardening in the matrix, a softening regime, and a macroscopic volume expansion which continuously increases with straining.

3:40 PM Break

3:45 PM
Hold-Time Effects on Low-Cycle-Fatigue Behavior of HAYNES® 230 Superalloy at High Temperatures: *Y. L. Lu*[1]; L. J. Chen[1]; G. Y. Wang[1]; M. L. Benson[1]; P. K. Liaw[1]; S. A. Thompson[2]; J. W. Blust[2]; P. F. Browning[2]; A. K. Bhattacharya[2]; J. M. Aurrecoechea[2]; D. L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA; [2]Solar Turbines Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

Total strain-controlled low-cycle-fatigue tests with and without hold times were performed at temperatures ranging from 816°C to 982°C in a laboratory air on a nickel-based superalloy, HAYNES 230. The influence of hold times on the cyclic-stress response and fatigue life was studied. At the temperatures considered, the alloy exhibited initial cyclic hardening, followed by a saturated cyclic-stress response or cyclic softening under low-cycle fatigue without hold times. For low-cycle-fatigue tests with hold times, however, the alloy showed cyclic hardening, cyclic stability, or cyclic softening, which is closely related to the test temperature and the duration of the hold time. It was also observed that the low-cycle-fatigue life of the alloy considerably decreased due to the introduction of strain hold times. Generally, a longer hold time would result in a greater reduction in the fatigue life. For the tests without hold times, the test temperature seems to have little influence on the fatigue life of the alloy at the test temperatures from 816ºC to 927ºC. However, when the test temperature increased to 982ºC, the fatigue life clearly shortened. In addition, the fracture surfaces of the fatigued specimens were observed, using scanning-electron microscopy, to determine the crack initiation and propagation modes. The fatigue life was predicted by the frequency-modified tensile-hysteresis-energy method. The predicted lives were found to be in good agreement with the experiment results. This work is supported by the Solar Turbines Inc., Haynes International, Inc., the University of Tennessee, the U. S. Department of Energy's Advanced Turbine Systems Program, the National Science Foundation (NSF), under Grant No. DMI-9724476, the NSF Combined Research-Curriculum Development Program, under EEC-9527527, the NSF Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the NSF International Materials Institutes (IMI) Program, under DMR-0231320, with Dr. D. Durham, Dr. M. Poats, Ms. W. Jennings, Dr. L. Goldberg, and Dr. C. Huber as contract monitors.

4:05 PM
Experimental Relationships Among Strain, Applied Load and Crack Advance During Stage II Fatigue Crack Growth: *Seon-ho Choi*[1]; Pedro Peralta[1]; James Gee[2]; Zhiyong Xie[2]; [1]Arizona State University, Dept. of Mechl. & Aeros. Engrg., PO Box 876106, Tempe, AZ 85287 USA; [2]University of Pennsylvania Medical Center, Dept. of Radiology, 3400 Spruce St., Philadelphia, PA 19104 USA

The quantification of the strain field ahead of a fatigue crack tip is important to understand fatigue crack growth via plastic blunting and to develop quantitative relationships between crack advance and applied load. The in-plane displacement fields ahead of the tip of a stage II fatigue crack in a CT specimen are measured using the digital image correlation (DIC) method. Pure polycrystalline nickel with an average grain size of 44 ?Ým was used. Cracks were grown under quasi-constant ?´K to prevent excessive stretch of the plastic zone ahead of the crack tip and a half cycle of fatigue load was applied under the scanning electron microscope with different values of the applied load equivalent to a maximum ?´K within the stage II growth regime. The measurements are used to formulate an empirical model to make a connection between the measured strain fields, the applied load and crack advance.

4:25 PM
Prediction of Crack Path Based on Grain Boundary Misorientation and Stress in a Near-Gamma TiAl Alloy: Boon-Chai Ng[1]; *Thomas R. Bieler*[1]; Martin A. Crimp[1]; Darren E. Mason[2]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]Albion College, Math. & Computer Sci., Albion, MI 49224 USA

The process of crack propagation in a duplex Ti-48Al-2Cr-2Nb alloy with equiaxed grains has been studied in a notched four point bend Mode I crack growth specimen. OIM (orientation imaging microscopy) is unable to identify the correct orientation of the c-axis, due to the near-cubic crystal structure, so a method to rapidly determine the true c-axis orientation using SACP (selected area channeling patterns) in conjunction with the OIM scans. A phenomenological fracture initiation parameter incorporating the contributions from deformation twinning and ordinary dislocation systems has been developed, that is capable of identifying boundaries that are likely to nucleate microcracks. This parameter was able to account for sharp turns in the crack path in an arrested crack, as well as predict the subsequent crack path after subsequent loading. Based upon this parameter, criteria for crystal orientations and misorientations are proposed for improving the toughness of duplex microstructures. Supported by AFOSR F49620-01-1-0116.

4:45 PM
Failure Mechanism Map and Minimum Weight Design of Sandwich Beam Consisting of Alumina Facesheet and Aluminum Foam Core: Kapil Mohan[1]; *Tick-Hon Yip*[1]; I. Sridhar[2]; [1]Nanyang Technological University, Sch. of Matls. Eng., N4.1 Nanyang Ave., Singapore 639798 Singapore; [2]Nanyang Technological University, Sch. of Mechanical. & Production Engrg., N3 Nanyang Ave., Singapore 639798 Singapore

Aluminum foams are lightweight, recyclable and have high corrosion resistance, high specific strength, isotropic behavior and good formability. Structural performance of these foams can be enhanced by using them as a core of a sandwich structure comprising thin plates of relatively high stiffness and high strength face sheet materials such as alumina and/or aluminum as face sheets. Cost of these structures in terms of bulk material can be reduced by rigorous design procedure along with the definition of constraint specific to the application. Design by minimum-weight involves consideration of failure modes of these sandwich beams, type of loading, geometrical parameters like thickness of core and face sheets, and length of core of sandwich beams. The constraint for the weight design includes stiffness and strength individually. In the present study, three failure modes, indentation, face yield and core shear were identified in the sandwich structure consisting of aluminum foam core and alumina face sheets und er four-point bending. Failure mode is expressed in terms of non-dimensional geometrical variables for a fixed core relative density. Equations for objective function, weight of the beam, were developed separately in terms of different constraints such as strength and stiffness for each of the failure modes. The design analyses was illustrated in terms of weight index plotted against stiffness and strength indices, all being dimensionless quantities.

5:05 PM
Conformation Structure and Chain Rupture Under Deformation of Linear Oriented Polyethylene: *Ulmas Gafurov*[1]; [1]Institute of Nuclear Physics, Tashkent, Ulugbek 702132 Uzbekistan

Macromolecular chain ruptures with different drawing ratio, that is with different molecular ordering, the conformation state of macromolecules in amorphous regions in stretching deformation of oriented linear polyethylene samples were studied. Macromolecular conformation structure and relative number of the chain rupture was determined by IR spectroscopy by concentration of different molecular terminal groupings measuring. Polymer stretching with larger draw ratio (less coiled isomers concentration and larger molecular ordering), in comparison with lower oriented sample, occurred at considerably larger loads and showed decreases in plastic and elastic deformation, increas-

ing in the number of macromolecular ruptures. Deformation at identical values was accompanied by a greater of molecular chain ruptures. It is proposed that main reason of it is decreasing with polymer sample drawing of coiled isomers concentration in amorphous section of interconnecting macromolecules, in folds and loops on crystalline surface and in entanglements leads to difficulty of macromolecular slippage processes that occur through polymer crystallites. The molecular models of chain slippage and chain rupture of a interconnecting macromolecule for an oriented loaded amorphous-crystalline polymer has been suggested. The crystalline polymer is considered as two-phase one with interchanging amorphous and crystalline regions. Using Frenkel-Kontorova's dislocation models tension, slippage of macromolecules are considered. It is taken into account complex interaction between slippage and rupture of polymer chains.

5:25 PM
Nitrogen Diffusion and Fracture of Nitrided and Nitrocarburized Blunt Notch Three-Point Die Steel Specimens: *Donato Firrao*[1]; Daniele Ugues[1]; [1]Politecnico di Torino, Dip. di Scienza dei Materiali e Ingegneria Chimica, Corso Duca degli Abruzzi, 24, Torino 10129 Italy

Nitriding and nitrocarburizing are among the most adopted surface treatments to enhance steel components wear and fatigue resistances. Yet, nitrogen diffusion causes the reduction of impact resistance and low cycle fatigue properties. A complete understanding of the reasons leading to the brittleness of nitrided and nitrocarburized components still lacking, results of studies on impact tested gas and plasma nitrided or nitrocarburized blunt notch die steel samples are presented. A detailed fracture mechanism sequence is proposed encompassing; (i) multiple mode-I radial cracks formation at the root of the notch beyond the N-diffusion zone; (ii) development of a cylindrical stress-free zone practically creating a notch with a radius larger than the previous one; (iii) concurrent mode-II fractures running along logarithmic spirals of the slip line field forming at the new larger notch; (iv) race restricting to two of them, with one hitting the notched sample centerline before the other and further propagating in to the region of minimum thickness with a mode-I fracture. Suggestions for improved treatments are given.

Microstructural Processes in Irradiated Materials: Carbides, Nitrides and Oxides

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Wednesday PM Room: 3011
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Lance Snead, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6138 USA; Robin Schaeublin, Swiss Federal Institute of Technology Lausanne, Ctr. of Rsch. in Plasma Physics, Villigen 5232 Switzerland

2:00 PM Invited
Microstructural Development in Silicon Carbide During Irradiation at Elevated Temperatures: *Yutai Katoh*[1]; Lance L. Snead[1]; Naoyuki Hashimoto[1]; Sosuke Kondo[2]; Akira Kohyama[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6138 USA; [2]Kyoto University, Inst. of Advd. Energy, Gokasho, Uji, Kyoto 611-0011 Japan

Effect of atomic displacement damage during irradiation is of particular importance for silicon carbide for advanced nuclear and semiconductor applications. As for nuclear applications, utilization of silicon carbide and composites is proposed for gas reactor fuel coatings, gas fast reactor core structures, and fusion blanket structures including ITER test blanket modules. However, understanding of irradiation effects in silicon carbide and the underlying physical processes is very limited. The present paper discusses irradiation effects in cubic silicon carbide with the emphasis on microstructural evolution at temperatures of interest for nuclear applications. Microstructural examination primarily by transmission electron microscopy was carried out to chemically vapor-deposited high-purity cubic silicon carbide samples which had been either neutron-irradiated in High Flux Isotope Reactor (Oak Ridge, TN) at 300-800C or ion-irradiated at DuET dual-beam static accelerators facility (Kyoto University, Kyoto, Japan) at 600-1400C. Based on the results of microstructural characterization, an attempt was made to develop a map of evolution of various microstructural features in cubic silicon carbide as a function of irradiation temperature and displacement damage level.

2:40 PM
Multi-Time-Scale Approach in Simulations of Damage Accumulation in Silicon Carbide: *Fei Gao*[1]; Matthias Posselt[2]; Ram Devanathan[1]; William J. Weber[1]; [1]Pacific Northwest National Laboratory, Fundamental Sci., MS K8-93, PO Box 999, Richland, WA 99352 USA; [2]Forschungzentrum Rossendorf, Inst. of Ion Beam Physics & Matls. Rsch., PO Box 510119, Dresden D-01314 Germany

Damage accumulation irradiation with electrons, ions, and neutrons is a complex process, which can not completely be modeled by molecular dynamics (MD) method, particularly at low temperatures. MD and long-time dynamics based on the dimer method have been combined to perform multi-time-scale simulations of defect accumulations due to electron irradiation of SiC at room temperature. Molecular dynamics simulations are employed to simulate collisional phase of defect production, while long-time dynamics are used to treat thermally activated processes of defects and defect clusters. The close Frenkel pairs often annihilate within a few hundred nanoseconds, but the recovery events occur within milliseconds for the well separated vacancy-interstitial pairs. Interstitials contribute to long-range migrations at room temperature and thus, aggregate into clusters, forming disordered domains. The significant recovery and annihilation of defects during thermal activated processes explains the high amorphization dose for electron irradiation of SiC at room temperature.

3:00 PM
Modeling Thermal Conductivity Degradation in Irradiated SiC: *Lance L. Snead*[1]; Douglass P. White[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA; [2]Merrimack College, N. Andover, MA USA

SiC has many potential applications in Generation IV and Fusion Reactor Components. However, an important consideration is the degradation of thermal conductivity as a result of neutron irradiation. In this work, we present a theoretical mdoel for the thermal conductivity changes expected in neutron irradiated SiC. Phonon scattering by radiation induced vacancies and regions of disorder are considered. The model results are compared to recent experimental observations.

3:20 PM
Irradiation Induced Dislocations and Vacancy Generation in Single Crystal Yttria Stabilized Zirconia: *Jill Noel Johnsen*[1]; Tien Tran[1]; Joanna R. Groza[1]; Fritz Prinz[2]; [1]University of California, Cheml. Engrg. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA; [2]Stanford University, Mechl. Engrg & Matls. Sci., Bldg. 530, 440 Escondido Mall, Stanford, CA 94305 USA

A determination of the most effective method of introducing edge dislocations and defect clusters in single crystal Yttria Stabilized Zirconia (YSZ) has been investigated using several techniques. High-energy particle irradiation using 800 keV electrons, 20 MeV protons, or 1 MeV neutrons promotes the introduction of defects. Thermal annealing and temperature cycling were performed both ex-situ and in-situ in a TEM to study the dynamic recovery behavior of the defects introduced by irradiation and to determine the edge character of dislocations. Defect clusters formed in samples exposed to 20.4 MeV protons with a fluence of 1.00×10^{13} p/cm^2 and thermally annealed at temperatures between 800°C and 1000°C. TEM defect analysis offered evidence that edge type dislocations were formed in YSZ irradiated for two hours with 800 keV electrons and thermally annealed at 1000°C for one hour.

3:40 PM
Microstructure Stability of ZrC, ZrN, TiN, TiC and SiC Irradiated with 1 MeV Kr Ions to 10 and 70 dpa at 800°C: *Jian Gan*[1]; Robert C. Birtcher[2]; Mitchell K. Meyer[1]; [1]Argonne National Laboratory, Nucl. Tech. Div., PO Box 2528, Idaho Falls, ID 83403 USA; [2]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., MSD/212, Argonne, IL 60439-4838 USA

Ceramics of ZrC, ZrN, TiC, TiN and SiC have been considered as the candidate materials for dispersion fuel matrix for gas cooled fast reactor (GFR) in Generation-IV nuclear reactor system. TEM disc samples of the hot-pressed ZrC, ZrN, TiN, TiC and SiC were irradiated using 1 MeV Kr ions in an intermediate voltage electron microscope (IVEM) at Argonne National Laboratory. The irradiations were con-

ducted at a dose rate approximately (2.7~4.0)x10-3 dpa/s at 800ºC to a dose up to 70 dpa that is the proposed lifetime dose for GFR fuel. The in-situ microstructural changes were monitored and recorded using a video camera. Post-irradiation microstructural examination will reveal the changes in microstructure such as precipitate development, lattice expansion and phase change. The impact of the microstructure changes on the GFR dispersion fuel development will be discussed.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Applications of Multicomponent Multiphase Diffusion

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Wednesday PM Room: 3007
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Yuri Mishin, George Mason University, Fairfax, VA 22030 USA; Carelyn E. Campbell, National Institute of Standards and Technology, Metall., Gaithersburg, MD 20899-8555 USA

2:00 PM Invited
Multicomponent Diffusion Effects in Joints of Dissimilar Materials' Engineering Applications: *John A.L. Ågren*[1]; [1]Royal Institute of Technology, Dept. of Matls. Sci. & Engrg., Stockholm 100 44 Sweden

Many engineering applications involve combination of dissimilar materials, as well as processing and usage of such joints at high temperatures where diffusion is rapid. For example, one may mention welded joints of dissimilar steels, composite steel tubes, aluminide coatings on superalloys etc. In addition to the diffusion processes predicted by Fick's law a number of new phenomena occur in multicomponent systems. The Darken effect; up-hill diffusion, is well known in ternary Fe-C-X alloys and of practical importance in steels where carbon is a fast diffusing interstitial. Other effects involve formation of secondary phases even in combinations of one-phase materials of the same phase. Combinations of two-phase materials yield the so-called zigzag diffusion path predicted and analyzed by Morral and coworkers. The Kirkendall effect is caused by differences in individual mobilities and may cause porosity leading to inferior mechanical strength of a joint. Nevertheless, the Kirkendall effect is less well understood in multicomponent systems. A proper understanding of the above phenomena requires a detailed knowledge of the underlying thermodynamic driving forces and phase relations as well as the intrinsic mobilities. In this presentation some recent advances in the modeling of the above phenomena will be discussed.

2:30 PM
Indirect Paths to Final Equilibrium: Kinetics in Multicomponent Systems: *J. M. Vitek*[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Bldg. 4508, MS 6096, Oak Ridge, TN 37831-6096 USA

Currently available software makes it possible to calculate diffusion-controlled phase transformation kinetics in multicomponent systems. However, the paths toward equilibrium are often not direct. Several examples dealing with the austenite/ferrite transformation in steels will be given which reveal the formation and subsequent dissolution of the same phase en route to final equilibrium. While this behavior is certainly indirect, and perhaps not intuitively apparent, the various stages in the transformations can be readily explained by considering the interactions in multicomponent systems. Much of the behavior can be traced to the significantly different diffusion rates in austenite and ferrite, and the same general behavior can be expected in any two-phase system with phases that have grossly different diffusion rates. This research sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

2:55 PM Invited
Simulation of Solution Treatment in Multicomponent Aluminum Alloy Castings: *Harold D. Brody*[1]; [1]University of Connecticut, Matls. Sci. & Engrg., Unit-3136, Storrs, CT 06169-3136 USA

In collaboration with Professor John Morral researchers at the University of Connecticut and the Ohio State University are developing software to simulate the redistribution of alloying elements and the dissolution of intermetallic phases during the solution treatment of Al-Si-Cu-Mg casting alloys. The simulations require increased understanding of solution treatment processes in multicomponent alloys, acquisition and representation in databases of the multicomponent phase equilibria and diffusivity data, and validation of the software by comparison with experiment. The strong dependence of solution treatment kinetics on as-cast microstructure has stimulated revision of solidification models. A major objective of the project sponsored by the Department of Energy and the Center for Heat Treating Excellence is the reduction of heat treatment cycle time. A demonstration project with industry collaborators will test the utility of the simulation to reducing solutionizing time.

3:25 PM
Carburization Process Modeling: *Richard D. Sisson*[1]; [1]Worcester Polytechnic Institute, Ctr. for Heat Treat Excellence, Matls. Sci. & Engrg., Worcester, MA 01609 USA

A brief history of carburization process modeling will be presented followed by an n assessment of the current status of our modeling capability. The database needs will be addressed in terms of the temperature and composition dependence of carbon diffusion coefficients as well as the surface mass transfer of coefficients as a function of temperature, gas composition and steel surface condition. The sensitivity of case depth to process parameters will also be discussed.

3:50 PM Break

4:05 PM Invited
Denisfication of Powdered Steel Preforms by Liquid-Steel Infiltration: *Samuel M. Allen*[1]; Brian D. Kernan[2]; Christoph Sachs[1]; Emanuel M. Sachs[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA; [2]Massachusetts Institute of Technology, Dept. of Mechl. Engrg., Cambridge, MA 02139-4307 USA

The direct manufacture of metal parts by rapid prototyping often employs a metal powder. Densification of the powder can be accomplished either by sintering or infiltration. Infiltration avoids the shrinkage and distortion that typically accompanies sintering. However, conventional steel powder-metal parts are usually infiltrated with copper or bronze infiltrants, and these limit the usefulness of parts because of the resulting non-homogeneous structure and properties. This talk will describe how a gated infiltration process can be used to make parts of a conventional tool steel alloy composition by infiltrating a steel powder preform (made using MIT's Three-Dimensional Printing process) with molten steel alloy having a lower melting point than the base powder. Extensive use of Calphad techniques is necessary to design suitable powder and infiltrant compositions and to select the infiltration temperature. Using this process, we have produced heat treatable D2 tool steel parts having hardness and impact properties comparable to those of wrought D2.

4:35 PM
Interdiffusion and High Temperature Coatings for Gas Turbine Applications: *Jing Liu*[1]; Balaji Jayaraj[1]; Sankar Laxman[1]; Emmanuel Perez[1]; Barbara Franke[1]; Jaewon Byeon[1]; Yongho Sohn[1]; [1]University of Central Florida, AMPAC & MMAE, Box 162455, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA

Science and technology of high temperature coatings is a subject of great interest for its intellectual merit and practical applications. While a myriad of fundamental concepts in materials engineering can be studied from the processing and degradation of these coatings, the reliability, durability and maintainability of these coatings have a significant impact on the performance and efficiency of critical systems for energy production. This talk will survey the importance of multicomponent-multiphase interdiffusion with specific examples from coating-substrate interdiffusion, high temperature oxidation, and composition-dependent phase transformations in thermal barrier coatings and oxidation-resistant protective coatings for advanced gas turbine applications. Through these examples, the role of multicomponent-multiphase interdiffusion on the reliability and failure mechanisms of the high temperature coatings will be highlighted.

Neutron Diffraction Characterization of Mechanical Behavior: Residual Stress II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Wednesday PM Room: 3004
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Ronald B. Rogge, National Research Council, Neutron Prog. for Matls. Rsch., Chalk River, Ontario K0J 1J0 Canada; Thomas Gnaupel-Herold, National Institute of Standards and Technology, Ctr. for Neutron Rsch., Gaithersburg, MD 20899 USA

2:00 PM Invited
Imaging of Single Crystallite Deformation by Neutron Diffraction: *Thomas Gnaupel-Herold*[1]; [1]National Institute of Standards and Technology, Ctr. for Neutron Rsch., 100 Bureau Dr., Stop 8562, Gaithersburg, MD 20899-8562 USA

Diffraction is ideally positioned to study the behavior of grains in select orientations. The domain of neutron diffraction is the analysis of average strains in groups of crystallites having the orientation [hkl] parallel to the scattering vector. This type of investigation often assumes that that sufficiently many grains contribute to the strain measured. This paper will present results of neutron diffraction measurements on deformed coarse grained materials in which the grains are sufficiently large (mm sized) such that the behavior of single grains can be measured. The topics of this investigation are spatially resolved measurements of anisotropic peak broadening and the stress state of selected grains.

2:20 PM Invited
Residual Stress Measurements in Laser Clad Repaired Low Pressure Turbine Blades for the Power Industry: *Philip John Bendeich*[1]; David G. Carr[1]; Ken Short[1]; Richard Blevins[1]; Caroline Curfs[1]; Oliver Kirstein[1]; Gerard Atkinson[1]; Thom M. Holden[2]; Ron Rogge[3]; [1]Australian Nuclear Science and Technology Organisation, Matls. & Engrg. Sci., New Illawarra Rd., Lucas Hgts., New South Wales 2234 Australia; [2]Northern Stress Technologies, Deep River, Ontario Canada; [3]National Research Council, Neutron Program for Matls. Rsch., Chalk River, Ontario Canada

Low pressure turbine blades in power stations suffer from leading edge erosion damage due to water impingement. In an effort to extend the life of these blades, repair of the eroded regions of the blades has been proposed using laser cladding with Stellite material. However, the addition of Stellite results in residual stresses being generated in the parent metal due to contraction during cooling and differences in thermal expansion between the two materials. In this work test coupons and a laser clad blade were examined for residual stresses using both the L3 diffractometer at the NRU reactor, Chalk River, Canada and the TASS neutron diffraction instrument at the HIFAR reactor, Lucas Heights, Australia. In addition XRD results were used to measure residual stresses on the surface of the blade to compliment the neutron measurements. The results of the measurements were used to "calibrate" an FEA model of the weld process.

2:40 PM
Characterization of Residual Stresses in Turbine Discs by Neutron Diffraction and Finite Element Modeling: *Ulrike Cihak*[1]; Peter Staron[2]; Helmut Clemens[1]; Wilfried Marketz[3]; Martin Stockinger[3]; [1]Mining University Leoben, Dept. Physl. Metall. & Matls. Testing, Franz-Josef-Str. 18, Leoben 8700 Austria; [2]GKSS Forschungszentrum, Inst. of Matls. Rsch., Max-Planck-Strasse 1, Geesthacht 21502 Germany; [3]Böhler Schmiedetechnik GmbH & CoKG, Mariazeller Strasse 25, Kapfenberg 8605 Austria

Knowledge of the evolution of residual stresses developing during fabrication of industrial components is steadily gaining importance. Therefore, the prevailing residual stresses in a number of identical, hot-forged, water quenched turbine discs made of nickel-base alloy IN718 have been studied by neutron diffraction. Simultaneously, independent finite element simulations (FEM) have been performed characterizing different quenching rates. The trend of these simulations agrees with that obtained from diffraction measurements. Although the (311) peak was used, which is generally recommended for fcc materials, the results exhibit an offset compared to the FEM results. To clarify this discrepancy further investigations, using an alternative peak, were carried out and a geometrically simple model plate of IN718 applying identical heat treatment conditions was studied. The results, received from the thin model plate and the thick commercial part, are compared with FEM predictions, which are based on detailed measurements of the heat transfer velocity during quenching.

3:00 PM
Residual Strain Distribution in Bent Composite Boiler Tubes and Welded Panels: *Camden Richards Hubbard*[1]; E. Andrew Payzant[1]; Fei Tang[1]; James Keiser[1]; Adam Willoughby[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., MS 6064, Bldg. 4515, Oak Ridge, TN 37831-6064 USA

Kraft recovery boilers, essential chemical and energy recovery units in pulp and paper mills, are typically constructed of carbon steel boiler tubes (SA210 Gd A1) clad with a more corrosion resistant material. These composite tubes are bent and welded together to form air port panels to permit the combustion air to enter at a number of locations around the boiler. Cracking of the bent tubes in the air port is a serious concern. Crack propagation through the clad layer into the carbon steel inner tube likely involves stress corrosion cracking or corrosion-fatigue cracking. The stresses in bent tubes and air ports as manufactured and after service as well as of different designs are being characterized with the goal of contributing to the reduction of failures at air ports.

3:20 PM
Strain Measurements at Railway Wheels: *Mirco Grosse*[1]; Peter Ottlinger[2]; [1]Paul Scherrer Institut, Spallation Neutron Source Div., Villigen 5232 Switzerland; [2]University of Applied Sciences, Dresden 01008 Germany

Strain measurements in railway wheels in the as manufactured state, two usage levels and the end of live state were performed by neutron diffaction using the POLDI facility at SINQ/PSI. In the as manufactured state four railway wheels were investigated. In all four wheels no significant strains were found. After 18400 km usage first strain gradients close to the outer surface of the wheels were detected. In middle ranges including the positions of the nominal highest loads the changes in strain are weak. After an usage of 61000 km the gradients becomes strong at the axial outer position between about +700 ìå close below the tread and - 500 ìå in a depth of 12.5 mm below the tread. At axial middle positions also strain gradients are formed. The end of live state (510000 km) differs only slightly from the state measured after a usage of 61000 km.

3:40 PM Break

4:00 PM Invited
Measurement of Residual and Loading Stress in Composite Materials Using Neutron Diffraction: *Yoshiaki Akiniwa*[1]; [1]Nagoya University, Mechl. Engrg., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

For the composite materials, since the coefficient of thermal expansion of reinforcements is different from that of matrix, the residual stress is induced during cool down from the fabrication temperature. The residual stress has a significant effect on the mechanical properties and strength of the composite. Furthermore, elastic constants of the reinforcement are different from those of the matrix. Then the stress state in each constituent phase during loading depends on the configuration of the composite. In the present study, thermal residual stresses in ceramic composites of alumina mixed with various volume fractions of zirconia and of silicon carbide were measured by the neutron diffraction method. Then phase stresses in an aluminum alloy reinforced with silicon carbide particles were measured under loading. The measured residual stress and the loading stress were compared with the predicted values calculated by the inclusion models.

4:20 PM Invited
Strength Differential Effect in PM 6061Al-15 vol%SiCw Composites: Ricardo Fernandez[2]; *Giovanni Bruno*[1]; Gaspar Gonzalez-Doncel[3]; [1]ILL, Diffraction Grp., 6, rue Jules Horowitz, BP 156, Grenoble F-38042 France; [2]Indo SA, Thin Film Rsch., R&D Dept., Sta Eulalia 181, L'Hospitalet de LLobregat (Barcelona) E-08902 Spain; [3]CENIM, CSIC, Physl. Metall., Avda. de Gregorio del Amo, 8, Madrid E-28040 Spain

The correlation was studied between the strength-differential effect (SDE) in metal-matrix composites and the residual stress (RS)

determined by neutron diffraction (ND). Three 6061Al-15 vol.% SiCw composites were used, with different reinforcement orientations and distributions. Tensile RS in the matrix and compressive in the reinforcement were observed. This agrees with the higher thermal contraction of the matrix than that of the reinforcement. The large hydrostatic microscopic RS agrees with the large fraction of the randomly oriented whiskers. An axial deviatoric stress arises from the remaining whiskers, aligned along the extrusion axis. The RS obtained correlates well with the experimental SDE data. A modified Eshelby model, taking into account the orientation and the spatial distribution of the whiskers was introduced. This agrees with ND results and is able to explain the different SDEs observed in the various composites. The micro-RS can be considered as the origin of the SDE.

4:40 PM
Neutron Diffraction Study of Residual Stress in Thermally Sprayed Metallic Deposits: *Werner Wagner*[1]; Thomas Keller[1]; Nikolaus Margadant[2]; Thilo Pirling[3]; [1]Paul-Scherrer-Institute, Spallation Neutron Source Div., Villigen 5232 Switzerland; [2]EMPA Swiss Federal Laboratories for Materials Testing and Research, Thun Switzerland; [3]Institute Laue Langevin, Grenoble France

The present study investigates metallic NiCrAlY deposits on steel substrates. Neutron diffraction was used to obtain spatially resolved strain and stress profiles in the deposits and the underlying steel substrates. For the neutron diffraction measurements special emphasis was given to a high spatial resolution when entering the surface and crossing the interface to the substrate. Samples of four different spray techniques were analyzed: atmospheric and water-stabilized plasma spraying, flame spraying and wire arc spraying. The results are quantitatively compared with the average in-plane residual stress determined by complementary mechanical profilometry. While the stress profiles are similar for all investigated spray techniques, their absolute values and gradients vary strongly. This is attributed to different quenching stresses from the impinging particles, different thermal histories the deposit/substrate systems undergo during the spraying, and to different coating properties. Crack formation is found to be a dominant mechanism for stress relaxation in the surface plane.

Neutron Scattering in Materials Research: Diffusion and Other Processes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Wednesday PM Room: 3022
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Mike Manley, Los Alamos National Laboratory, Matls. Sci. & Tech. - 6, Los Alamos, NM 87545 USA; Doug L. Abernathy, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831-6474 USA

2:00 PM Invited
Opportunities for Quasielastic Neutron Scattering at the Spallation Neutron Source: *Kenneth W. Herwig*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bethel Valley Rd., Oak Ridge, TN 37831-6474 USA

The Spallation Neutron Source (SNS) is an accelerator-based neutron source currently under construction at the Oak Ridge National Laboratory in Tennessee. Scheduled for completion in 2006, the facility will provide the most intense pulsed neutron beams in the world for scientific and industrial research and development. Fifteen of the available 24 beam line locations have been assigned through a review process to neutron scattering instruments for condensed matter research and a sixteenth has been assigned for fundamental physics studies. Two of the early instruments will provide exceptional opportunities for quasielastic neutron scattering studies of local diffusive motions on time scales from 100's of psec to 0.1 psec and length scales of 2 to 50 Angstroms. The capabilities of these two instruments will be presented with a particular emphasis on applications in materials science and engineering.

2:30 PM Invited
Scientific Outlook for the SNS Liquids Reflectometer: *John F. Ankner*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, PO Box 2008, MS 6474, Oak Ridge, TN 37831-6474 USA

Neutron reflectivity has evolved in recent years from an exotic method used by a small community of experts to a powerful tool for the investigation of thin films and interfaces on the nanoscale. In contrast to x-ray reflectivity, which provides electron density profiles, neutron reflectivity reveals nuclear and magnetization density variation. This is an essential difference when exploring hydrogenous materials such as polymers, Langmuir-Blodgett films, and membranes. Deuterium (2H) features dramatically different scattering properties from hydrogen (1H), allowing contrast enhancement of hydrogenous materials by selective doping of chemically identical nuclei. The SNS liquids reflectometer, due for commissioning in 2006, is designed to accommodate a wide range of scientific studies of both liquid and solid surfaces, over length scales of 10-10,000 Å. Flexible handling of incident angle, wavelength bandwidth, angular resolution, and position-sensitive detection will allow the user to focus on the length and time scales of interest in a particular material. An increase in beam brilliance of a factor of 20-50 over existing instruments will provide new capabilities for kinetic studies, pump-probe, and small-sample experiments.

3:00 PM Invited
Neutron Diffraction and Neutron Scattering Investigations of Cement: *Ronald R. Berliner*[1]; [1]North Carolina State University, Nucl. Reactor Prog., PO Box 7909, Raleigh, NC 27695-7909 USA

Ordinary portland cement is a mixture of several Ca and Al oxide mineral phases that when mixed with water bind rocks and sand together to make concrete. A basic understanding of this material is desirable because of its widespread use as a construction material. Approximately 80M metric tons of cement is consumed annually in the US. Of particular interest are the structure and chemical activity of the isolated cement minerals, the effect of impurities and polytype structural variations on their chemical activity, the nature of the cement hydration reactions and the structure of hardened cement paste. In addition, much remains to be learned about the chemical and environmental avenues of cement and concrete deterioration. For a variety of reasons, neutron methods are particularly effective in the investigation of this system. Neutron diffraction investigations of cement constituent mineral structure will be described. The results of neutron diffraction and quasielastic neutron scattering experiments on the structure, formation, decomposition and water dynamics of cement hydration products will be presented. Finally, the use of neutron quasielastic and inelastic scattering for the investigation of the cement hydration reactions will be described.

3:30 PM Break

3:50 PM
Neutron Diffraction of Hydrogen Absorption in Potassium-Intercalated Graphite: *Channing Ahn*[1]; John J. Vajo[1]; Rachid Yazami[1]; Brent T. Fultz[1]; [1]California Institute of Technology, Mail 138-78, Pasadena, CA 91125 USA

Stage 2 and higher-stage potassium-intercalated graphites are known to adsorb hydrogen at 77K. In the absence of deuterium, the basal planes of graphite show the expected lattice contraction as a function of temperature reduction. In the presence of 4 bar of deuterium, we observe an increase in the basal plane lattice parameter as the temperature is reduced from ambient to 77K due to the absorption of deuterium into the potassium-containing plane. While deuterium saturation is reached at 77K, further expansion of the basal plane lattice parameter is seen as the temperature is lowered to 16 K. This additional expansion is due presumably to the alignment of deuterium molecules within the intercalated layer. While a basic hard sphere model's can account for the known absorption limits of approx. 1.2 wt% at pressures of below 1 bar, a precise determination of preferred sites gives information on the nature of the interaction between potassium and deuterium. This behavior is also seen in Stage 3 and Stage 4 compounds.

4:20 PM
DANSE (Distributed Data Analysis for Neutron Scattering Experiments): Extending the Scientific Toolkit for the Neutron Community: *Michael M. McKerns*[1]; Michael A.G. Aivazis[2]; Tim M. Kelley[1]; June Kim[2]; Brent T. Fultz[1]; [1]California Institute of Technology, Matls. Sci., 1200 E. Calif. Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]California Institute of Technology, Ctr. for Advd. Computing Rsch., 1200 E. Calif. Blvd., MC 158-79, Pasadena, CA 91125 USA

The DANSE system will merge the various computational tasks of neutron scattering into a unified, component based run-time environ-

ment. Standard components will implement data analysis, visualization, modeling, and instrument simulation for all areas of neutron scattering. A core technology of DANSE is an open source framework that supports the software components and mediates their interactions. DANSE will provide tools to help instrument scientists and expert users migrate their existing routines to components, and allow new and casual users to access a stock set of standard analysis applications or configure their own new computing procedures for novel experiments. The modular structure of DANSE parallels the steps of data analysis performed by scientists, thus making it a natural environment for creating flexible computing procedures. DANSE will lower barriers to sharing software, and extend the experimentalist's toolkit with capabilities of analysis and interpretation such as high-performance simulations (band structure, molecular dynamics, etc.), co-analysis of data from multiple experiments, and real-time feedback for experimental control.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Effects of Alloying Additions on the Microstructural Evolution of Solders and Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Wednesday PM Room: 3016
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Sung K. Kang, IBM, T. J. Watson Rsch. Ctr., Yorktown Hgts., NY 10598 USA; Katsuaki Suganuma, Osaka University, Inst. of Scientific & Industrial Rsch., Ibaraki, Osaka 567-0047 Japan

2:00 PM
Interfacial Reactions of Sn-Ag-Cu Solders Modified by Minor Zn Alloying Addition: *Sung K. Kang*[1]; Da-Yuan Shih[1]; Donovan Leonard[1]; Donald W. Henderson[2]; Sungil Cho[3]; Jin Yu[3]; [1]IBM, Rsch., T. J. Watson Rsch. Ctr., 1101 Kitchawan Rd., PO Box 218, Yorktown Hgts., NY 10598 USA; [2]IBM Microelectronics, Endicott, NY USA; [3]KAIST, Dept. of Matls. Sci. & Engrg., Daejon Korea

The near-ternary-eutectic Sn-Ag-Cu alloys have been identified as a leading Pb-free solder candidate to replace Pb-bearing solders for microelectronic applications. However, recent investigations on the processing behaviors and solder joints reliability assessment have revealed several potential reliability risk factors associated with the alloy system. The formation of large Ag3Sn plates in Sn-Ag-Cu joints, especially solidified in a relatively slow cooling rate, is one issue of concern. In the previous studies, the implications of large Ag3Sn plates on solder joint performance and several methods to control them were discussed. The minor Zn addition was found to be effective in reducing the amount of undercooling required for tin solidification and thereby to suppress the formation of large Ag3Sn plates. The Zn addition also caused the changes in the bulk microstructure as well as the interfacial reaction. In this study, an in-depth characterization of the interfacial reactions of Zn-added Sn-Ag-Cu solders on Cu and Au/Ni surface finishes is presented. The effects of Zn addition on modification of the interfacial IMCs and their growth kinetics are also discussed.

2:30 PM
Effect of Rare Earth Element Addition on the Microstructure of Sn-Ag-Cu Solder Joint: *Bo Li*[1]; Yaowu Shi[1]; Yongping Lei[1]; Fu Guo[1]; Zhidong Xia[1]; [1]Beijing University of Technology, Key Lab. of Advd. Functl. Matls., Ministry of Educ., Coll. of Matls. Sci. & Engrg., 100 Ping Le Yuan, Chaoyang Dist., Beijing 100022 China

Addition of small amount of rare earth (RE) alloying elements has been effective in providing solders with improved strength, creep rupture life, as well as refined microstructure. The current study reports the effects of RE element addition on the microstructure of Sn-3.8Ag-0.7Cu solder joint, especially on the intermetallic compounds (IMCs). The growth of IMCs could be dramatically depressed with appropriate addition of RE elements, resulting in a finer microstructure. The effective range of RE addition was determined. In addition to the typical morphology of Ag3Sn and Cu6Sn5 IMCs, other IMCs that have irregular morphology and uncertain constituents were also observed. The cross sections of Cu6Sn5 whiskers exhibited various morphologies. Different eutectic-like structures, with either lamellar, rod or needle-like morphology, dominated the microstructure with different RE content in the solder alloy. It is suggested that such eutectic-like structures directly affect the creep property of the solder joint.

2:50 PM
Microstructure, Solderability and IMC of Sn-Ag-Cu-RE Lead-Free Solder Alloys: Chi-Man Terence Law[1]; *Chi-Man Lawrence Wu*[1]; Daquan Yu[2]; Lai Wang[2]; [1]City University of Hong Kong, Dept. of Physics & Matls. Sci., 83 Tat Chee Ave., Hong Kong SAR China; [2]Dalian University of Technology, Dept. of Matls. Engrg., Dalian 112063 China

The near-eutectic Sn-3.5wt%Ag-0.7wt%Cu (Sn-3.5Ag-0.7Cu) alloy was doped with rare earth (RE) elements, primarily Ce and La, of 0.05, 0.10 and 0.25 wt% to form Sn-3.5Ag-0.7Cu-xRE solder alloys, to investigate the effect of RE additions on the microstructure and solderability of Sn-3.5Ag-0.7Cu-RE solder alloys on copper coupon, and compared with Sn-37Pb. Their intermetallic layer (IML) thickness between the solder and the Cu substrate was also investigated upon thermal aging at 170°C up to 1000 hr. It was found that, with the addition of RE elements, the microstructure was refined such that the β-Sn grain size was decreased. The Sn-3.5Ag-0.7Cu-0.1RE alloy had the best wetting performance, which had become very close to that of Sn-37Pb. It was also found that the thickness of the IML during thermal aging was decreased after the addition of RE elements to Sn-3.5Ag-0.7Cu.

3:10 PM
Characterizing Metallurgical Reaction of Sn-Ag-Cu Composite Solder by Mechanical Alloying with Electroless Ni-P/Cu Under-Bump Metallization After Various Reflow Cycles: *Li-Yin Hsiao*[1]; Szu-Tsung Kao[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec. 2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Electroless Ni-P/Cu under-bump metallization (UBM) is widely used in electronics packaging. The SnAgCu lead-free composite solder pastes were produced by mechanical alloying (MA) process with doping Cu_6Sn_5 nanoalloys. In this study, the interfacial reaction of SnAgCu composite solders with electroless Ni/Cu UBM was investigated with different reflow cycles. Transmission electron microscopy (TEM) analysis was used to identify the intermetallic compounds by the derived selected area diffraction (SAD) patterns and to observe the interfacial region between the SnAgCu composite solder and electroless Ni-P/Cu UBM by bright field (BF) image. Field-emission scanning electron microscopy (FE-SEM) analysis was employed to analyze the morphology and structure in intermetallic compounds. The intermetallic compound (IMC) formed at the interface between the SnAgCu composite solders and electroless Ni/Cu UBM during reflowing were mainly $(Ni_{1-x}, Cu_x)_3Sn_4$ and $(Cu_{1-y}, Ni_y)_6Sn_5$. The elemental distribution near the interfacial region was evaluated by an electron probe microanalyzer (EPMA). Based on the observation and characterization by FESEM, TEM and EPMA, the reaction mechanism of interfacial phase transformation between Sn-Ag-Cu composite solders and electroless Ni-P/Cu UBM with different reflow cycles was proposed.

3:30 PM Break

3:40 PM
An Investigation of the Formation and Presence of Intermetallic Compounds at the Interface Between Substrate (Copper) and Nanoparticle Reinforced Solder: *D. C. Lin*[1]; G.-X. Wang[1]; T. S. Srivatsan[1]; [1]University of Akron, Dept. of Mechl. Engrg., 302 Buchtel Mall, Akron, OH 44325-3903 USA

Eutectic Sn-3.5%Ag solder is an economically affordable and genetically attractive material for performing research studies primarily because it has a simple eutectic microstructure and is a viable and attractive alternative to replace the lead-containing solders. Using the eutectic solder, a series of experiments were conducted to convincingly demonstrate the role of nano-sized powder additions as a logical

sound and economically feasible method for enhancing the strength of Sn-3.5%Ag solder for even trace additions the nanopowders. The increase in strength is ascribed to be due to the conjoint and mutually interactive influences of interfacial enhancement between the copper substrate and the solder, and a refinement of the solder matrix. This presentation will highlight experimental findings and observations relating to wetting of the copper substrate of the solders: (a) eutectic Sn-3.5%Ag solder, and (b) a composite solder resulting from blending the eutectic solder with nanopowders of nickel. Microstructural observations at the solder-substrate interface revealed the nature and morphology of the intermetallic compounds for the nickel nanopowders reinforced Sn-Ag composite solder to be totally different when compared to the eutectic Sn-3.5%Ag counterpart. The kinetics governing microstructural development and intrinsic microstructural features will be highlighted in light of the conjoint influence of reinforcement influences and its resultant influence of heat transfer.

4:00 PM
Effect of Trace Elements on the Interface Reactions Between Two Lead-Free Solders and Copper or Nickel Substrates: *D. Soares*[1]; C. Vilarinho[1]; J. J. Barbosa[1]; R. Silva[2]; P. Bré[3]; F. Castro[1]; [1]University of Minho, Dept. of Mechl. Engrg., Campus de Azurem, Guimarães 4810-058 Portugal; [2]TecMinho - Associação Universidade Empresa para o Desenvolvimento, Campus de Azurem, Guimarães 4810-058 Portugal; [3]Peixinhos, Lda, Rua Silva Aroso, 1311, Apartado 2136, Perafita 4451-901 Portugal

Traditional Sn-Pb solder alloys are being replaced, because of environmental and health concerns about lead toxicity. Among some alternative alloy systems, the Sn-Zn and Sn-Cu base alloy systems have been studied and reveal promising properties. The reliability of a solder joint is affected by the solder/substrate interaction and the nature of the layers formed at the interface. The solder/substrate reactions, for Sn-Zn and Sn-Cu base solder alloys, were evaluated in what concerns the morphology and chemical composition of the interface layers. It was studied the effect of the addition of P or Al, at low levels, on the chemical composition of the layers present at the interface. The phases formed at the interface between the Cu or Ni substrate and a molten lead-free solder were studied, at 250ºC, with different stage times and alloy compositions. The melting temperatures, of the studied alloys, were determined by Differential Scanning Calorimetry (DSC). Identification of equilibrium phases formed at the interface layer, and the evaluation of their chemical composition were performed by Scanning Electron Microscopy (SEM/EDS). Results of the studied systems were compared with the interface characteristics obtained for a traditional Sn-Pb solder alloy. Different interface characteristics were obtained, namely for the alloys containing Zn. The oxidation susceptibility, of both kinds of solder alloys, was measured by TGA. It was studied the effect of the presence of oxygen on the chemical composition of intermetallic compounds formed at the solder/substrate interface.

4:20 PM
Phase Transformations in Doped Lead-Free Solder Paste: *Mark A. Palmer*[1]; [1]Kettering University, IMEB Dept., 1700 W. Third Ave., Flint, MI 48504 USA

Small amounts of eutectic Sn-Bi powder have been added to eutectic Sn-Ag-Bi solder paste to reduce the processing temperature. Solder joints with mechanical properties comparable to joints prepared with eutectic Sn-Pb solder have been prepared at processing temperatures below 220°C. DSC Analysis has been performed to determine if transient liquid phase sintering has occurs. It will also be determined if the small amount of low melting powder can be used to decrease the reflow time at temperatures just above the melting point, without reducing the melting temperature of the solder paste. Undergraduate students have been extensively involved in this work which has been supported by the Rodes Professorship at Kettering University.

4:40 PM
Effects of Bi and Pb on Oxidation in Humidity of Low Temperature Lead-Free Solder Systems: *Keun-Soo Kim*[1]; Katsuaki Suganuma[1]; [1]Osaka University, Inst. of Scientific & Industl. Rsch., Mihogaoka 8-1, Ibaraki, Osaka 567-0047 Japan

As Sn-Ag-Cu lead-free solder has somewhat higher melting temperature than the conventional Sn-Pb solder, and is much more expensive, it is required to establish a certain kind of low temperature soldering techniques. Sn-Zn and Sn-Ag-In solders with or without Bi have relatively low melting temperature close to Sn-Pb eutectic solder. These solders can provide an excellent solution to those requirements mentioned above. However, there are still unknown features on the effects of Bi, which is added for the improvement of wettability, and of Pb, which can be incorporated from Sn-Pb plated components, on Sn-Zn and Sn-Ag-In soldered joints both on oxidation and on Sn whisker formation in humid atmosphere. The purpose of present work is to investigate the microstructure and joining strength changes of Sn-Zn(-Bi) and Sn-Ag-In-Bi soldered joints during humidity exposure. The formation processes of oxide and Sn whiskers in Sn-Zn-Bi and Sn-Ag-In-Bi soldered joints have been examined.

5:00 PM
Additive Alloying Effects on the Generation of Intermetallic Compounds Between Sn-Ag-Ni-Co Solder and Cu: *F. Gao*[1]; T. Takemoto[1]; H. Nishikawa[1]; A. Komatsu[1]; [1]Osaka University, Joining & Welding Rsch. Inst., Osaka 565-0871 Japan

The characteristics of intermetallic compounds (IMC) generated between Sn-3.5Ag solder alloying with additive element couples (0.2wt%Ni and 0.1wt%Co) and Cu substrate was presented. The additive element couples, say, Ni and Co, were all detected in the IMC produced during soldering. The microstructure of IMC was identified as $(Cu_{(1-x-y)}, Ni_x, Co_y)_6Sn_5$ by EPMA and XRD. However, the morphology of $(Cu_{(1-x-y)}, Ni_x, Co_y)_6Sn_5$ was coral-like, and not as dense as the typical scallop-like Cu_6Sn_5. A duplex structure, say, two distinct regions bearing differing concentration of Ni and Co within the $(Cu_{(1-x-y)}, Ni_x, Co_y)_6Sn_5$ were verified. Much higher Ni and Co concentration were probed in the outer region adjacent to the matrix of solder, while lower concentration of Ni and Co at the inner region of IMC connected with Cu. After aging, the $(Cu_{(1-x-y)}, Ni_x, Co_y)_6Sn_5$ tended to be dense. And the Cu_3Sn phase could not be detected after aging at 110ºC, while appeared at 130ºC and 150ºC for 504h.

Phase Transformations Within Small-Size Systems: Transformations in Thin/Thick Films and Multilayers

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee

Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Wednesday PM Room: 3002
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Xinghang Zhang, Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; Christopher A. Schuh, Massachusetts Institute of Technology, Matls. Sci. & Engrg., Cambridge, MA 02139 USA

2:00 PM Invited
Phase Transformations in Epitaxial Thin Films: Y. L. Li[1]; M. Biegalski[1]; A. Sharan[1]; V. Gopalan[1]; D. Schlom[1]; *L. Q. Chen*[1]; K. J. Choi[2]; C. B. Eom[2]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 102 Steidle Bldg., Univ. Park, PA 16802 USA; [2]University of Wisconsin, Matls. Sci. & Engrg., Madison, WI 53706 USA

Many applications of materials require the growth of thin films on a substrate. It is known that the interface between an epitaxial film and a substrate is coherent when the film thickness is small, i.e. below the critical thickness for interfacial dislocation nucleation. The thermodynamics and kinetics of phase transformations in such an epitaxial film is profoundly affected by both the coherent strain due to the substrate and by the film thickness when it is below a few nanometers. The focus of this presentation will be on the effect of substrate constraint on the phase transformations of an epitaxial thin film. It is shown that for the simple cases of isostructural phase separation of a film or of the transformations of a single crystal film to a single domain state, the shifts in critical temperatures of phase transformations can be analytically calculated using independently measured parameters. As an example, BaTiO3 ferroelectic thin films are studied using thermodynamic theories based on the phenomenological Landau theories. It is shown that ferroelectric transition temperatures can be shifted by about 300°C with about 1% compressive strain and about 450°C with 1.6% strain as compared to the bulk ferroelectric transition temperature of ~ 120°C. The predictions are confirmed by ex-

perimental measurements of lattice parameters and second-harmonic generation on fully coherent (001) BaTiO3 thin films epitaxially grown on (110) GdScO3 and DyScO3 substrates as a function of temperature.

2:35 PM
Grain Boundary Segregation and the Equilibrium Grain Size in Nanocrystalline Ni-W Alloys: Andrew J. Detor[1]; *Christopher A. Schuh*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 8-211, Cambridge, MA 02139 USA

In binary solid solution alloys with a tendency for grain boundary segregation, nanocrystalline grain structures can be thermodynamically stable, and grain size can in fact be controlled quite precisely through tailoring of alloy composition. We have explored this issue in binary Ni-W alloys prepared by electrodeposition, where the alloy composition can be varied through changes in electrolytic current density, the shape of the applied current waveform, and temperature of the deposition bath. Films with grain sizes from d ˜ 50 nm down to the amorphous limit (d ˜ 1 nm) have been produced, and their grain structure correlated with tungsten content. The experimental results are rationalized on the basis of thermodynamic calculations as well as computer simulations employing embedded atom method potentials for Ni and W. Finally, the prospect for fundamental studies of mechanical property variations with changes in grain size will be discussed.

3:00 PM
Phase Transformations in Metals Being Electrodeposited: *Oleg B. Girin*[1]; [1]Ukrainian State University of Chemical Engineering, Dept. of Matls. Sci., Pr. Gagarina, 8, Dnipropetrovsk 49005 Ukraine

It has been found that during electrodeposition of metals a supercooled metal liquid is being formed that is solidified at the deposition temperature in the form of a crystalline and/or amorphous phase. These phase transformations are caused by a very fast (explosive) character of metal precipitation as a result of the chain reaction of electrochemical formation of atoms and of the transfer of atom clusters from the liquid state to a more stable solid state. These phase transitions are proved by the existence in the electrodeposited metals of metastable structures corresponding to the amorphous structure of the solidified metal liquid, the highly defective crystalline structure of the metal quenched from its liquid state, and the intermediate modifications resulting from the super-fast crystallization of the liquid phase in the polymorphous metal. This research project is financed by the Ministry of Education and Science of Ukraine, R&D project No. 0102U001953.

3:25 PM Break

3:40 PM Invited
Size Induced Transformations in Nanostructured Thin Films and Multilayers: *Rajarshi Banerjee*[1]; Arda Genc[1]; Gregory B. Thompson[2]; Sangita Bose[3]; Pushan Ayyub[3]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]University of Alabama, Metallurgl. & Matls. Engrg., Tuscaloosa, AL USA; [3]Tata Institute of Fundamental Research, Condensed Matter Physics & Matls. Sci., Mumbai India

Size effects in nanostructured materials have attracted a lot of attention in recent years. The dimensional constraints imposed on either one, two, or all three dimensions in such materials often results in interesting structural changes and consequently influences their properties. In this presentation illustrative examples from our work on a number of different systems will be discussed. Firstly, the case of stabilization of pseudomorphic phases in nanoscale metallic multilayers will be discussed wherein reduction in the layer thickness to the nanometer regime results in one of the layers undergoing a structural transformation and adopting the same crystal structure as that of the adjacent layer. Secondly, size-induced structural transformations in nanostructured thin films will be discussed, including a fcc to hexagonal 4H phase transformation in nanocrystalline Ag and a hexagonal 2H to cubic 3C phase transformation in nanocrystalline CdS. Finally, an interesting example of size-induced metal-insulator transition in nanocrystalline Nb will be presented.

4:15 PM
Atom Probe Tomography of Pseudomorphic Phases in Thin Multilayered Films: *G. B. Thompson*[1]; M. K. Miller[2]; R. Banerjee[3]; H. L. Fraser[3]; [1]University of Alabama, Metallurgl. & Matls. Engrg., A101, Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA; [3]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

Ti/Nb and Zr/Nb thin film multilayers have been sputtered deposited. As the individual layer thickness of either Ti or Zr is decreased, an allotropic phase transformations from hcp to bcc occurred within the Ti or Zr layer. Using a thermodynamic model, the stabilization of the pseudomorphic bcc phase has been rationalized as a competition between interfacial and volumetric free energies. These energies are sensitive to the compositional variations within the layers and at the interfaces. Atom probe tomography (APT) has been used to characterize the interdiffusion and intermixing between the layers. It was found that the amount of interdiffusion and interfacial intermixing was significantly influenced by the stacking sequence of the films as well as the phase of the individual layer. For example, Nb achieved a pseudo-equilibrium concentration of ~ 20at%Nb in the bcc Ti layer where as it had negligible interdiffusion into the hcp Ti layer.

4:40 PM
Oscillatory Reaction in Nanostructured Multilayer Foils: *Jiaping Wang*[1]; Jonathan C. Trenkle[1]; Etienne Besnoin[2]; Omar M. Knio[3]; Todd C. Hufnagel[1]; Timothy P. Weihs[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Reactive Nanotechnologies, 111 Lake Front Dr., Hunt Valley, MD 21030 USA; [3]Johns Hopkins University, Mechl. Engrg., Baltimore, MD 21218 USA

We have observed oscillatory self-propagating exothermic reactions in both Al/Ni and Zr/CuNi/Al multilayer foils. Self-propagating reactions can be initiated in these foils with a small thermal pulse and are driven by a reduction in atomic bond energy. As atoms mix normal to the layers, heat is released and conducted parallel to the layers. Experimental studies and numerical models have demonstrated that the formation reactions propagate in the foils in an unsteady way, characterized by superadiabatic temperature excursions and large variations in instantaneous reaction velocity. Evidence for the oscillations can be seen in the microstructure of the reaction products, the length scale of which correlates with an observed texture on the surfaces of reacted foils. This texture arises from differential thermal expansion in the foil as the reaction propagates unsteadily. We examine and describe the effects of bilayer (or trilayer) thickness, heat of reaction, premixing, ambient temperature, and cooling rate on the magnitude and period of the oscillatory reactions.

5:05 PM Closing Remarks: Vijay Vasudevan, George Spanos, Robert Shull and Xinghang Zhang

Powder Metallurgy Research and Development in the Transportation Industry: Sintering and Densification - P/M Processing

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Powder Materials Committee
Program Organizer: James W. Sears, South Dakota School of Mines & Technology, Additive Manufacturing Laboratory, Rapid City, SD 57701 USA

Wednesday PM Room: 3008
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Dept. of Matls. & Metallurgl. Engrg., Rapid City, SD 57701 USA

2:00 PM
Microwave Sintering of Functionally Graded Composites (Preliminary Study): *Eugene Al Olevsky*[1]; Xuan Wang[1]; Mark Russakoff[1]; Marc Andre Meyers[2]; [1]San Diego State University, Mechl. Engrg. Dept., 5500 Campanile Dr., San Diego, CA 92182-1323 USA; [2]University of California, MAE Dept., La Jolla, CA 92093-0416 USA

Functionally graded materials (FGM) are a promising class of materials for a broad range of industrial applications. In automotive industry, graded composites for claddings, exhaust valves, coatings for tribological applications, electronic circuitry components are examples of the potential usage of FGM where gradual spatial distribution of material composition is essential for solving the problem of thermomechanical properties' mismatch. Due to the inherent nonuniformity of properties, the fabrication of functionally graded materials offers substantial challenges. In particular, during sintering a differential shrinkage can lead to distortions and damage of produced components. In the present study a mathematical model for microwave sintering of functionally graded composites is developed. Based on the model, the evolution of sintered macroscopic shape is predicted

and optimized. The modeling effort is supported by experiments on microwave sintering of green parts shaped by electrophoretic deposition. For several metal/ oxide ceramics material systems it is shown that microwave sintering of graded composites provides both high process efficiency and high final relative density.

2:25 PM
Characteristics of W-Ni-Fe-(Cr) Powder in Metal Injection Molding: *Fei Yi Hung*[1]; Truan Sheng Lui[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., No.1, Univ., Rd., Tainan 701 Taiwan

W-Ni-Fe-(3~6Cr) alloy powders used for metal injection molding (MIM) to understand the sintering structures. The effect of Cr content and tungsten-powder particle size in mechanical properties of W-Ni-Fe-(Cr) alloy were also discussed. According to the experimental results, the shrink rate of volume in W-Ni-Fe alloy decreased with decreased the particle size of tungsten-powder. Even if slightly decreased the particle size of tungsten-powder that could still play an important role on raising fracture resistance and matrix hardness. If added the Cr element, the fracture strength decreased but the salt spray property could be improved. Besides, the Cr element affected the microstructure and it's concentration was also different in composed of phases.

2:50 PM
Repair of Gas Turbine Engine Components with Laser Powder Deposition: Seth Miller[1]; Eric Henderson[1]; Bryan Woods[1]; Matt Heath[1]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technology, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Laser Powder Deposition (LPD) for component repair offers some unique solutions for Gas Turbine Engine applications. LPD is a CAD/CAM solid freeform fabrication technology that uses metal powder and laser fusion to repair components. Inherent to LPD is the ability to add material for repair of critical GTE components with minimal heat affect to the under lying material. Also, due to the nature of LPD, hard coatings can be achieved without heat treatment allowing for repair of heat-treated steels. In some cases LPD repair can be used to replace hard chrome or carburized surfaces. Details of several GTE components that have been repaired will be disclosed.

3:15 PM Break

3:25 PM
Use of Combustion Synthesis in Preparing Ceramic-Matrix and Metal-Matrix Composite Powders: *K. Scott Weil*[1]; John Hardy[1]; [1]Pacific Northwest National Laboratory, Matls. Dept., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

A standard combustion-based approach typically used to synthesize nanosize oxide powders has been modified to prepare composite oxide-metal powders for subsequent densification via dynamic compaction into ceramic- or metal-matrix composites. Metal nitrate salts of interest were dissolved in the appropriate ratio in water and combined with glycine, then heated to cause autoignition. The resulting product consists of an intimate and well-dispersed mixture of nanometer size oxide and metal cystallites. The effects that various processing parameters, such as metal salt ratio, salt-to-glycine ratio, and combustion atmosphere, have on powder composition, reinforcement phase dispersion, and reinforcement and matrix particle size distribution have been examined and will be discussed.

3:50 PM
Structure Property Characteristics of Shock Compacted Bulk Exchange-Coupled Pr2Fe14B/a-Fe Nanocomposite Magnets: Zhiqiang Jin[1]; *Naresh N. Thadhani*[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Erskine Love Mfg. Bldg., Atlanta, GA 30332-0245 USA

Shock compaction offers the potential of fabricating bulk nanocrystalline materials via consolidation of amorphous and/or nanocrystalline alloy powders, while retaining the metastable structure and nano-scale grain size. In this work, shock waves generated using explosives and gas-gun impact, were utilized to consolidate exchange-coupled Pr2Fe14B/a-Fe hard/soft phase nanocomposite powders into bulk compacts. Design of the consolidation fixtures, densification conditions, and starting powder properties allowed control of the final density and retention of the nanoscale structure of the hard/soft magnetic phases in the recovered shock-compacted samples compacts of ~99% of full density. TEM observations revealed retention of ~15-25 nm grain size, ensuring exchange coupling between the hard and soft phases. The resulting properties including remnance, coercivity, and energy product, indicate potential for the use of shock compaction for making bulk nanocomposite magnets for applications in motors for military vehicles. The unique attributes of shock-densification in forming and retaining the nanocrystalline structure, and therefore leading to improved magnetic properties will be described. Funded by DARPA through ARO under grant DAAD19-03-1-0038.

4:15 PM
Bi-Material Transportation Components Using Powder Injection Molding: Densification, Shape Complexity, and Performance Attributes: *Randall M. German*[1]; John L. Johnson[1]; [1]Pennsylvania State University, Ctr. for Innovative Sintered Products, 147 Rsch. W., Univ. Park, PA 16802 USA

Tailored materials, possible via selective placement of different compositions within a component, are useful in several transportation systems ranging from rocket nozzles to automotive engine fuel injectors. Research at Penn State has focused on use of powder injection molding to combine shape complexity with functional design. Controlled sintering of two different compositions within the component is the major difficulty with these structures. Differential shrinkage strains during heating often result in delamination, warpage, or cracking. Via experimentation and modeling, options have been isolated to allow co-sintering; these are through adjustments in solids loading, particle size, minor alloying, and heating cycle to minimize damage during heating. Consequently, co-injection molding of bi-material structures has been realized in several material and property combinations, such as glass sealing alloys linked to heat dissipation alloys, and other combinations based on magnetic response, corrosion resistance, hardness, and low cost. This presentation will introduce the problems, opportunities, sintering protocol, and show a products fabricated using this new technology.

4:40 PM
Preparation and Evaluation of Magnetic Fe-Zn Alloys by Explosive Compaction: *Robert Pennington Corson*[1]; Sivaraman Guruswamy[1]; Michael K. McCarter[2]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [2]University of Utah, Mining Engrg., 135 S. 1460 E., Rm. 313, Salt Lake City, UT 84112-0113 USA

Gallium has been shown to significantly change the magnetic properties of Fe relevant to automotive sensor applications. Like its neighbor gallium in the periodic table, zinc has a large solubility in Fe and a completely filled "d" shell. In this work, the effect of alloying Zn with Fe on the magnetic properties is examined. The low boiling point of Zn makes it difficult to alloy with Fe which has a higher melting point. A novel method of creating Fe-Zn alloys is examined. Powders of each metal are mixed together, and are dropped through a magnetic field to enhance sample texturing. Then the sample is liquid phase sintered to create a compact which is then explosively compacted. The resulting alloy is annealed to ensure chemical homogeneity. An examination of the influence of different processing parameters and the Zn content on the alloy properties is presented. Work supported by NSF-DMR Grant #0241603.

Rare Earths: Science, Technology and Applications V: Reactive Metal Processing

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Renato G. Bautista, University of Nevada, Department of Chemical and Metallurgical Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada, Chemical and Metallurgical Engineering, Reno, NV 89557 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA

Wednesday PM Room: 3001
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Renato G. Bautista, University of Nevada, Cheml. & Metallurgl. Engrg., Reno, NV 89557-0136 USA

2:00 PM
Thermodynamic and Phase Diagram Studies of Some Rare Earth Compounds and Nitrogenation of Fe17Nd Alloy: R. E. Aune[1]; *S. Seetharaman*[1]; [1]Royal Institute of Technology, Div. of Matls. Process Sci., SE-100 44 Stockholm Sweden

With a view to enable a deeper understanding of the electrolytic process route for the manufacture of rare earth alloys, the thermodynamic properties of the rare earth oxides have been compiled and compared with earlier compilations. The data has been corrected for differences in molecular weights and temperature scales. The vapor-

ization of DyF3 was studied by high temperature mass spectrometry. Using the value of enthalpy of formation of solid DyF3 available in literature, the enthalpy of formation of gaseous molecule of DyF3 and its atomization energy were estimated. The Gibbs energy of the gaseous reaction Dy2F6 2DyF3 was evaluated. Mass spectroscopic studies were also carried out in the present laboratory in the case of the system DyF3-Dy2O3 at 1357 K. Phase diagram studies in the case of NdF3-Nd2O3 were also carried out in the present laboratory. Nitogenation studies of Fe17Nd alloys were carried out in the temperature range 1173-1473 K at different N2 partial pressures in a thermogravimetric equipment. The mass changes indicate that there is a long incubation period before the alloy starts picking up nitrogen. This is followed by a sharp mass increase and later a slower increase. At extremely low oxygen partial pressures in nitrogen, the nitrogenated alloy exhibited a Curie temperature of about 600 K. Magnetization studied that the alloys were soft magnets contrary to the behaviour reported in literature.

2:20 PM

Direct Electro-Deoxidation of Metal Oxides to Form Ti-Al and Ti-V Alloys: Stuart L. Finch[1]; *Derek J. Fray*[1]; [1]University of Camabridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Porous pellets containing TiO2, Al2O3 and V2O5 were sintered and then reduced by making the pellet the cathode in a bath of molten calcium chloride. It was found that all the oxides were readily reduced to form Ti-Al and Ti-V alloys. In the case of Ti-Al, there was some loss of aluminium from the pellet. It was found that the final oxygen concentration depended upon the time and temperature of reduction. After 25 hours of reduction the oxygen content of the reduced pellets varied from 600 pmm to 2500 ppm and this was a function of the starting oxides and was in the order TiO2-V2O5<TiO2<TiO2-Al2TiO5<TiO2-Al2O3. The mechanism of the reduction process will be discussed.

2:40 PM

A New High Speed Titanium Production by Subhalide Reduction Process: *Osamu Takeda*[1]; Toru H. Okabe[2]; [1]University of Tokyo, Grad. Sch. of Engrg., 7-3-1 Hongo Bunkyo-ku, Tokyo 113-8656 Japan; [2]University of Tokyo, Inst. of Industl. Sci., 4-6-1 Komaba Meguro-ku, Tokyo 153-8505 Japan

A new titanium production process by magnesiothermic reduction of titanium subhalides using a titanium reaction vessel was investigated. This study discusses the possibility of establishing a high speed, semi-continuous process for the production of high-purity titanium. The titanium subhalide feed material, either titanium dichloride (TiCl2) or titanium trichloride (TiCl3), and magnesium reductant were charged into a titanium reaction vessel, and the vessel was heated at rate 3.3 K min-1 in an argon atmosphere. In the experiment, the sample temperature rapidly increased from around 973~1003 K, and magnesiothermic reduction of titanium subhalide proceeded at high speed. After the reaction, excess magnesium and reaction product magnesium chloride (MgCl2) were removed by draining and vacuum distillation. At this stage, titanium sponge with 99.2% purity was successfully obtained. The titanium reaction vessel showed no signs of damage and thus showed itself to be suitable for magnesiothermic reduction of titanium subhalides.

3:00 PM

Ti Via the Reaction of Molten TiCl2-MgCl2 Salt With Molten Mg: *Akio Fuwa*[1]; S. Takaya[1]; E. Fukasawa[3]; N. Nakahara[3]; [1]Waseda University, Dept. of Matls. Sci. & Engrg., Okubo 3-4-1, Shinjuku-ku, Tokyo 169-8555 Japan; [3]Toho Titanium Co, Chigasaki, Kanagawa 253-8510 Japan

TiCl2 and the molten TiCl2-MgCl2 salt can be well considered as reaction intermediate species in the present Kroll process, where TiCl2 can be considered being produced in the reaction between incoming TiCl4 gas with sponge Ti produced. In this present investigation, this molten salt of TiCl2-MgCl2 has been focused and taken as a reactant in titanium metal production process. First, the molten TiCl2-MgCl2 salt of a specific composition was made through the reaction of solid Ti sponge placed in molten MgCl2 salt with TiCl4 bubbling gas of specific quantity at around 900C, and then, this molten mixed salt was reacted with molten Mg added at the similar temperatures. In our study, titanium metal has been successfully obtained and observed with SEM for their physical characterization. Further, the reaction mechanism of Mg reduction of the TiCl2 constituent in the mixed salt has been discussed in terms of "Electronically Mediated Reaction" mechanism.

3:20 PM

Study on Improving the Current Efficency of the Direct Reduction TiO2 to Ti in Molten CaCl2: *Huimin Lu*[1]; *Chunsen Xu*[1]; *Chunfa Liao*[1]; *Huanqing Huan*[1]; [1]University of Science and Technology, Metallurg. Engrg. Sch., No. 30 Xueyuan Rd., Beijing 100083 China

The direct reduction experiments of TiO2 to Ti in molten CaCl2 (FFC process) are conducted. Reaction mechanism of reduction TiO2 into Ti in molten CaCl2, and the relationships of reduction potential with temperature are studied by Cycle Voltammetry and Chronoamperometry in the range of 800ºC~860ºC. The results of V-A curves, SEM and micro-area elemental analysis show that TiO2 can be reduced to Ti by molten salt electrolysis, and the reduction is conducted in two steps, firstly TiO2 is reduced to TiO, then TiO is reduced to Ti, The reduction potentials in both steps are decreased with the increase of the temperature. In the experiments, the current efficiencies are also estimated; the main reasons of lower current efficiency of FFC process are parasitic reactions such as carbon precipitation and the back-reaction. Some important measures like the modification of cell design are adopted for improving the current efficiency.

3:40 PM Break

3:50 PM Invited

Hydrogen Desorption of TiCl3-Doped NaAlH4 and Na3AlH6: *J. H. Schneibel*[1]; S. A. Speakman[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

Commercial NaAlH4 was doped in a Spex mill with varying amounts of TiCl3 and then desorbed resulting in Na3AlH6. The Na3AlH6 obtained in this manner was doped again with varying amounts of TiCl3. In agreement with work by Kiyobayashi et al. (J. Phys. Chem. A, 2003, vol. 107, pp. 7671-7674), programmed thermal desorption of Na3AlH6 occurred at lower temperatures, when the dopant was primarily added to the Na3AlH6 instead of the NaAlH4. In-situ x-ray diffraction work is in progress to determine the structural reasons for this effect. This research was sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U. S. Department of Energy under Contract No. DE-AC05-00OR22725.

4:10 PM Invited

Fabrication and Testing of a Larger Scale Reactive Materials Processing Facility for the Development of Sodium Based Complex Hydride Materials: *Thad M. Adams*[1]; H. B. Peacock[1]; C. S. Stripling[1]; G. B. Rawls[1]; R. Zidan[1]; M. G. Scott[1]; [1]Savannah River National Laboratory, Matls. Applications & Process Tech. Grp., Bldg. 773-41A/151, Aiken, SC 29808 USA

Many of the materials used in hydrogen service including advanced metal hydride systems are based on highly reactive metal systems. These materials tend to be moisture sensitive, pyrophoric, and/or toxic. Additionally, many of these materials can only be synthesized from their elemental or precursor components under high pressures and at temperature. These aspects make synthesis of both lab scale (gms) and prototype scale (kgs) quantities extremely difficult. The design and implementation of a processing system at SRNL capable of producing and handlingconsolidating/loading—prototype scale quantities of these new storage materials is paramount for SRNL and for the success of a national hydrogen economy. The focus of this paper will be a presentation of the development program related to the design, fabrication, install, and start-up an inert high pressure processing and handling station for the production and loading of environmentally sensitive particulate materialsNaAlH4, Mg-Ni, Li alloys and initial results from processing runs on sodium aluminum hydride materials.

4:30 PM

Annealing and Mechanical Property Study of Processed Zirconium: *Joel W. House*[1]; Philip Flater[1]; Robert J. De Angelis[2]; [1]Air Force Research Laboratory, Eglin AFB, FL 32542 USA; [2]University of Florida/GERC, Shalimar, FL 32579 USA

A study of pure zirconium was conducted to develop an engineering specification of structure for use in high strain rate applications. All specimens were water jet cut from a disk. The disk was 228 mm (9 in) in diameter and 6.4 mm (0.25 in) thick. The annealing specimens were cut in a circular pattern centered about the plate axis. These specimens were annealed one hour in vacuum at temperatures between 150 and 700°C. Compression specimens were cut for quasi-static and high rate mechanical properties. These cylindrical specimens had their axis oriented either in the plane of the plate or through the plate thickness. The hardness values of the specimens annealed at the various temperatures were determined. Optical microscopy was used to characterize the microstructural changes with annealing temperature. These data showed slow grain growth in the zirconium material at low temperatures, less than 400°C, but at higher temperatures grain growth was rapid. X-ray texture determinations were made on the mid-plane of the annealed specimens. Grain growth was accompanied by the formation of a strong basal plane texture. Based on the annealing study,

compression specimens with two different grain sizes were fabricated. The effects of grain size, specimen orientation, and texture on mechanical properties will be reported.

4:50 PM
Recovery of Neodymium from NdFeB Magnet Scrap by Leaching with Sulfuric Acid: *Ho-Sung Yoon*[1]; Sung-Don Kim[1]; Jae-chun Lee[1]; [1]Korea Institute of Geoscience and Mineral Resources, Minerals & Matls. Procg. Div., 30 Gajeong-dong, Yuseong-gu, Daejeon 305-350 Korea

The recovery of neodymium from NdFeB magnet scrap has been investigated by roasting in air and leaching selectively with sulfuric acid. The selective extraction of neodymium was examined in terms of roasting temperature, sulfuric acid concentration, and leaching temperature and time. The solubility difference between ferrous sulfate and neodymium sulfate was utilized to precipitate neodymium as neodymium sulfate which was separated as residue during the solid/liquid separation process after leaching of NdFeB magnet scrap. Also, neodymium could be separated from iron by double salt precipitation using sodium sulfate and neodymium hydroxide was prepared easily by adding double salt to sodium hydroxide solution.

5:10 PM
CFD Modeling of the Vacuum Refining of a 2% Na - 1% K - Li Melt: *Ioannis John Roumeliotis*[1]; Ka Wing Ng[1]; Mainul Hasan[1]; Ralph Harris[1]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., M.H. Wong Bldg., Rm. 2160, Montreal, Quebec H3A 2B2 Canada

CFD modeling was performed using FLUENT to predict the performance of a prototype vessel for the vacuum refining of molten lithium containing 2 wt% sodium and 1 wt% potassium. Refined, high purity lithium metal would find application in the production of thin foil lithium electrodes for rechargeable lithium metal polymer batteries. These batteries are prone to failure when there is an excess of 200 ppm alkali metal impurities such as sodium and potassium in the lithium foil. The modeling has identified design issues that need to be addressed.

5:30 PM
Calciothermic Reduction of Neodymium Fluoride: *R. G. Reddy*[1]; P. T. Velu[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Metallothermic reduction is an important industrial process route for the production of high purity rare earth metals. Production of neodymium metal by the reduction of NdF3 with calcium in the presence of CaCl2 flux, effect of various parameters such as temperature, pressure and salt composition on the yield and impurities content of the Nd metal was analyzed. Thermodynamic calculations using Gibbs energy minimization method was used to characterize the process. Both commercial grade and pure NdF3 showed yield greater than 97%. The yield of Nd decreased with increase in temperature and the flux, and increased with increasing pressure. An excellent agreement was obtained between the calculated and process results. Application of the results to industrial Nd processing systems was discussed.

Recycling - General Sessions: Non-Ferrous Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, LMD/EPD-Recycling Committee
Program Organizer: Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA

Wednesday PM Room: 2011
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Mark E. Schlesinger, University of Missouri, Dept. of Matls. Sci. & Engrg., Rolla, MO 65409-0340 USA; Ragnhild Aune, Kungliga Tekniska Högskolan, Dept. of Matls. Sci. & Engrg., SE-100 44 Stockholm, Sweden

2:00 PM Cancelled
Recovery of Chromium and Nickel from Stainless Steel Dusts

2:25 PM
Bench-Scale Study of the Chromium and Nickel Recovery from Dusts and Sludges Generated in the Stainless Steel Production: *Pedro José Nolasco-Sobrinho*[1]; *Jorge Alberto Soares Tenório*[1]; [1]University of São Paulo, Metallurgl. & Matls. Dept., 2463, Prof. Mello Moraes Ave., São Paulo 05508-900 Brazil

Currently Brazilian stainless steel production is around 350,000 t per year. The stainless steel industry generates dusts and sludges with high amounts of chromium, nickel, and iron. The recycling of these wastes is the best means of treatment. The waste characterization is an essential step in a recycling process definition. The techniques used to characterize the wastes were chemical analysis, particle size distribution, determination of the apparent density, X-ray diffraction analyses, and SEM/EDS. Briquettes were prepared using the wastes, Fe-Si and CaO. The briquettes were introduced in molten liquid steel at 1570°, 1600° and 1635°C. A bench-scale piece of equipment was used to melt the steel. A high recovery of chromium (99%) and nickel (100%) from dusts was found. The time required to recover both chromium and nickel was about 15 minutes at 1600°C.

2:50 PM Cancelled
Kinetics Study of the Chromium Oxide Reduction from Brazilian Stainless Steel Dusts

3:15 PM
Electroslag Crucible Remelting for In-House Recycling Heavily Contaminated Scrap of Ni-Base Superalloys: *Vladyslav M. Sokolov*[1]; Vitaly D. Babyuk[1]; Ewgeny A. Zhidkov[1]; [1]PTIMA, Recycling, 34/1 Vernadsky Ave., Kiev-142 03680 Ukraine

Generation of the significant amount of the scrap contaminated by detrimental admixtures is common in the manufacture of nickel-base superalloy components. The analysis of the current technologies for scrap recycling has demonstrated their complexity and expense. All valuable alloying elements with strong affinity for oxygen are irreversibly lost. Besides, the technologies realization requires additional facilities of outside contractors. A novel cost-effective approach has been proposed for increasing the part of the scrap that can be recycled in-house. It is based on the rarely-used electroslag crucible remelting furnace where a charge is melted and refined in an ambient flux. The furnace replaces a traditional induction furnace in basic application. Moreover, the usage of the specific environmentally friendly flux protects the melt from oxidation. The trials have demonstrated that the recycled metal tolerates the stringent requirements of the superalloy industry for wrought and cast products.

3:40 PM Break

3:55 PM
The Dynamics of an ISF Recycling Zn Containing Materials: *Markus Andreas Reuter*[1]; Antoinette van Schaik[1]; [1]TU Delft, Applied Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

Due to the large variation in recycled materials fed to an industrial Imperial Smelting Furnace in Germany, the operation can vary much. With the aid of sophisticated data analysis including neural nets, PCA and decision tree analysis it will be shown how an industrial furnace moves between different operational states. These states are for example a stable thermodynamic state, a mass transfer driven state and various in-between transitional (dynamic/chaotic) states. It will be shown what drives these states and what initiates them. Furthermore, it will be shown that if these states are not identified it is rather difficult to model this type of reactor, especially since the feed varies so often. This analysis has also been applied on a pig-iron blast furnace showing similar trends.

4:20 PM
Kinetic Study on the Volatilization Reaction of Lead in Electric Arc Furnace Dust: *Jae-Min Yoo*[1]; Byung-Su Kim[1]; *Jae-Chun Lee*[1]; Min-Suck Kim[1]; Jin-Ki Jeong[1]; [1]Korea Institute of Geoscience & Mineral Resources, Minls. & Matls. Procg. Div., 30 Gajeong-dong, Yuseong-gu, Daejeon 305-350 Korea

The volatilization reactions of lead by chlorine components as NaCl and KCl in EAF dust were investigated in the temperature range of 973 to 1223 K under an air atmosphere by using a weight-loss technique. The main volatilization reaction of lead was: 2NaCl + PbO + 2SiO2 + Al2O3 = PbCl2(g) + 2NaAlSiO4. At 1223 K with a reaction time of 180 minutes, the volatilization ratio was 99.0% for lead and 98.3% for chlorine, while only 1.3% for zinc. The Jander equation was found to fit the volatilization reaction rate well over the entire temperature range. The volatilization reaction rate of lead was controlled by solid-solid diffusion. An activation energy of 175 kJ/mol (41.8 kcal/mol) was obtained.

4:45 PM
A Thermochemical Study of Different Options for Halogen Removal from Nonferrous Metal-Containing Wastes: *Jürgen Antrekowitsch*[1]; *Markus Hochenhofer*[2]; Dieter Offenthaler[1]; [1]University of Leoben, Nonferrous Metall., Franz-Josef-Straße 18, Leoben, Styria 8700 Austria; [2]Christian Doppler Laboratory for Recycling of Nonferrous Metals, Franz-Josef-Straße 18, Leoben, Styria 8700 Austria

The recyclability of various nonferrous metal wastes often suffers from a relatively high fluorine and chlorine content. Both are inserted in most cases by the use of plastic contaminated scrap and a wide variety of different coatings. These halogens lead to poor product qualities and off gas contamination. Especially the history of zinc recovery from steel mill dusts shows, that the today common hydrometallurgical options are on their limit and therefore are not sufficient in most cases. This thermochemical study should give more information about different pyrometallurgical options, that offer both a chlorine and fluorine reduction and efficient zinc recovery, preferably in one process step. Selective evaporation in different atmospheres and conversion with the use of hydrogen are the main parts in this study. The results combined with the relatively small information available from literature should provide a basis for further investigations in this field.

5:10 PM
Plutonium Pyrochemistry Spent Salts Treatment by Oxidation and Distillation: *Christophe Robert Thiebaut*[1]; Gilles Bourges[1]; David Lambertin[1]; Laurent Pescayre[1]; [1]CEA Centre de Valduc, Bâtiment 119, Is Sur Tille 21120 France

The pyrochemical processing of actinide metals generates pure metal and contaminated by-products such as salts, crucibles, agitators, anodes and cathodes. Chemical treatment can be used to remove these actinides from metal or refractory material. It is more difficult to treat ceramic materials and the salt baths themselves. We propose a way to treat these salts baths: first, an oxidation of the salt bath; then, the chlorides are removed from this concentrate and separated from the actinide oxides by vacuum distillation. This requires a specially designed apparatus, due to the temperature. We will present the results obtained so far: inactive qualification of the process for NaCl/KCl base salt, carried out with lanthanide surrogates; design and inactive tests of pilot equipment for production scale distillation of oxidized plutonium salts; and modifications in order to nuclearize the pilot plant in order to have the first active tests done in 2005.

Refractory Metals in Electronic Applications: Processing and Properties

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee, EMPMD-Thin Films & Interfaces Committee, SMD-Refractory Metals Committee, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Gary A. Rozak, Fabricated Products, Cleveland, OH 44117 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA

Wednesday PM Room: 3010
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Gary A. Rozak, H.C. Starck, Cleveland, OH 44117-1117 USA

2:00 PM
A Model for the Consolidation of Ultrafine Refractory Metal Powders: *Randall M. German*[1]; [1]Pennsylvania State University, Ctr. for Innovative Sintered Products, 147 Rsch. W., Univ. Park, PA 16802 USA

The refractory metals are fabricated from powders and often this is via the press and sinter route. The resulting components are used in electronic applications ranging from heat sinks to capacitors. However, the unique properties required in the final product often create challenges in processing. A model has been formed for the press-sinter processing of ultrafine and nanoscale refractory metal powders. It predicts apparent density, green density, sintered density, sintered grain size, sintered hardness and strength, and performance attributes using simple inputs of particle size, temperature, pressure, impurity level, and sintering time based on our current understanding of the microstructure evolution during processing. It has been applied to several refractory metal systems. The success is contingent on a new work of sintering concept captured in a single parameter master sintering curve. Demonstrations of property predictions show how novel press-sinter cycles can be isolated for a variety of applicatio ns. This predictive capability allows for process and product optimization based on selected performance features.

2:30 PM
The Effect of Geometry on Residual Strains in Graphite-Mo-Cu Brazed Heatsinks: *F. Michael Hosking*[1]; John J. Stephens[1]; Michael K. Neilsen[2]; [1]Sandia National Laboratories, PO Box 5800, MS 0889, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, PO Box 5800, MS 0893, Albuquerque, NM 87185-0893 USA

Copper backing-plates are routinely used to dissipate heat from temperature-sensitive components. Heat sinks intended for higher temperature service conditions can be attached by brazing. We will describe an application where graphite is brazed to copper with a molybdenum interlayer to mitigate residual stresses caused by thermal expansion and contraction differences during heating and cooling. The effects of brazing area, interlayer thickness, and heat sink thickness on flatness were investigated. The experimental results are compared against finite element analysis, which incorporates unified creep-plasticity constitutive relationships for the Ag-Cu-Ti filler metal. The molybdenum interlayer is generally effective in maintaining flatness with thinner copper substrates. Residual strains increase as the brazing area increases, although smaller vertical displacement occurs if the rigid interlayer is thicker. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

3:00 PM
Direct Brazing Process to Alumina Ceramic Using Conventional Braze Alloys and a Niobium Interlayer: *John J. Stephens*[1]; Gordon E. Boettcher[1]; Charles A. Walker[1]; Paul F. Hlava[1]; Thomas J. Headley[1]; [1]Sandia National Laboratories, Dept. 1861, PO Box 5800, MS0889, Albuquerque, NM 87185 USA

We will present the results of an exploratory study conducted to determine if hermetic seals could be made between bare (non-metallized) sapphire and niobium piece parts using the following three braze alloys: AWS BVAu-3 (62Cu-35Au-3Ni), BVAu-8 (92Au-8Pd), and BVAu-10 (50Au-50Cu). We chose to evaluate these alloys with ASTM F19 tensile buttons pairs made from AL-500 alumina ceramic, brazed in a configuration containing a 0.010 inch thick interlayer of unalloyed Nb. With respect to the tensile button results, the highest loads to fracture and 100% hermeticity were observed with the BVAu-10 alloy. This talk will present the results of microstructural analysis of the braze/ceramic interface region, in order to characterize the bonding mechanism. We will also compare the tensile button test results with the results for brazing sapphire parts to unalloyed Nb with the same three braze alloys. This work was conducted at Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

3:30 PM Break

3:45 PM
New Approach to Direct Element Analysis of Refractory Metals and Their Carbides and Oxides: *Karol Putyera*[1]; Damodaran Sathymurthy[1]; [1]6707 Brooklawn Pkwy., Syracuse, NY 13211 USA

A number of analytical techniques can be used for elemental survey analysis of refractory metals or their carbides and oxides. The techniques currently in use include wet chemistry, X-ray fluorescence and spark source mass spectrometry in the middle of others. However, these hard-to-dissolve materials tend to require complex fusion/dissolution procedures and the use of combinations of nitric, hydrochloric, hydrofluoric and perchloric acids prior to introduction to solution-based methods. Wet chemical methods can be laborious and time consuming, they also dilute the sample by several fold, introduce contaminants, and may result in partial loss of some of the volatile analytes during the fusion/dissolution processes. The disadvantages and limitations of the wet methods prompted us to develop new analytical procedures for glow discharge mass spectrometry (GDMS), which compliment and/or supersede the wet techniques. The most dramatic change is the development of a new flat cell arrangement, in which samples can be analyzed as bulk solids, coatings, films or powders using the same plasma sputtering conditions. In addition, a new approach has been developed for analyzing non-conductive coatings.

4:15 PM
Thermophysical Properties of Molten Refractory Metals Measured by an Electrostatic Levitator: *Takehiko Ishikawa*[1]; Paul-Francois Paradis[1]; Shinichi Yoda[1]; [1]Japan Aerospace Exploration

Agency, Inst. of Space & Astronautical Sci., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

Thermophysical Properties such as the density, the surface tension, and the visocsity of molten refractory metals including Ti, Zr, Nb, Ta, Mo, and W were measured by non-contact techniques combined with an electrostatic levitator. Due to their high melting temperatures and risk of contamination from crucibles, measurements of their properties in liquid phase are very difficult with conventional techniques. Principle of electrostatic levitation, measurement techniques, and experimantal results will be presented.

Shape Casting — The John Campbell Symposium: Applications

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee

Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 2008
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, Pontiac, MI 48340 USA

2:00 PM

Development of Cast Steel Backup Roll: *Xiuhong Kang*[1]; *Dianzhong Li*[1]; Lijun Xia[1]; John Campbell[2]; Li Yiyi[1]; [1]Chinese Academy of Sciences, Inst. of Metal Rsch., Special Environment Matl., 72 Wenhua Rd., Shenyang, Liaoning 110016 China; [2]University of Birmingham, Metall. & Matls., Elms Rd., N. Campus, Edgbaston, Birmingham, W. Midlands B15 2TT UK

This paper concerns the development of a novel casting process for a heavy cast steel backup roll. Total weight of the roll was about 50 tones and the mold was about 7m tall. A "naturally pressurized" gating system was employed to avoid air and inclusion entrainment and to ensure smooth filling. In this way high quality liquid steel was introduced into the mold. It included an offset stepped pouring basin with stopper, hyperbolic-tapered down-sprue with 113 mm entrance diameter and 63 mm exit diameter, radiussed transition between down-sprue and runner, and tangential ingate. The thickness of the sand lining of the steel die at the barrel and journals was optimized by computer simulation to guarantee that the casting solidified progressively towards the feeder. In addition, all shrinkage was arranged to be completely located in the feeder by use of insulation tiles and exothermic powder. Sound castings were produced with zero defects.

2:20 PM

Applications of Campbell's Casting Rules on High Quality Aluminum Castings: *Chen-Chieh Wong*[1]; [1]Metal Industries Research & Development Centre, Casting Tech. Sect., 1001 Kaonan Hwy., Kaohsiung, Taiwan China

Professor Campbell's 10 casting rules have great help in developing high quality aluminum castings. In our research, we apply these concepts to produce CVD heater and gate valve body castings for semiconductor equipment used under vacuum environment. Housings for aerospace purposes are also developed. At first, Campbell's rules are adopted to design runner and gating system. The proper designs of runner and gating system include bottom filling, low filling rate, and good design of pouring basin, riser size and venting. Then CAE software is used to confirm the designs. By such process, we are able to reduce to the leadtime of development and to achieve low scrap ratio and cost as well. To meet the first requirement of the rules, good quality liquid metal, special degassing method is used to reduce the hydrogen content below 0.02cm3/100g measured by first bubble detection. The CVD heater is cast by sand casting and the gate valve body is cast by permanent mold die casting process. Aerospace housing is cast by the Quickcast process. Instead of wax patterns, SLA pattern — resin model — is used to make ceramic mold for investment casting in Quickcast process. All these three castings pass X-ray examinations and even fluorescent penetrant inspections.

2:40 PM

A Rational Approach to Casting—Building Quality into the Process: Marcos I. Cardoso[1]; Andres F. Rodriguez[1]; Geoffrey K. Sigworth[1]; Jose Talamantes[1]; [1]Nemak, S.A. de C.V., R&D Dept., A.P. 100, Bosques del Valle, Garza Garcia, Nuevo Leon 66221 Mexico

During the last twenty years producers and consumers of aluminum castings have increasingly come to realize the importance of eliminating defects (inclusions and gas porosity) to obtain highest quality. And the procedures used in the cast shop determine metal cleanliness, which is why the plant processes are often qualified when a new part goes into production. In this paper, a review is first presented of the theoretical and experimental data which quantify the loss of tensile and fatique strength when defects are found in a casting. Then liquid metal treatment procedures are considered, and their effect on metal cleanliness presented. The review shows that modern fluxing, degassing and filtration procedures must first be used to clean the metal. Then, clean metal must be introduced directly into the mold, without any splashing or metal turbulence. It is only in this way that castings of consistently high quality may be produced.

3:00 PM

Campbellology for Ferrari: *Vian Francis Coombe*[1]; [1]Ferrari Spa/ Gestione Sportiva, Direction Tecnica - Metall., Via Ascari 55/57, Maranello, Mo 41053 Italy

With the ever-increasing demands to save weight, increase performance and reduce costs, the auto industry is increasing the use of light alloy materials. Ferrari Auto is no exception. Ferrari,in 1996 adopted to use an all aluminium space frame chassis for their new 360 Modena car. The design partnership of Ferrari and Alcoa called for a mix of extrusions welded to 12 strategic castings. The quality and mechanical characteristic requirements of the castings meant considerable changes to the Ferrari in-house foundry. Using an example of the methods and process route of the Apost door pillar casting, this paper describes how the foundry was transformed utilising the philosophy of John Campbell as a base for achieving the specification aims for the casting components.

3:20 PM

Controlled Diffusion Solidification - Manufacturing Quality Net Shaped Al Based Wrought Alloy Parts: *Deepak Saha*[1]; Sumanth Shankar[2]; Diran Apelian[1]; Makhlouf M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Metal Procg. Inst., 100 Inst. Rd., Worcester, MA 01609 USA; [2]McMaster University, Dept. of Mech. Engrg., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada

Aluminum combined with differing percentages of other metals such as silicon, copper, magnesium, and manganese form alloys that are used in many domestic, automotive and aerospace applications. Net shape manufacturing of wrought alloys (via casting) has been prohibitive due the proclivity of these alloys to "hot tears". Hot tearing tendency coupled with coherency temperature, that is close to the liquidus temperatures, lead to the development of cracks in the final "as cast" structure. The microstructure of wrought alloys is predominantly dendritic, and by altering the dendritic morphology to a globular one (providing a continuous interdendritic liquid network) one can considerably reduce the hot tearing issues. A novel method has been developed at WPI, termed Controlled Diffusion Solidification or CDS. In this process, two precursor liquid alloys of precisely controlled chemistry and temperature are mixed to produce a predetermined alloy composition. CDS provides an effective process for the net shape manufacturing of these alloys. The CDS process is discussed and reviewed; results from industrial trials utilizing a variety of different casting processes are presented, along with the resultant microstructural data and mechanical properties.

3:40 PM Break

3:50 PM

Effect of Die Temperature and Melt Quality on a Low Pressure Die Cast Engine Cylinder Head: *Geoffrey Robert de Looze*[1]; [1]CSIRO, Mfg. & Infrastruct. Tech., Locked Bag 9, Cnr. Albert & Raglan Sts., Preston, Victoria 3072 Australia

Automotive casting plants are traditionally interested in reducing in-house scrap and increasing productivity. The global research effort supporting this activity is arguably separated into pragmatic factory-based developments, and simplified laboratory investigations. To help bridge this gap, this paper describes a laboratory-based investigation into the production of a low pressure die cast (LPDC) engine cylinder head made using a modified water-cooled casting die mounted on a commercial LPDC machine. Comprehensive process temperature measurements were combined with quantitative metallography. These measurements showed how the machine operating parameters and the melt quality level affected the casting cooling rate and/or microstructure. Micro-porosity form and distribution in the castings was used as an indicator of casting quality and solidification conditions, and experimental evidence for the operation of burst feeding in LPDC was detected. Significant improvements to casting directional solidification and microstructural refinement were shown with use of forced die

cooling, however the limitations of this technique also became apparent. These results are illustrated in terms of the geometry of the casting cross-section.

4:10 PM
Breaking the Grip of Quartz: Silica-Free Foundry Sands at Silica Sand Prices: *Kenneth Peter Harris*[1]; [1]Noram Technology Ltd., Britannia House, 960 High Rd., London N12 9FB England

Noram Technology has developed technology that is being used to convert surplus fine fractions from construction aggregate manufacture to good quality, silica-free foundry sand. Since very many foundries are located within 200 miles of a quarry producing suitable aggregates, foundry sand made this way can be offered for sale at prices close to silica sand. Unlike quartz, these silica-free sands have low, linear thermal expansions; they are compatible with all commonly used binders and free from the health and safety issues affecting silica. A Noram plant also recycles foundry sand at exceptionally high yields, enabling sand consumption per ton good of casting to fall by 50% or more independent of binder system. If local demand for foundry sand is insufficient to warrant manufacture at the quarry, a foundry operating a Noram recycle plant can make its own from the abovementioned fine fractions. This technology will allow many foundries to replace silica sand and increase the use of low-cost environmentally benign inorganic binders.

4:30 PM
The Cosworth Casting Process: Evolution and Benchmark: *Nicholas R. Green*[1]; Andrew M. Tomkinson[1]; Thomas C. Wright[1]; Jon P. Evans[1]; [1]Cosworth Technology Ltd., Wainwright Rd., Shire Business Park, Worcester WR4 9FA UK

The Cosworth Casting Process was developed to allow manufacture of small volumes of high performance castings and the pedigree of the process established through a record number of wins in Formula 1 racing. The process has evolved successfully into high volume production through the application of rollover of the mould under pressure from the pump immediately after filling. To benchmark the reliability of components manufactured with the high volume casting process, elevated temperature fatigue tests were performed and fatigue life distributions characterised with Weibull statistics. Material samples from the bearing panels of precision sand-cast AlSiCuMg production cylinder blocks from Cosworth Technology and two other European suppliers to Audi were tested at 85 MPa and 150°C under fully reversed rotating-bending. All materials exhibited similar upper fatigue life limits. However, the Cosworth Technology cast material exhibited a Weibull modulus of fatigue of 3.0, whilst the other precision sand processes each showed significantly greater scatter in fatigue life with Weibull moduli of only 1.5. No correlation was found between initiating defect size and fatigue life. It is concluded that the presence of oxide films entrained in the gravity casting of the other components accounted for their increased unreliability.

4:50 PM
John Campbell's Closing Remarks: *John Campbell*[1]; [1]University of Birmingham, Metall. & Matls., Elms Rd., N. Campus, Edgbaston, Birmingham, W. Midlands B15 2TT UK

Prof. Campbell will offer his unique perspective on the papers presented during this conference.

5:30 PM Conference Close

WEDNESDAY PM

Superalloys and Coatings for High Temperature Applications: Superalloys - II

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3
Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Wednesday PM Room: Nob Hill A/B
February 16, 2005 Location: San Francisco Marriott

Session Chairs: Qiang (Charles) Feng, University of Michigan, Dept. of Matl. Sci. & Engrg., Ann Arbor, MI 48109 USA; Richard S. Bellows, Solar Turbines Inc., Matls. & Process Engrg., San Diego, CA 92186-5376 USA

2:00 PM Invited
Advances in Material and Coating Technologies for Industrial Gas Turbine Applications: *Zaher Z. Mutasim*[1]; [1]Solar Turbines, Matls. Engrg., 2200 Pacific Hwy., San Diego, CA 92101 USA

The demand for increased gas turbine efficiency and improved durability, while reducing life cycle cost, has evoked a lot of attention from gas turbine original equipment manufacturer (OEMs) and end users. While OEMs are continuing their drive to improve engine performance by introducing novel concepts to the engine designs, it becomes apparent the many limitations the engine designer experiences. These include the levels of mechanical and thermal stresses that the turbine will experience at the expense of improved performance. This presentation describes the applications and the environment that industrial gas turbines experience, and the consequent degradation mechanisms that the turbine materials are subjected to. Market and customer drivers are also defined, and used to develop new material and coating solutions to satisfy customer demands. Advances in materials and coating systems are presented for gas turbine combustor liner and gas turbine blades and nozzles.

2:30 PM Invited
Crystallographic Implications of Creep Deformation of Single Crystal Superalloys Subject to Multiaxial Loading: Hector C. Basoalto[1]; Mamoud G. Ardakani[1]; R. N. Ghosh[2]; Barbara A. Shollock[1]; *Malcolm McLean*[1]; [1]Imperial College London, Exhibition Rd., London SW72AZ UK; [2]National Metallurgical Laboratory, Jamshedpur India

Most models of anisotropic creep appeal to two or more active slip systems to account for the observed creep behaviour as a function of orientation, stress and temperature. Validation of the models by comparing predicted crystal rotations for uniaxially loaded off-axis specimens with EBSD measurements is complicated by the progressive development of triaxial stresses in nominally uniaxial tests due to geometrical constraints. The present paper presents a comparison of (i) predictions of creep deformation in multiaxial stresses, including crystal rotations, from an implementation of a multiple slip model in ABAQUS via a User Creep Sub-Routine and (ii) EBSD measurements of the spatial variation in crystal rotation in circumferentially-notched creep specimens.

3:00 PM
Creep of Ru-Containing Nickel-Base Single Crystal Superalloys: *Laura J. Rowland*[1]; Q. Feng[1]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 2300 Hayward, Ann Arbor, MI 48109 USA

There is a continuing demand for development of nickel-base superalloys that can maintain structural integrity at temperatures of 1100°C and beyond. Ru additions have been found to increase the liquidus temperature of nickel-base superalloys, reduce the propensity for formation of TCP phases, and improve creep rupture strength at 1100°C. The experimental Ru-containing nickel-base single crystal superalloys investigated had cuboidal, intermediately-shaped, or spherical γ' precipitates. The rafting behavior of the experimental alloys

also varied dramatically indicating there is a range of γ-γ' lattice misfit from negative to near zero to positive. The objective of this study was to investigate the role of Ru in influencing creep properties in a matrix of alloys with a wide range of composition, with moderate amounts of Re and W, and Ru content up to 9.7 wt%. It was determined the impact of Ru on creep strength is strongly dependent on microstructure, rafting direction and dislocation substructure.

3:25 PM
Miniature Specimen Creep Testing of a Low Density Single Crystal Super Alloy: *Gotthard Mälzer*[1]; Aleksander Kostka[1]; Gunther Eggeler[1]; Thomas Mack[2]; [1]Ruhr-Universität Bochum, Lehrstuhl Werkstoffwissenschaft, Bochum D-44780 Germany; [2]MTU Aero Engines, München 80995 Germany

We present mechanical and microstructural results on miniature specimen creep testing of a low density Ni-base superalloy in the 1000°C temperature regime. We give some back ground information on the development of our miniature specimen creep testing procedure. We show that the main features of creep can be captured. We present creep curves and show how the secondary creep rate depends on stress and temperature for three crystallographic directions (tensile loading in <100>, <110> and <111> directions). Moreover we use scanning and transmission electron microscopy to characterize the microstructural evolution during creep. Emphasis is placed on the coarsening of the ã/ã?-microstructure (rafting), the nucleation of cracks at cast micro pores, the appearance of rupture surfaces and on the cutting of the ã?-phase by dislocations. Our results show that our miniature test technique provides reasonable creep data and is ideally suited to study the microstructural processes occurring.

3:50 PM Break

4:15 PM
Microstructural Study of Nickel-Base Superalloys for Ultra Supercritical (USC) Coal Power Plants: *Quanyan Wu*[1]; Vijay K. Vasudevan[1]; John Shingledecker[2]; Robert Swindeman[2]; [1]University of Cincinnati, Cheml. & Matls. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [2]Oak Ridge National Laboratory, Ceram. & Metals, Bldg. 4500S, MS6155, PO Box 2008, Oak Ridge, TN 37831 USA

The demand for higher efficiency and reduced environmental effects in coal-fired power boilers has to result in the use of higher steam temperatures and pressures. A significant materials effort is to reach a target steam condition of 760°C and 35MPa to meet USC requirements. This will require the use of Nickel-base superalloys, in which long-term creep strength is a critical factor. In this study, the microstructural stability and evolution of Haynes 230, CCA617 and HR6W after ageing and creep-test under various temperatures are examined and discussed. Creep test results at ORNL showed that CCA617 exhibits a significant improvement over the standard alloy 617 in creep rupture strength while in HR6W, failure to meet the expected performance was encountered. Microstructural analysis focusses on the formation, distribution, size and volume fraction of gamma prime, carbides, and their interaction with dislocations. TEM is the main characterization tool in addition to SEM, EDS and microhardness tests.

4:40 PM
Nondestructive Characterization of Microstructural Degradation in Creep Damaged Ni-Based Superalloys by Ultrasonic Techniques: *Jaewon Byeon*[1]; Jinhun Song[2]; Sookin Kwun[2]; Yongho Sohn[1]; [1]University of Central Florida, AMPAC & MMAE, Box 162455, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA; [2]Korea University, Matls. Sci. & Engrg., Anam-dong, Sungbuk-gu, Seoul 136-701 Korea

The creep damage of two Ni-based superalloys, IN738LC and PM1000 were non-destructively examined by ultrasonic velocity and attenuation. Microstructural features related to creep damage were quantitatively correlated to the ultrasonic velocity and attenuation. The creep damage in PM1000, at 1000°C under the tensile stress range of 110-123MPa, occurred by the formation of cavity, and decreased the ultrasonic velocity, because the increase in the volume fraction of creep cavity decreased the Young's Modulus. The creep damage in IN738LC, at a temperature range of 850-950°C and tensile stress range of 116-255MPa, occurred by the directional coarsening (rafting) of gamma-prime precipitates. A linear correlation was observed between the ultrasonic attenuation coefficient and the mean length of precipitates in IN738LC. The potential of ultrasonic technique to assess creep damage in high temperature alloys is discussed with an emphasis on the relationship between the microstructural damage mechanisms and the governing principles of ultrasonic response.

5:05 PM
Ultrasonic Fatigue of a Single Crystal Superalloy at Elevated Temperature: *JianZhang Yi*[1]; Chris J Torbet[1]; Tresa M Pollock[1]; J Wayne Jones[1]; [1]University of Michigan, Matls. Sci. & Engrg., H. H. Dow Bldg., Ann Arbor, MI 48109-2136 USA

Turbine airfoils are usually subjected to high temperatures, aggressive environment and vibratory stresses arising from a wide range of stimuli, such as the turbulent flow around the airfoil itself. Traditionally, high cycle fatigue testing is confined to 10^7 cycles due to the time consumed with the conventional testing equipment. In the present study, an ultrasonic fatigue testing system, operating at a frequency of approximately 20kHz, was used to explore the high cycle fatigue behavior of a single crystal PWA 1484 superalloy at high temperature (1700°F) and in the lifetime regime of 10^6-10^9 cycles. The resultant fatigue properties were thus analyzed and discussed by identifying the crack initiation and characterizing the development of fatigue damage evolution in the superalloy.

5:30 PM
The Effect of Stress on the Long-Term Phase Stability of Ni-Base Superalloy U720LI: *Satoshi Takahashi*[1]; Yoshinori Ito[1]; Sadao Nishikiori[1]; [1]Ishikawajima-Harima Heavy Industries Co., Ltd., 1 Shin-Nakaharacho, Isogo-ku, Yokohama, Kanagawa 235-8501 Japan

Ni-base superalloy U720LI has been widely used for the rotating components in aircraft-engines. In this study, we focused on the effect of stress on sigma phase precipitation. Alloy forged and heat-treated consists of duplex grain size, which is caused by distribution of primary ã'. Exposure heat treatments were performed at 700°C and 750°C for times up to 3000 hours in air. For evaluation of mechanical properties, creep rupture test, LCF test and dwell LCF test were carried out at temperatures described above. Microstructural observation was conducted for these specimens, and then the influence of the stress on precipitation kinetics was discussed. From this work, it is found that stress accelerates the precipitation of sigma phase, and regardless of stress present, sigma phase is prone to precipitate in fine grain area.

Surface Engineering in Materials Science - III: Plasma Processing for Surface Modification

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Wednesday PM Room: 2022
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Arvind Agarwal, Florida International University, Dept. Mech. & Matls. Engrg., Miami, FL 33174 USA

2:00 PM Invited
Extended Thermal Cycle Lifetime in Thermal Barrier Coatings with Bond Coats Made from Cryomilled Powders: *Julie M. Schoenung*[1]; Feng Tang[1]; Leonardo Ajdelsztajn[1]; George Kim[2]; Virgil Provenzano[3]; [1]University of California, Chem. Engrg. & Matls. Sci., 2017 Kemper Hall, Davis, CA 95616 USA; [2]Perpetual Technologies, Montreal H3E1T8 Canada; [3]National Institute of Standards & Technology, Gaithersburg, MD 20899 USA

The objective of this study is to increase the thermal cycle lifetime of thermal barrier coatings (TBCs) by modifying their bond coats. The main idea is the cryomilling of the NiCrAlY powder that is used to make the thermal sprayed bond coat. The cryomilled powder has nanocrystalline grains and contains in-situ dispersed nano-scale oxides and nitrides. These features are expected to remain in the bond coat after thermal spray and, therefore, to modify the oxidation behavior of the TBC. LPPS and HVOF thermal spray processes have been used to produce the bond coats. The top coats have been applied with the APS process. The thermal cycle test is conducted in air at 1121°C with a one-hour cycle. Our results show that the lifetime of the TBCs with

cryomilled bond coats can be greater than 1300 cycles, which is comparable to the longest lifetimes reported for high-cost EB-PVD TBCs.

2:25 PM
Synthesis and Characterization of Vacuum Plasma Sprayed Tantalum Carbide Coating: *Kantesh Balani*[1]; Gabriela Gonzalez[1]; Arvind Agarwal[1]; Robert Hickman[2]; [1]Florida International University, Mechl. & Matls. Engrg., EAS 3400, 10555 W. Flagler St., Miami, FL 33174 USA; [2]Plasma Processes Inc., 4914 Moores Mill Rd., Huntsville, AL 35811 USA

Tantalum carbide (TaC) is a candidate coating material for high temperature applications as in rocket nozzle throats and liners. But, the high melting temperature and brittleness of TaC poses hurdles in synthesizing such coatings with conventional processing routes. Vacuum plasma spraying (VPS) has been employed for coating TaC and surpassing processing difficulties. High cooling rates experienced during the VPS process inherently leads to formation of non-equilibrium phases in the final microstructure. X-Ray Diffraction analysis has detailed the generation of Ta2C phase during VPS. SEM and TEM studies clearly show the disparity of non-homogeneous and non-stoichiometric phases in the sprayed coating. Microhardness analysis was extended to gather fracture toughness data through Vicker indentation cracks in the vacuum plasma sprayed TaC. The dissociation of decarburization of TaC to form Ta2C during plasma spraying is a processing problem and requires further optimization work for ongoing research.

2:40 PM
Molybdenum Silicides as High-Temperature Corrosion-Resistant Coatings: *Peter F. Tortorelli*[1]; Michael P. Brady[1]; Ian G. Wright[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6156 USA

The high-temperature corrosion behavior of Mo-Si-B alloys under various environmental conditions is being studied for possible use in advanced fossil-fuel systems as corrosion-resistant coatings. Alloys of Mo-Mo5SiB2-Mo3Si with different compositions and phase morphologies were oxidized in dry air or exposed to an H-2-H2S-H2O-Ar environment. Effects of the multiphase nature (composition, morphology) of the Mo-Si-B system on environmental resistance under these conditions were evaluated. Microstructural characterization indicated that the oxidation reactions resulted in cooperative behavior among the different phases while preliminary analyses suggested that sulfide formation mimicked the starting alloy microstructure. Quite low corrosion rates under sulfidizing conditions were observed. Implications of these findings for design of smart coatings for oxidation and/or sulfidation resistance will be discussed. Research sponsored by the Advanced Research Materials Program, Office of Fossil Energy, U. S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

2:55 PM
Plasma Spray Formed Near-Net-Shape MoSi2-Si3N4 Nanocomposites: *Viswanathan Venkatachalapathy*[1]; [1]University of Central Florida, Mechl., Matls. & Aeros. Engrg., 381 Engrg. Bldg., 4000 Central Florida Blvd., Orlando, FL 32816 USA

The commercial applications such as high temperature aerospace (gas turbine engines) and automotive applications of MoSi2 based nanocomposites rely on the successful consolidation of these materials into bulk-sized components while preserving their nanostructures. This article summarizes the challenge for the successful consolidation of MoSi2-Si3N4(nano/micro) nano-composite by plasma spray forming. Plasma sprayed cylindrical reinforced MoSi2 composite have been fabricated by the plasma spray forming without any defect on the surface. A detail characterization of the spray formed bulk nano-composite has been performed along the radius of the composite using optical microscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and Vickers hardness tester. Vickers hardness and fracture toughness of the nanocomposite showed a little deviation from the expected, that might be due to the difference in particle size and distribution in the MoSi2 matrix as a funct ion of component thickness. The higher hardness and fracture toughness were observed at the middle of the composite and the average fracture toughness of the plasma sprayed bulk nanocomponent was 6.3 MPa m1/2. The higher fracture toughness value could be attributed to the retention and the presence of nanoparticles in the matrix, though with some porosities. High temperature oxidation behavior was studied and showed excellent high temperature resistance. Furthermore, the electronic structure of the composite was studied using the ab-initio QM principles and the DFT theory. The changes of the density of states and the band structure, together with the decreased Fermi energy and the total energy of the reinforced MoSi2 system were calculated and related to its superior mechanical and physical properties.

3:10 PM
Erosion-Oxidation Behavior of Steels and Thermal Sprayed Cermet Coatings: *Lalgudi V. Ramanathan*[1]; [1]Cidade Universitaria, IPEN, Matls. Sci. & Tech. Ctr., Av. Prof. Lineu Prestes 2242, São Paulo 05508-000 Brazil

An apparatus consisting of a fluidized bed of erodent particles through which a specimen assembly enters and leaves at periodic intervals has been used to determine the erosion-oxidation (E-O) behavior of various steels and HVOF sprayed alloy and cermet coatings of Ni20Cr, WC-20Cr7Ni and Cr3C2-Ni20Cr on a steel substrate. Alumina powder (~200 μm) was used as the erodent. The E-O tests were carried out in the range 100-850ºC, with particle impact velocities of 3-19 ms-1 and impact angle of 90º. The erosion-oxidation behavior was determined as wastage, as a function of temperature. The effect of chromium content on E-O behavior of the steels was determined. Above 500ºC, wastage increased with temperature, reaching a maximum at 700ºC and then decreased with further increase in temperature. The surface roughness of the coatings was determined to corroborate the E-O regimes.

3:25 PM Break

3:40 PM Invited
Recent Advances in Thermal Spray Processing of Materials: *Sanjay Sampath*[1]; [1]State University of New York, Ctr. for Thermal Spray Rsch., Stony Brook, NY 11794-2275 USA

Thermal sprayed coatings is a highly versatile tool for surface engineering. These coatings have found wide spread applicability in both aero and power generation turbine engines. In the past, coatings have been principally added as an after thought with the goal of life extension of engineering components, however, as the need for prime reliant coatings grow, so do the requirement of reliability and reproducibility. Depositing reproducible coatings is an implicit requirement for the application of prime reliant coatings, so as to reduce/eliminate infant mortality of coatings. Advances process diagnostics and property extraction for these lamellar materials has significantly enhanced our understanding of the process dynamics during thermal spray. This has enabled improving performance as well as assessing process sensitivity, establishing optimizing protocols and reducing variability. In this presentation, we will discuss a fundamental approach to understanding the process through complete plume diagnostics, singe and multiple point measurement of particle state and measuring deposit properties insitu. The outcome of this effort is displayed in what is referred to as 1st order (process-particle interactions) and 2nd order (particle-coating interactions) process maps. For example, these maps allow reducing variability for deposition of ceramic thermal barrier coatings. Implications of these maps towards industrial applications will be addressed. Acknowledgements: Supported by the NSF MRSEC program DMR 0080021.

4:05 PM
Plasma Sprayed Multicomponent Coatings for Textile Machinery with Emphasis on Self Lubrication: *Suman Saurabh*[1]; Krishna M. Gupt[1]; [1]Indian Institute of Technology Bombay, Dept. of Metallurgl. Engrg. & Matls. Sci., Rm. No-B402, Hostel-13, Powai, Mumbai, Maharastra 400076 India

All textiles manufacturing machines encounter continuous friction between machine components and moving thread. It leads, among other things to wear of components, thus causing mis-alignment, vibration and damage to fibers and yarn. Although lubrication is universally used to avoid friction related wear, but in case of fiber processing lubrication can deteriorate fiber/fabric quality. The development of self lubricating multicomponent coatings with solid lubricants is an answer to industry's increasing demand for alternatives to oil lubricating systems. The present work aims to study and analyze the tribological properties of plasma sprayed (Cr2O3.CaF2, Cr2O3.BaF2, Cr2O3.Ag2O) coatings for textile machinery components. Effect of addition of solid lubricants (CaF2, BaF2, Ag2O) and its self lubricating nature were studied. The influence of different process parameters on coating properties were studied by SEM and TEM analysis. Coatings were analyzed and were found to have much superior wear resistance and lower friction coefficient.

4:20 PM
Measurements of Residual Stresses in Plasma-Sprayed Hydroxyapatite Coatings on Titanium Alloy: *Yungchin Yang*[1]; [1]National Taipei University of Technology, Matls. & Minl. Resources Engrg., 1, Sec. 3, Chung-Hsiao E. Rd., Taipei 106 Taiwan

In an attempt to investigate the stress state and stress distribution in hydroxyapatite coatings (HAC), the residual stresses in thick HACs

on titanium alloy were studied by the materials removal method, as a function of cooling media during spraying. In addition, and the x-ray diffraction sin2?é method was adopted as a comparison. The Young's moduli of hydroxyapatite coatings were measured on separated free coating by a three-point bending test. The results show that the measured Young's modulus of the HACs with an average of 22.8 GPa was found to be much lower than the theoretical value of bulk HA. During measurements by the materials removal method, on the three types of HACs using different cooling media, the interface between the HAC and Ti-substrate displays higher residual stress than the top surface of HAC. The residual stresses in all the HACs measured by both methods were in a compressive mode, and the residual stresses on the top surface obtained from the two methods are consistent. It was also found that the compressive residual stresses on the top surface of HAC and at the interface between the coating and the substrate both increased with increasing temperature of the HAC. Therefore, the coating temperature, and the effect of varying the cooling media during plasma spraying, had a significant effect on the residual stress states of the HACs.

4:35 PM
Surface Mechanical Alloying Between an Aluminum Plate and Oxide Powders: *Laszlo Takacs*[1]; Aghasi R. Torosyan[2]; [1]University of Maryland, Dept. of Physics, 1000 Hilltop Cir., Baltimore, MD 21045 USA; [2]National Academy of Science, Inst. of Gen. & Inorganic Chmst., Yerevan 375051 Armenia

Mechanical alloying is usually carried out via ball milling a mixture of components in powder form. It is also possible to place a plate into the milling chamber and mill only the other component(s), pressing their particles onto or into the surface of the plate and inducing surface modification and alloying. As the processes at a macroscopic surface are less stochastic than between the powder particles during ordinary mechanical alloying, it is easier to understand the microscopic details of mechanical alloying in the powder-plate geometry. It is also possible to prepare a variety of coatings this way. In the present report, the interaction between an aluminum surface and PbO and WO3 powders is discussed from both points of view. It is shown that both mechanical and chemical processes contribute to the formation of the surface layer.

4:50 PM
Design and Tuning of a Micro Vacuum Plasma Spray System: *W. Scott Crawford*[1]; Mark A. Cappelli[1]; Friedrich B. Prinz[2]; [1]Stanford University, Mechl. Engrg., Bldg. 520, Rm. 520-I, Stanford, CA 94305-3032 USA; [2]Stanford University, Mechl. Engrg., Matl. Sci. and Engrg., Bldg. 530, Room 220, Stanford, CA 94305-3030 USA

A system has been developed and refined for small-scale vacuum plasma spraying of metals. This table-top system operates at arc power 1.5-3 kW and deposition rates below 0.1 g min^{-1}. System design is described with attention to issues heightened at these low levels of power and flow rate. These issues include design and operation of powder feeder and powder flow path, injector design, and momentum interactions between plasma jet and obliquely injected stream of powder and carrier gas. Tuning of operating conditions is also described, by a combination of system decomposition, scaling analysis and experiment. Key experiments included planar laser scattering for imaging of particle trajectories. Coating materials studied include 316L stainless steel and titanium alloy.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session VI

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Wednesday PM Room: 3003
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Chris Wolverton, Ford Motor Company, Physics & Environml. Sci., Dearborn, MI 48121 USA; J.-C. Zhao, GE Global Research, Niskayuna, NY 12309 USA

2:00 PM Opening Remarks

2:05 PM Invited
Precipitates in Ni-Rich Alloys: *Gernot Kostorz*[1]; [1]ETH Zurich, Angewandte Physik, Zurich 8093 Switzerland

A survey will be given on the numerous studies of phase separation in Ni-rich solid solutions initiated at the author's laboratory. Especially for Ni-Al and Ni-Ti, small-angle scattering of neutrons, diffuse scattering of neutrons and X-rays, and transmission electron microscopy have been combined to follow the transformation from the early stages. While in Ni-Al, initially coherent $L1_2$-ordered precipitates represent the stable decomposition product, they appear as a metastable phase in Ni-Ti. In both cases, elastic interactions lead to preferred alignments of the precipitates along the elastically soft directions, but there are also important differences in shape, chemical composition, degree of order, and temporal evolution of precipitates. While the alloys decompose, short-range order is also established in the matrix phases. From the wealth of experimental data, a comprehensive description of the differences and similarities of the two systems emerges.

2:30 PM Invited
Coarsening of Ni-Al Solid Solution Precipitates in a γ (Ni_3Al) Matrix; Contrasting Behavior Between Inverse and Conventional Alloys: Y. Ma[1]; *A. J. Ardell*[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095-1595 USA

Coarsening of γ (Ni-Al solid solution) precipitates in a γ' (Ni_3Al) matrix (the inverse alloy) was investigated in 5 alloys aged at 650°C. The rate constant for the kinetics of particle growth increases very rapidly as the equilibrium volume fraction, f_e, increases. This normal behavior contrasts dramatically with the coarsening of γ' precipitates in conventional Ni-Al alloys, wherein the rate constant is either independent of, or decreases anomalously, as f_e increases. The dependence of the rate constant on f_e in the inverse alloy agrees quantitatively with the predictions of the old MLSW theory of Ardell. Coalescence of γ precipitates is very easy, in stark contrast to the strong resistance to coalescence of γ' precipitates in conventional alloys. We regard this finding as indirect proof of the important role of anti-phase relationships during coarsening of γ' precipitates in conventional alloys, postulated by Wang and Khachaturyan.

2:55 PM
Experimental Study of Microstructural Evolution in Coherent System: *Y. S. Yoo*[1]; D. Y. Yoon[2]; [1]Korea Institute of Machinery and Materials, Dept. of Matls. Procg., 66 Sangnam-dong, Changwon, Kyungnam 641-010 S. Korea; [2]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., Daejon 305-701 S. Korea

The g/g' system in Ni base alloys shows various microstructural phenomena including alignment, splitting, morphological instability, and solid state dendrite. Experimental conditions for these microstructural evolutions are presented. Possible origin and mechanism of these phenomena are also discussed and compared with theoretical works. Initial precipitate density seems to be a crucial variable in determining the shape and distribution of gamma prime affecting both the strain field and the diffusion field of each particle. By deeply

etching the matrix and observing under a scanning electron microscope, the three dimensional precipitate morphologies are determined accurately.

3:10 PM Invited
Role of Composition During the Chessboard Pattern Formation in Co-Pt Alloys: *Y. Le Bouar*[1]; A. Loiseau[1]; A. G. Khachaturyan[2]; [1]LEM, CNRS/ONERA, 29 Ave. de la Division, Leclerc, BP 72, 92322, Châtillon France; [2]Rutgers University, Dept. of Ceram. & Matls. Engrg., Piscataway, NJ 08855-0909 USA

Co-Pt Binary alloys form at high temperature a FCC solid solution, and order on this lattice to form either the cubic L1_2 or the tetragonal L1_0 structure. For a small concentration range, the two ordered structure coexist at equilibrium, and we observe the formation of a chessboard-like microstructure. In this work, we present both a experimental investigation based on TEM observations and a phase field modelling of the chessboard pattern formation. We focus on the the role of the composition on the microstructural evolution. We show that the microstructural pattern is very sensitive to the concentration and that very surprising transient patterns can appear during an isothermal annealing.

3:35 PM
A Combined Study of Solid State Phase Changes in Steel Through Confocal Microscopy and Orientation Imaging: *Eric D. Schmidt*[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

A Confocal Scanning Laser Microscope (CSLM) has been used to directly observe austenite formation during heating and ferrite formation during cooling. The final microstructure can then be analyzed via electron backscattering diffraction (EBSD) to determine the crystallographic orientation relationship of the various ferrite morphologies—primarily allotriomorphic and Widmanstatten. These ferrite orientations can then be used to determine the prior austenite grain locations and orientations. A detailed and accurate analysis of the phase change kinetics is then possible by comparing direct observations and orientation relationships from the exact same location. The effect of heating rate, cooling rate, annealing temperature, annealing time, and atmosphere have all been explored in this study.

3:50 PM Break

4:15 PM Invited
Temporal Evolution of the Nanostructure of Ni-Al-Cr Base Alloys: *David N. Seidman*[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; Ronald D. Noebe[2]; [1]Northwestern University, Matls. Sci. & Engrg., Cook Hall, 2220 Campus Dr., Evanston, IL 60208-3108 USA; [2]NASA, MS 49-3, Glenn Research Ctr., 21000 Brookpark Rd., Cleveland, OH 44135 USA

The temporal evolution of the nanostructure of Ni-Al-Cr base alloys is studied employing three-dimensional atom-probe (3DAP) and transmission electron microscopies. The decomposition kinetics of the gamma (FCC)phase into the gamma and gamma prime (L12 structure), which is a first-order phase transformation, is followed through the nucleation, growth and coarsening stages from its genesis, initially employing radial distribution functions (RDFs) to detect local ordering in the absence of detectable gamma prime precipitates. The smallest detectable gamma prime precipitates contain about 20 atoms. The temporal evolution is followed by measuring the mean precipitate radius, the number density, and the supersaturations of all the solute elements in both the gamma phase and gamma prime precipitates. In addition, the width of the gamma/gamma prime interface and its interfacial free energy is determined from the 3DAP microsope observations. The coarsening process involves coalescence of gamma prime precipitates, in addition to classical Ostwald ripening.

4:40 PM Invited
Numerical Calculation and Atom Probe Analyses of Kinetics in Ni-Al-V System: *Hélèna Zapolsky*[1]; Sebastien Ferry[1]; Didier Blavette[1]; Long-Qing Chen[2]; [1]University of Rouen, GPM, UMR 6634, Ave. de l'univ., BP 12, Saint-Etienne-du-Rouvray 76801 France; [2]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., 102 Steidle Bldg., Univ. Park, PA 16802 USA

The ternary Ni-Al-V compound is a rare system where in some temperature domain the three different phases coexist at equilibrium. As follow from the experimental and theoretical phase diagram at 800°C, the ordered Ni3Al phase (with L12 structure) coexists with the ordered Ni3V phase (with the DO22 structure) and with the disordered matrix which have fcc lattice. An important feature of the current study is to analyze coarsening data in ternary Ni-based alloys. The morphological evolution and coarsening kinetics of ordered intermetallic precipitates with coherency stress were studied using the Önsager-type microscope diffusion equations and three dimensional atom probe (3DAP) analyses. The emphasis is on the effects of precipitate volume fraction. Specifically, we predict the variation of the rate constants - of coarsening with precipitate volume fraction. Comparison of numerical simulation results with experiments shows good quantitative agreement.

5:05 PM
Computer Simulation of Phase Decomposition in Fe-Cu-Mn-Ni Quaternary System Based on the Phase-Field Method: *Toshiyuki Koyama*[1]; Hidehiro Onodera[1]; [1]National Institute for Materials Science, Computatl. Matls. Sci. Ctr., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

The phase decomposition in Fe-Cu-Mn-Ni quaternary system during isothermal aging is simulated based on the phase field method. Since the chemical free energy used in this simulation is obtained from the CALPHAD database, i.e. the thermodynamic database of phase diagrams, the calculated microstructure changes are directly related to the phase diagram of the Fe-Cu-Mn-Ni system. At the early stage of aging, the Cu-rich zone with bcc structure begins to nucleate, and the component X (=Mn, Ni) is partitioned to the Cu-rich phase. When the Cu composition in the precipitate reaches equilibrium, the component X inside the precipitates moves toward to the interface region between the precipitate and matrix. Finally, the shell structure that the Cu precipitates surrounded by the thin layer with high concentration of component X appears. This microstructure change is reasonably explained by considering the local equilibrium at the diffused interface region of nano-particles.

5:20 PM
Compositional Pathways in a Model Ni-Al-Cr Alloy: *Chantal K. Sudbrack*[1]; Ronald D. Noebe[2]; David N. Seidman[1]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., 2220 Campus Dr., Evanston, IL 60208 USA; [2]NASA Glenn Research Center, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Due to complex interactions in multi-component metallic alloys, as well as experimental limitations, insight on the decomposition pathways of isothermal precipitation from a supersaturated solid solution is limited. We present a detailed three-dimensional atom-probe microscope study of the earliest stages of decomposition, γ (fcc) —> γ (fcc) + γ' ($L1_2$ structure), in a model Ni-Al-Cr superalloy. Short-range order and clustering tendencies are evaluated from the direct space images. It is found that Cr atoms are trapped in the forming precipitates, and that an increased solubility of Al can be attributed to capillarity effects. The composition trajectory of the matrix follows the tie-lie, while the precipitates' trajectory does not.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Ultrafine-Grained Materials II

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Wednesday PM Room: 3024
February 16, 2005 Location: Moscone West Convention Center

Session Chairs: Terry R. McNelley, Naval Postgraduate School, Mechl. & Astronautical Engrg., Monterey, CA 93943-5146 USA; Kenong Xia, University of Melbourne, Dept. of Mechl. & Mfg. Engrg., Parkville, Victoria 3010 Australia; Yuntian T. Zhu, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ruslan Z. Valiev, Ufa State Aviation Technical University, Inst. of Physics, of Advd. Matls., Ufa 450000 Russia

2:00 PM
Lattice Defect Investigation of SPD Cu by Means of X-Ray Line Profile Analysis, Calorimetry and Electrical Residual

Resistivity: Erhard Schafler[1]; *Gerd Steiner*[1]; Elena Korznikova[1]; Michael Kerber[1]; Michael J. Zehetbauer[1]; Leonhard F. Zeipper[2]; [1]University of Vienna, Matls. Physics Inst., Boltzmanngasse 5, Wien A-1090 Austria; [2]ARC Seibersdorf Research GmbH, Matls. Rsch., Seibersdorf, Austria A-2444 Austria

Samples of pure Cu have been subjected to different modes of SPD (ECAP, HPT), and deformation induced defects such as vacancies, dislocations and local internal strains have been studied by means of Calorimetry, Residual Electrical Resistivity (RER) and X-Ray Line Profile Analysis (XPA). The densities and arrangements of defects have been measured as a function of deformation degree and - in case of HPT - extent of hydrostatic pressure. It has been found that both the vacancy concentration and the dislocation density are higher than with usual deformation like torsion and rolling, and that they still increase with increasing deformation degree as well as increasing hydrostatic pressure. For both quantities, however, a saturation has been observed with respect to deformation and hydrostatic pressure applied. With the help of measurements of internal strains, the saturation effect is discussed in terms of static and dynamic recovery.

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Defect Based Micromechanical Modelling and Simulation of NanoSPD CP-Ti in Post-Deformation: Leonhard F. Zeipper[1]; *Michael J. Zehetbauer*[2]; [1]ARC Seibersdorf Research GmbH, Matls. Rsch., 2444-Seibersdorf Austria; [2]University of Vienna, Matls. Physics Inst., Boltzmanngasse 5, 1090 Vienna Austria

The paper concerns the modelling and simulation of dislocation based hardening at room temperature compression after Equal Channel Angular processing of CP-Ti (grade2). For the first time the post-deformation behaviour is successfully simulated for a nanoSPD material by the use of the Zehetbauer model, which was already applied on cubic materials during conventional and Severe Plastic Deformation. Unsuccessful calculations by the Estrin-Toth model support the numerical interpretations given in this paper. Based on the experimental data of uniaxial compression and the measured total dislocation density, the hardening behaviour is characterized and physical quantities are calculated. The results, like the calculated cell size, the dislocation evolution in the cell interior and the cell walls as well as the deformation induced vacancy concentration versus strain are discussed. Experimental data from TEM and residual electrical resistivity measurements confirm the simulation results.

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Tensile Properties of Consolidated Nanocrystalline Cu: *Evan Ma*[1]; Sheng Cheng[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 3400 N. Charles, Baltimore, MD 21218 USA

Over the past fifteen years, nanocrystalline (nc) Cu has been a primary model material for understanding the deformation behavior of nanocrystalline metals. To obtain truly nc grain sizes (well below100 nm), the nc Cu was usually obtained in powder form, via the gas phase condensation route for example. A consolidation step is then needed to obtain bulk compacts for mechanical testing. Due to the residual porosities, the intrinsic tensile behavior of nc Cu remains unclear so far. In fact, all the consolidated nc Cu (and other nc metals as well) showed virtually no ductility in tension. The strength values vary over a wide range and are often lower than expected. Many models have appeared over the years trying to fit to these experimental data, causing major confusions in understanding the intrinsic nc behavior. We have prepared nc Cu powder through ball milling at 77 K. The severe plastic deformation processing yields powders with internal grain sizes well below 100 nm. We used an in situ consolidation technique to produce fully dense bulk nc Cu millimeters in thickness. In this talk, we report the high tensile strength and the ductility achieved, as well as the strain rate and temperature dependence. The impurity effects on the properties above are examined. We also analyze the deformation localization observed, and discuss the results in comparison with those of other nc metals and in light of the proposed/established deformation mechanisms/modes in nc metals.

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Severe Plastic Deformation Through Adiabatic Shearing Banding in Fe-C Steels: *Donald R. Lesuer*[1]; Chol K. Syn[1]; Oleg D. Sherby[2]; [1]Lawrence Livermore National Laboratory, L-175, Livermore, CA 94551 USA; [2]Stanford University, Dept. of Matls. Sci. & Engrg., Stanford, CA 94305 USA

Severe plastic deformation takes place within adiabatic shear bands in iron-carbon steels. Strains in the order 5 or greater are commonly observed. These shear bands form under conditions of high strain rate in excess of 1000 s-1. Studies on shear band formation in an ultrahigh carbon steel (1.3%C) are described in the pearlitic and martensitic conditions. Extremely high hardness is obtained in the shear band in excess of Vickers Hardness of 10 GPa (equivalent to 4000 MPa tensile strength). A mechanism is described to explain the high strength based on phase transformation to austenite from adiabatic heating resulting from severe deformation. Rapid re-transformation leads to an ultrafine ferrite grain size containing carbon principally in the form of nanosize carbides. It is proposed that the same mechanism explains the ultrahigh strength of iron-carbon steels observed in ball-milling, ball drop tests and in severely deformed wires. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

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Nanostructure Formation in Pure Copper Deformed by Rolling: Mariana Gheorghe[1]; *Naresh N. Thadhani*[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Erskine Love Mfg. Bldg., Atlanta, GA 30332-0245 USA

Severe plastic deformation of copper at liquid nitrogen temperature suppresses dynamic recovery and allows formation of stable nanocrystalline structure. The effect of rolling conditions on the strength and microstructure of copper was investigated in this work. Pure copper (99.99%) was rolled to similar total reductions by single or multiple passes, at ambient and liquid nitrogen temperatures. The specimens rolled at liquid nitrogen temperature (-150°C) had strength and hardness higher than those rolled at room temperature. The crystallite size was found to decrease to 20-40nm, as the degree of deformation at liquid nitrogen reached 100%. Such a reduction in grain size was not obtained in the room temperature rolled samples due to effects of recovery and recrystallization. In this paper, the correlation of grain size, retained strain, and dislocation density and character (determined using x-ray diffraction and transmission electron microscopy), with microstructure evolution and properties, will be presented. The authors acknowledge the funding available through AFOSR Grant No. 1606U81 (Craig S. Hartley, program monitor).

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Mechanical Behavior of a Mg AZ61 Alloy Processed by Accumulative Roll Bonding: *Jorge Antonio del Valle*[1]; María Teresa Pérez-Prado[1]; Oscar Antonio Ruano[1]; [1]CENIM,CSIC, Physl. Metall., Gregorio del Amo,8, Madrid 28040 Spain

Research on the processing, structure and mechanical behavior of nanocrystalline ($d < 100$ nm) and ultra-fine grained ($100 \text{ nm} < d < 1$ mm) materials has thrived. These materials have promising structural properties, such as elevated strength, good wear resistance and high toughness, as well as the potential for superplasticity at low temperatures and high strain rates. In this work an ultra-fine grained AZ61 Mg alloy has been fabricated via accumulative roll bonding (ARB), using large thickness reductions per pass. The resulting microstructure is analyzed by optical microscopy as well as macro- and microtexture analysis in order to study the mechanisms responsible for grain refinement. The mechanical behavior of the processed material is investigated, both at room temperature and at high temperatures, with the aim of establishing a relationship between the initial microstructure and the operative deformation mechanisms.

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High Temperature Behavior of a Cryomilled Ultrafine-Grained Al-7.5%Mg Alloy: *Bing Q. Han*[1]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

In the present study, the tensile behavior at temperatures of 298 K to 673 K of an ultrafine-grained Al-7.5%Mg alloy processed by consolidating cryomilled powders was investigated. Microstructure of the Al-Mg alloy shows a high thermal stability with the insignificant grain growth after high-temperature testing. The Al-Mg alloy exhibits an intrinsic behavior similar to that of other mechanically alloyed aluminum alloys, but different from that of classic superplastic aluminum alloys although the grain size of the cryomilled Al-Mg alloy is much smaller than that of superplastic aluminum alloys. The possible effect on the ductility and deformation mechanisms at elevated temperatures of the cryomilled Al alloys is discussed.

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Characterization of Severely Deformed Cu/Ag and Cu/Zr Nanolamellar Structure: S. Ohsaki[2]; S. Katoh[3]; N. Tsuji[3]; *K. Hono*[1]; [1]National Institute for Materials Science, 1-2-1 Sengen, Tsukuba 305-0047 Japan; [2]University Tsukuba, Grad. Sch. of Pure & Appl. Scis., Tsukuba 305-8571 Japan; [3]Osaka University, Dept. Adaptive Machine Sys., Suita, Osaka 565-0871 Japan

To investigate the nanostructure evolution process by the accumulative role bonding (ARB) process of two phase lamellar structures, we have selected a Ag-Cu eutectic alloy and Cu/Zr stacked plates as model systems. A round bar of a Ag-39.5at.%Cu eutectic alloy with a diameter of 10 mm was first cold-rolled and then ARB processed to cumulate a large plastic strain of 6.8. 5 pieces of Cu sheets 0.2 mm thick and 4 pieces of Zr sheets 0.2 mm thick were mutually stacked and then roll-bonded, so that the total composition of the stacked material was Cu-45at%Zr. The ARB process was repeated for the multilayered material up to a total strain of 13.6. An intermixing of Ag and Cu layers was observed in the Ag-Cu eutectic lamellae. 3DAP analysis showed that the compositions of Cu and Ag lamellae are 60at.%Cu and 5 at.%Cu, respectively. Although such a mixing was observed in Cu/Ag lamellae, no amorphization was confirmed. On the other hand, the Cu/Zr stacked lamellae deformed to a total strain of 13.6 exhibited various interesting local nanostructures such as the amorphous/nanocrystal composite and the amorphization of some of the Zr lamellae. In both systems, substantial mixing was observed in the shear bands, where large shear deformation localized without any remains of the lamellar structure.

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Large Strain Deformation and Ultra-Fine Grained Materials by Machining: *Alexander H. King*[1]; Srinivasan Chandrasekar[2]; W. Dale Compton[2]; Kevin P. Trumble[1]; Travis L. Brown[2]; Seongeyl Lee[2]; Balkrishna C. Rao[2]; M. Ravi Shankar[2]; Srinivasan Swaminathan[2]; [1]Purdue University, Sch. of Matls. Engrg., 501 Northwestern Ave., W. Lafayette, IN 47907-2044 USA; [2]Purdue University, Sch. of Industl. Engrg., 315 N. Grant St., W. Lafayette, IN 47907-2023 USA

The characteristics of deformation field in plane strain machining, including the distributions of strain, strain rate and strain gradient, are reviewed with reference to recent experimental and analytical results. The nature of this deformation field is compared and contrasted with that prevailing in Equal Channel Deformation processing. The creation of ultra-fine grained materials by high strains produced in machining is demonstrated in pure metals, age hardening aluminum alloys, carbon steels, high temperature materials and amorphous alloys. These results indicate that machining provides a simple experimental configuration for studying various effects of large strain deformation in materials.

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Electron Microscopy of Mechanically Alloyed Oxide-Dispersion-Strengthened 14YWT Ferritic Steel: *James Bentley*[1]; David T. Hoelzer[1]; Dorothy W. Coffey[1]; Kathy A. Yarborough[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA

Advanced characterization techniques have revealed that the exceptional high-temperature mechanical properties of a new class of mechanically alloyed (MA) oxide-dispersion-strengthened (ODS) ferritic steels are due to a high concentration (~1 x 10^{24} m^{-3}) of small (a few nanometers diameter) Ti-enriched oxide clusters that are resistant to coarsening even at 1200°C. Pre-alloyed powders of the base alloy are ball-milled with yttria powder (to incorporate supersaturations of oxygen, yttrium and vacancies) and then extruded at typically 850°C to produce Fe-14.2%Cr-1.95%W-0.22%Ti-0.25%Y_2O_3, designated MA 14YWT. In order to complement atom probe tomography and small-angle neutron scattering, energy-filtered transmission electron microscopy (EFTEM) has been used to image the clusters and reveal intergranular segregation. Additional analytical electron microscopy has been used to supplement the EFTEM. Examination of as-milled powders was enabled by focused ion beam (FIB) milling for TEM specimen preparation. The electron microscopy results have provided useful guidance for helping to optimize processing conditions.

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Computational Description of Nanocrystalline Deformation Based on Crystal Plasticity: Hsueh-Hung Fu[1]; David Benson[1]; *Marc A. Meyers*[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

The effect of grain size on the mechanical response of polycrystalline metals was intestigated computationally and applied to the nanocrystalline domain. A phenomenological constitutive description is adopted to build the computational crystal model. Two approaches are implemented. In the first, the material is envisaged as a composite: the grain interior is modeled as a monocrystalline core surrounded by a mantle (grain boundary) with a lower yield stress and higher work hardening rate response. Both quasi-isotropic and crystal plasticity approaches are used to simulate the grain interiors. The grain boundary is modeled either by an isotropic Voce equation (Model I) or by crystal plasticity (Mode II). Elastic and plastic anisotropy are incorporated into this simulation. An Implicit Eulerian Finite Elemene Formulation with von Mises plasticity or rate dependent crystal plasticity is used to study the non-uniform deformation and localized plastic flow. The computational predictions are compared with experimentally determined mechanical response of copper with grain sizes of 1 micro meter and 26 nm. Shear localization is observed during work hardening in view of the inhomogeneous mechanical response. In the second approach, the use of a continuous change in mechanical response, expressed by the magnitude of the maximum shear stress orientation gradient, is introduced. It is shown that the magnitude of the gradient is directly dependent on grain size. This gradient term is inserted into a constitutive equation that predicts the local stress-strain evolution.

5:00 PM

Severe Plastic Deformation Within a Friction-Stir Weld: *John F. Bingert*[1]; Richard W. Fonda[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Naval Research Laboratory, Code 6324, 4555 Overlook Ave. SW, Washington, DC 20375 USA

The deformation gradient within the stir zone of a friction-stir weld results in locally severe plasticity. The dominant stress state is simple shear resulting from the interaction between a rotating cylindrical tool and the workpiece. This shear, combined with rigid rotations due to bulk material movement from the baseplate, introduces significant microstructural evolution. Friction-stir butt welds in 2195 and 2519 aluminum alloys were interrogated by optical and electron microscopy to elucidate some of the details of the structural changes. In particular, the post-mortem microhardness, precipitation reactions, and crystallographic texture were characterized to provide insight into the thermomechanical history experienced during the weld. Consequences of rigid-body spin and elevated-temperature shear deformation will be explored in relation to their influence on the observed texture. Experimental results were compared to polycrystalline plasticity simulations performed to predict texture evolution in the stir zone. LA-UR-04-4936.

5:15 PM

Bimodal Structured Bulk Nanocrystalline Al-Mg Alloys: *Zonghoon Lee*[1]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; [2]University of California, Dept. of Chem. Engrg. & Matls. Sci., Bainer Hall, Davis, CA 95616-5294 USA

The microstructure, mechanical properties and deformation mechanism of bimodal structured nanocrystalline Al-Mg alloys were investigated. Grain refinement was achieved by cryomilling of atomized Al-Mg powders, and then cryomilled nanocrystalline powders blended with 15%, 30% and 50% unmilled coarse-grained powders were consolidated by hot isostatic pressing followed by extrusion to produce bulk nanocrystalline alloys. Bimodal bulk nanocrystalline Al-Mg alloys, which were comprised of nanocrystalline grains separated by coarse-grain regions, show balanced mechanical properties of enhanced ultimate strength and reasonable ductility/toughness compared to conventional Al-Mg alloys and other nanocrystalline metals. The investigation of tensile test, TEM analysis and finite element analysis suggests unusual deformation mechanisms and interactions between ductile coarse-grain and nanocrystalline regions. The bimodal microstructure and its ductility toughening effect inspire us to design microstructures and to select processing parameters leading to optimal performance characteristics on various nanocrystalline metals.

5:30 PM

The Use of Severe Plastic Deformation for Consolidating Nanostructured Metallic Powders: *Deliang Zhang*[1]; Nathan J. Scott[1]; Carl C. Koch[2]; [1]University of Waikato, Dept. of Matls. & Process Engrg., PB 3105, Hamilton, Waikato 2001 New Zealand; [2]North Carolina State University, Dept. of Matls. Sci. & Engrg., Raileigh, NC 27695-7907 USA

Certain severe plastic deformation processes such as equal channel angular pressing have been widely used to process bulk metallic materials to generate ultra fine grained structures with grain sizes greater than 100nm. In the meantime, high energy mechanical milling, as another severe plastic deformation process, has been widely used to produce nanostructured metallic powder materials with grain sizes as small as a few nanometres, but turning the nanostructured powders into bulk nanostructured materials is a great challenge. Severe plastic deformation, on the other hand, is also capable of producing a large area of new surfaces of powder particles due to the large amount of shear deformation. Under certain conditions, the large area of new surfaces can be well utilised to cause cold welding, or cold sintering in powder metallurgy term. This paper will critically examine the relationships between processing conditions, microstructure and quality of

a few nanostructured copper based metallic materials produced by consolidating nanostructured powders using severe plastic deformation processes such as high energy mechanical milling, rolling and forging, and discuss the associated scientific and technological issues.

TMS Featured Presentations

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Wednesday PM Room: 2016
February 16, 2005 Location: Moscone West Convention Center

Session Chair: Richard Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

2:00 PM
Bauxite Mining Sustainability: *Patrick Riley Atkins*[1]; [1]Alcoa, Inc., 390 Park Ave., New York, NY 10022 USA

Beginning in 1990, The international Aluminum Institute began a program to report on the bauxite mineing and rehabilitation activities of the worldwide industry. A survey process was initiated and reports were published in 1992, 1998 and 2004. The recently published 2004 report includes extensive data on mines that represent over 70% of the world's output of bauxite. This paper describe the latest report and will focus on the mine rehabilitation technologies,progress and performance, on company/community interactions and the sustainability goals of the industry.

2:25 PM
On the Preparation and Stability of Scorodite: *George P. Demopoulos*[1]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Scorodite is advocated as a viable option for the fixation of arsenic from aqueous process effluents, especially for arsenic-rich and iron-deficient solutions. It has a high arsenic content, it requires stoichiometric amounts of iron, and it has excellent dewatering and disposal characteristics. Because of its high degree of crystallinity and small specific surface area, scorodite is also, very importantly, thought to have high inherent stability at least from a kinetic point of view. As a mineral, scorodite can be found in a wide variety of geological settings. This suggests that it is stable under specific weathering conditions. Hence our interest in designing a cost-effective process for returning arsenic to the environment in this mineral form. In this paper, (1) the preparation of scorodite in lime neutralisation ?type circuits is discussed and (2) the long term stability of scorodite is evaluated in the light of newly generated accelerated ageing kinetic data.

2:50 PM
Use of CO2-Snow for Protecting Molten Magnesium from Oxidation: Friedrich-Wilhelm Bach[1]; *Alexander Karger*[1]; Christoph Pelz[2]; Mirko Schaper[1]; [1]University Hanover, Inst. for Matls. Sci., Schoenebecker Allee 2, 30823 Garbsen Germany; [2]Linde Gas AG, Unterschleissheim Germany

When processing magnesium alloys, prevention of oxidation of the liquid metal is of prime importance, because of the high oxygen affinity of molten magnesium. A special danger of environmental pollution occurs from the usage of protective gases for molten magnesium. The protective gas SF6, which is widely used nowadays, increases the greenhouse effect due to it's GWP of approximately 23900 relative to CO2. At the Kyoto summit in 1997, this gas was detected as one of six gases to be restricted in use. The environmentally friendly alternatives are being examined by authors of this paper. The research goals of this group are to develop and to evaluate new methods for protecting the surface of magnesium melts. One possible alternative is covering the magnesium melt with CO2-snow. The current results will be presented in the following paper.

3:15 PM
Identifying Economic and Scrap Reuse Benefits of Light Metals Sorting Technologies: Preston P. Li[1]; Sigrid Guldberg[2]; Hans Ole Riddervold[2]; *Randolph E. Kirchain*[1]; [1]Massachusetts Institute of Technology, Matls. Sys. Lab., Rm. E40-421, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]Hydro Aluminum, Drammensveien 264, N-0240, Oslo Norway

The changing pattern of aluminum scrap usage has created material reuse challenges for the industry. For instance, mixed scraps consisting of wrought and cast alloys often cannot be directly re-melted and reused due to compositional incompatibility. Various new sorting technologies promise to address these challenges. It is critical to understand how, when, and to what extent sorting should be applied in different circumstances. This paper examines the use of linear programming methods to identify economically efficient sorting strategies and their impact on scrap usage. Economic efficiency was tested for various states of scrap material supply, finished good demand, sorting technology type, and sorting performance. The model can be used to identify optimized specific sorting schemes. The overall goal is to support industry decision-making regarding the application of sorting technologies to increase scrap use and lower production costs.

3:40 PM Break

3:50 PM
Developing an Atomic-Level Understanding of the Mechanisms that Govern CO2 Mineral Carbonation Reaction Processes: *Michael J. McKelvy*[1]; Andrew V.G. Chizmeshya[1]; Jason Diefenbacher[2]; Hamdallah Béarat[2]; R. W. Carpenter[1]; George Wolf[3]; [1]Arizona State University, Ctr. for Solid State Sci., Sci. & Engrg. of Matls. Grad. Prog., Tempe, AZ 85287 USA; [2]Arizona State University, Ctr. for Solid State Sci., Tempe, AZ 85287 USA; [3]Arizona State University, Dept. of Chmst. & Biochmst., Tempe, AZ 85287 USA

Mineral carbonation is an intriguing CO2 sequestration candidate technology, which produces environmentally benign and geologically stable materials. The primary challenge is economically viable process development. Serpentine and olivine minerals are exciting candidate feedstock materials, due to their wide availability, low-cost, and rapid mineral carbonation potential. Cost-effectively enhancing their carbonation rate is critical to reducing mineral sequestration process cost. We will discuss our recent research into the mechanisms that govern serpentine/olivine mineral carbonation reaction processes, including in situ observations of the mineral carbonation process and a novel mechanistic approach to enhance carbonation reactivity that avoids the cost of mineral activation. Our goal is to develop the necessary atomic-level understanding to engineer improved carbonation materials and processes to reduce process cost.

4:15 PM
Diffusion Paths and Interdiffusion Microstructures: Applications and Remaining Challenges: *John E. Morral*[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

Interdiffusion can influence the properties and cost of products that experience high temperature during processing or service (e.g. coated turbine blades, solid oxide fuel cells, carburized sun gears, etc.). Accordingly the ability to predict interdiffusion microstructures can be a useful alloy design tool. Software such as DICTRA can predict interdiffusion microstructures via diffusion paths and phase diagrams. Although useful for certain applications, both DICTRA and the theory of diffusion paths have serious limitations. Software for the Phase Field Method (PFM) requires more computer time, but has fewer limitations and can predict microstructures directly. Also the PFM takes into account both precipitate morphology and diffusion in precipitates, which yields a more accurate result. However without adequate databases and a better understanding of interdiffusion fundamentals, the value of these programs to alloy design will be limited.

4:40 PM
Deformation at the Nanometer and Micrometer Length Scales: Effects of Strain Gradients and Dislocation Starvation: *William D. Nix*[1]; Gang Feng[1]; Julia R. Greer[1]; [1]Stanford University, Dept. Matls. Sci. & Engrg., 416 Escondido Mall, Stanford, CA 94305-2205 USA

Size effects in plasticity are now well known. Plastic deformation in small volumes requires higher stresses than are needed for plastic flow of bulk materials. Here we review the various effects that appear to be responsible for this. The size dependence of the hardness of metals at the micron scale can be described in terms of the geometrically necessary dislocations or, correspondingly, the strain gradients, created in small indentations. But such accounts break down when the size of the deformation volume begins to approach the spacing of individual dislocations or when the crystal becomes dislocation starved. Nanoindentation of epitaxial films at the nanometer depth scale reveals irregular load-displacement curves. In this domain the nucleation of dislocations and plasticity under dislocation-starved conditions appears to be more important than strain gradients. Recent uniaxial compression experiments on tiny samples of gold made by focused ion beam machining and integrated circuit fabrication methods show strong size effects on plasticity, with sub-micron sized crystals showing remarkable strengths after plastic deformation. These experiments involve small deformation volumes and minimal strain gradients. These size effects may be explained by considering a process of strain hardening by dislocation starvation, wherein existing dislocations leave the crystal more frequently than they reproduce themselves by multiplication.

5:05 PM
Semiconducting and Piezoelectric Nanobelts, Nanosprings, and Nanorings: *Zhong Lin Wang*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

Nanowire and nanotube based materials have been demonstrated as building blocks for nanocircuits, nanosystems and nano-optoelectronics. Quasi-one-dimensional nanostructures (so called nanobelts or nanoribbons) have been successfully synthesized for semiconducting oxides of zinc, tin, indium, cadmium and gallium, by simply evaporating the desired commercial metal oxide powders at high temperatures. The belt-like morphology appears to be a unique and common structural characteristic for the family of semiconducting oxides with cations of different valence states and materials of distinct crystallographic structures. Using the technique demonstrated for measuring the mechanical properties of carbon nanotubes based on in-situ transmission electron microscopy, the bending modulus of the oxide nanobelts has been measured, and the nanobelt is shown to be a dual mode nanoresonator for NEMS technology. Field effect transistors and ultra-sensitive nano-size gas sensors, nanoresonators and nanocantilevers have also been fabricated based on individual nanobelts. Thermal transport along the nanobelt has also been measured. Very recently, nanobelts, nanorings and nanosprings that exhibit piezoelectric properties have been synthesized, which are potential candidates for nano-scale traducers, actuators and sensors. The discovery of single-crystal perfect nanorings and its "slinky" growth model will be presented.

5:30 PM
Aluminum Alloy Thermodynamics and Kinetics from First Principles: *Chris Wolverton*[1]; Vidvuds Ozolins[2]; [1]Ford Motor Company, Physl. & Environml. Sci., MD 3083/SRL, PO Box 2053, Dearborn, MI 48121-2053 USA; [2]University of California, Dept. of Matls. Sci., Los Angeles, CA 90095-1595 USA

We present an extensive survey of the thermodynamic and kinetic properties of binary Al alloys, as obtained from first-principles atomistic calculations. We consider a wide range of properties: 1) Energetic properties of ordered compounds, impurities, and mixing energies of solid solutions, 2) first-principles calculations of interatomic force constants, phonon spectra and vibrational entropies, 3) thermodynamic properties and solubility, and 4) kinetic quantities such as solute-vacancy binding, migration energies, and diffusion coefficients. We compare our results critically with experimental and CALPHAD databases to ascertain inaccuracies in the theoretical methods, and cases in which experimental data should be re-evaluated. In addition, the extensive nature of the database facilitates understanding the trends in energetic, thermodynamic, and kinetic properties. In addition, this large first-principles database should enable many future applications, such as improving existing CALPHAD databases, as well as providing key information to phase-field models of microstructural evolution.

Notes

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Bulk Metal Processing

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Thursday AM Room: 2009
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Cheml. Engrg. & Matls., E. Lansing, MI 48824 USA; Ibrahim Karaman, Texas A&M University, Dept. Mechl. Engrg., College Sta., TX 77843 USA

8:30 AM
Microstructural Engineering of Structural and Smart Materials Using Severe Plastic Deformation for Transportation Industry: G. Guven Yapici[1]; *Ibrahim Karaman*[1]; [1]Texas A&M University, Dept. of Mechl. Engrg., MS 3123, College Sta., TX 77843 USA

This talk will summarize our recent work on severe plastic deformation processing of several structural and smart materials using Equal Channel Angular Extrusion (ECAE). ECAE was achieved by extruding bulk materials through two channels of equal cross section intersecting at an angle of 90° achieving a simple shear deformation in a thin layer at the crossing plane of the channels. We will present the means of engineering of microstructure for high strength and ductility using severe plastic deformation in difficult to work materials such as Ti-6Al-4V, AISI 316L stainless steel and NiTi shape memory alloys and challenges and opportunities for transportation industry. The selected mechanical properties and microstructure will be exhibited. Ti-6Al-4V extrusions revealed that decreasing extrusion temperature and increasing number of passes decreases α plate size and grain size, leading to significant increase in tensile and compressive flow stresses at room temperature. Significant deformation twinning activity was observed for the first time in Ti-6Al-4V and 316L stainless steel at temperatures as high as 800°C (0.65 Tm!). ECAE of NiTi led to the observation of highly organized, twin-related nanograins in the high temperature phase which enhance cyclic stability and fatigue resistance of this alloy. The formation of well-organized twin-related nanograins via severe plastic deformation opens a new opportunity for twinning induced grain boundary engineering in B2 NiTi intermetallics. ECAE is applicable to numerous materials critical to industries ranging from aerospace to automobile and railroad. Therefore, it is of paramount importance to gain control over this processing method for producing desired end microstructures and mechanical properties in favor of transportation industry.

8:50 AM
Semisolid Microstructures of ECAP-Processed Al-9mass%Si-3mass%Cu Alloy: *Yuanwei Song*[1]; Yoshinori Nishida[1]; Teiichi Ando[1]; [1]Northeastern University, Mechl. & Industl. Engrg., 360 Huntington Ave., 334 Snell Engrg., Boston, MA 02115 USA

Effects of equal-channel-angular-pressing (ECAP) on the evolution of semisolid microstructures in an Al-9mass%Si-3mass%Cu alloy were investigated. ECAP-processed specimens held at the eutectic temperature (572¢X C) and semisolid temperatures for various holding times and quenched into water were examined. All of the specimens showed a microstructure indicative of formation of semisolid microstructures consisting of spheriodized ?Ñ grains fully separated by the liquid. The solid fraction of ECAP-processed specimens at the eutectic and semisolid temperatures stayed much higher than the low ?Ñ fractions expected for the ?Ñ ¡V liquid equilibrium in the alloy. Prior processing by ECAP refines the Si particles and homogenizes the cored ?Ñ matrix in the alloy, which helps to retard the formation of liquid. The sluggish liquid formation assures ample time for semisolid forming, providing a workable window for bulk-stock semisolid part forming with near eutectic aluminum die-casting alloys.

9:10 AM
Thermo-Mechanical Processing of a Cast 7XXX Al Alloy Modified with Sc: *Radhakrishna B. Bhat*[1]; Sesh Tamirisa[2]; Oleg N. Senkov[1]; [1]UES, Inc., Matls. & Processes, 4401 Dayton-Xenia Rd., Dayton, OH 45431 USA; [2]Ohio University, Mechl. Engrg., Athens, OH 45701 USA

Al alloys modified with Sc offer significant enhancements in mechanical properties and are attracting attention for aerospace applications. Thermo-mechanical processing (TMP) is an important step in the shape-forming of these alloys to obtain desired shape, microstructure, and mechanical property combinations. The Sc addition is expected to affect the TMP response of Al alloys and a thorough understanding would help in the design and optimization of the process sequences. In this paper, we describe the TMP response of an advanced Sc-modified 7XXX Al alloy produced via direct chill continuous casting. Hot compression tests were conducted in the temperature range of 250 - 500°C and strain rate range of 3´10-4 - 10 s-1. A processing map for hot working is generated based on the analyses of temperature and strain rate sensitivity of the flow stress over the above ranges. Hot deformation mechanisms are identified and correlated with microstructural analysis and hot tensile behavior. The influence of Sc on the hot workability of the alloy is established, and the optimum parameters for TMP will be discussed.

9:30 AM
Microstructure and Tensile Properties of Developmental Al-Zn-Mg-Cu Cast Alloys Modified with Sc and Zr: *Svetlana V. Senkova*[1]; Radhakrishna B. Bhat[1]; Oleg N. Senkov[1]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The effect of combined addition of Sc and Zr on the microstructure and tensile properties of the direct chill (DC) cast ingots of developmental Al-Zn-Mg-Cu alloys has been evaluated in this work. The properties in both the longitudinal and transverse directions of the cast ingots were determined in as-cast and heat-treated conditions, at room and cryogenic temperatures. Homogenization, solution treatment and aging schedules were optimized to obtain superior mechanical properties. In particular, the Sc-containing developmental cast alloys showed the tensile properties, which are much better than the properties of any commercial cast Al alloys and are similar to the properties of a wrought 7075-T6 alloy. The strengthening mechanism, optimum content of the dispersoid-forming elements, and the processing parameters responsible for these improvements are discussed.

9:50 AM
Effect of Crystallographic Texture on Tensile Properties of a 7XXX Alloy with Sc Addition: *Oleg N. Senkov*[1]; Svetlana V. Senkova[1]; Radhakrishna B. Bhat[1]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Room temperature tensile properties of a 7XXX series alloy containing 0.18% Sc were studied after high-temperature extrusion and forging followed by heat treatment. The extruded alloy showed superior tensile strength (UTS=730 MPa) and ductility (El=15%) in the longitudinal direction; however, the properties in this direction reduced considerably, to 630 MPa and 8%, respectively, when forging was applied in the direction opposite to extrusion. Orientation image microscopy (OIM) analysis was conducted to understand why the properties of the extruded product degrade after forging. It was found that the main processes responsible for such behavior are the dynamic recrystallization occurring during forging and leading to grain coarsening and change in the crystallographic texture. The decrease in the tensile strength directly correlates with a decrease in the Taylor factor and the decrease in ductility can be related to large differences in the Schmidt factor of neighboring grains in the forged material.

10:10 AM Break

10:25 AM
Age-Hardening and Plastic Anisotropy in Extruded AA6xxx and AA7xxx Profiles: *Hans Bjerkaas*[1]; Snorre Kjørstad Fjeldbo[1]; Hans Jørgen Roven[1]; Jarle Hjelen[1]; Trond Furu[2]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Trondheim N-7491

Norway; [2]Hydro Aluminium R&D Materials Technology, Sunndalsøra N-6600 Norway

To understand plastic anisotropy in extruded aluminium profiles and how this anisotropy changes during precipitation is essential in order to control forming of aluminium profiles. Several authors have reported large changes in the plastic anisotropy as a function of precipitation in textured alloys. However, the predicted anisotropy deviates from the observed anisotropy when coherent and semi-coherent precipitates are introduced to the microstructure, indicating that such precipitates strongly affects the deformation process. In the present work alloys from the AA6xxx and AA7xxx systems are studied and compared. The extruded profiles investigated have a strong texture, giving rise to a substantial plastic anisotropy in the solution treated condition. Both the effects of natural and artificial ageing are studied. The observed plastic anisotropy is compared with predictions from the Taylor/Bishop/Hill model based on input from orientation data obtained by microdiffraction in a FE-SEM.

10:45 AM

Further Developments in the Precision Forging Technology for High Duty Automotive Parts: Bernd-Arno Behrens[1]; Steffen Reinsch[1]; Axel Specker[1]; *Kathrin Telkamp*[1]; [1]IPH-Institut für Integrierte Produktion Hannover gGmbH, Process Tech., Hollerithallee 6, Hannover 30419 Germany

Precision forging is defined as a flashless forging operation, which generates high quality parts by means of surface quality and accuracy to dimension. So far precision forging processes have been designed for connecting rods, gears and hand tools. The diversification of this group of parts with the design of a precision forging process for crankshafts not only expands the applicable product range but also requires new technical solutions. The most obvious differences between currently produced precision forged parts and crankshafts are the bigger mass of the crankshaft and the extremely asymmetric mass distribution. To meet this challenge a new forging chain with three forming processes was developed. Finite-element-programs have been used for the verification of the applicability definition of the process limits. Practical testing of the processes showed the feasibility of forging crankshafts with flashless precision forging technology and gave references for the verification of the processes.

11:05 AM

Effects of Working, Heat Treatment, and Aging on Microstructural Evolution, Phases, and Crystallographic Texture in Ti-6Al-4V Wire: Liang Zeng[1]; *Thomas R. Bieler*[1]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Crystallographic texture and microstructure was measured on Ti-6Al-4V wire in as-received, cold extruded, heat treated in the alpha+beta phase field, water quenched, and aged samples. Extrusion strengthened the as-received prism fiber texture, but after solutionization and quenching, a secondary basal fiber texture emerged, indicating that a strongly preferred variant selection occurred during quenching. The selection of preferred variants in the alpha-beta-alpha transformation cycles is discussed in relation to shears imposed in the beta phase due to differential thermal expansion between misoriented a regions. After artificial aging, alpha' and beta phases were present, but subsequent natural aging for a year led to elimination of alpha" and a reduction in the amount of the beta phase. The effect of extrusion and solutionization time on the orientations of alpha, alpha', alpha", and beta phases are discussed in the context of the known physical metallurgy of titanium alloys.

11:25 AM

Effect of Oxygen Content on Fatigue Properties of Single-Melt PAM Processed Forged Ti-6Al-4V Bell Housings for Lightweight 155mm Howitzer: *Mustafa Guclu*[1]; Ibrahim Ucok[1]; Hao Dong[1]; Chris Hatch[2]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA; [2]U.S. Army ARDEC, Bldg. 151, Picatinny Arsenal, NJ 07806 USA

Ti-6Al-4V (Ti-6-4) is the main structural alloy for the new lightweight 155mm Howitzer (LW155) and was selected for reduced weight and enhanced performance. However, Ti-6-4's relatively high cost compared to steel and aluminum alloys has limited its usage in other non-aerospace applications. It is clear that, in the near term, the use of single-melt (SM), high oxygen titanium alloys such as those made by plasma arc melt (PAM) processing is at the forefront of low-cost titanium alloys. In this study, Ti-6-4 bell housings for the Lightweight 155mm Howitzer were manufactured by forging using low-cost SM PAM billet stock at three oxygen levels, 0.16, 0.20 and 0.24 wt%, along with a standard double vacuum arc remelted (2XVAR) billet stock at 0.17 wt% oxygen for baseline comparison. To demonstrate the viability of SM PAM material, mechanical properties of mill-annealed forgings were characterized by microstructure, tensile and smooth bar axial fatigue testing. SM PAM forgings met mechanical property requirements of ASTM B381 and AMS 4928 specifications, and exhibited higher tensile and yield strengths than 2XVAR material. Tensile and yield strengths increased with increasing oxygen content with a gradual reduction in ductility values. All SM PAM forgings exhibited slightly higher fatigue strengths compared to those of 2XVAR in the longitudinal orientation. Microstructures of all forgings were similar to those of conventional a-b forgings. The total a content in SM PAM forgings increased with increasing oxygen content. Based on the testing and characterization results, forgings made using SM PAM material up to 0.24 wt% oxygen are viable for bell housing application. The data presented in this paper can be useful to designers in their efforts to introduce low-cost SM PAM Ti-6-4 alloy forgings into various U.S. defense applications. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the U.S. Navy Manufacturing Technology (ManTech) Program.

11:45 AM

Texture and Microstructure Development in Ti-10-2-3: *Seema L. Raghunathan*[1]; Martin Jackson[1]; Richard J. Dashwood[1]; David Dye[1]; [1]Imperial College, Dept. of Matls., Royal Sch. of Mines, Prince Consort Rd., London SW7 2BP UK

Ti-10V-2Fe-3Al (Ti-10-2-3) is a high-strength, high-toughness, deep-hardenable, near-beta titanium alloy widely used for near-net shape forging. It has become clear that the microstructure and resulting properties of most near-beta alloys are very sensitive to the forming variables, even during isothermal forging, and this has, to date, required empirical optimization. The cost associated with this approach is a significant driver for the development of models capable a predicting the final textures and microstructures that result. This work, which is based on an earlier microstructural study, aims to examine the effect of the forming variables on texture development. Neutron diffraction and EBSD results for the misorientation and macroscopic textures are presented, and an attempt is made to rationalize them with the aid of a finite element model of the strain path and a sequentially coupled visco-plastic self-consistent (VPSC) model of deformation that excludes the effects of recrystallization.

Alumina and Bauxite: Alumina Quality

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Thursday AM — Room: 2005
February 17, 2005 — Location: Moscone West Convention Center

Session Chair: Carl Behrens, Hydro Aluminium, Alumina & Bauxite, Porsgrunn N-3907 Norway

8:30 AM

Alumina Quality Improvements at the Alpart Refinery, Nain, St Elizabeth, Ja.: *Patrick Donovan James*[1]; [1]Alumina Partners of Jamaica, Techl., PO Box 529, Arabi, LA 70032 USA

In a quest to be named among the preferred Alumina suppliers in the world market, Alumina Partners of Jamaica (Alpart) embarked on a stepwise program over the past few years to demonstrate significant improvement in its product quality. The area of focus has surrounded particle size, particle strength and dustiness. The approach involved structural, procedural and technological changes which are a blend of known practices worldwide. This paper describes the approach taken, the step changes taken to date, the pitfalls along the way as learning for the future and results achieved.

8:55 AM

Effect of the Total Soda on the CMR of the Agglomeration Zone and Occluded Soda in the Precipitation Circuit at CVG Bauxilum: *Jesús Alcalá*[1]; Nelson Angulo[1]; [1]CVG Bauxilum Alumina Plant, Av. Fuerzas Armadas, Zona Industl. Matanzas, Ciudad Guayana, Edo. Bolívar Venezuela

The occluded soda is one of the main quality characteristic of metallurgic grade calcined alumina and the CMR in the agglomeration zone of the precipitation circuit defines the productivity behavior throughout the system, these two parameters are related with the

levels of impurities and other variables of the process. At CVG Bauxilum the Caustic Molar Ratio (CMR) is defined as: CMR = 1.645 * [Na2O]/[Al2O3] where the constant 1,645 is: Al2O3 Molecular Weight/Na2O Molecular Weight and the changes of this expression are produced specially in the agglomeration stage. The following work shows the difference in the behavior among agglomeration and growth stages and influence of the total caustic and the impurities levels on the changes of CMR, especially during the agglomeration stage of the precipitation circuit. The effect of the major impurities and its relations to the total caustic and occluded soda were analyzed through empirical equations by means of statistical programs. The effects of variables that influence the precipitation circuit productivity were also determined.

9:20 AM
Effect of Residual Hydrate on Properties of Metallurgical Grade Aluminas: *Miguel Ángel Llavona*[1]; *Roberto Zapico Amez*[1]; [1]University of Oviedo, Dept. Matls. Sci., Gonzalo Gutiérrez Quirós s/n, Mieres, Asturias 33600 Spain

There are different types of water in metallurgical grade aluminas: physically adsorbed, chemisorbed, water of constitution and lattice water, and the aluminium industry uses several tests to determine it. If the moisture of the aluminas is determined to 110°C-4h, the MOI to 300°C-4h and the LOI to 1200° C-2h, according to the standard tests, and the alumina contains hydrate, the values of the MOI and LOI will be seen altered, and they will not have meaning. The loss of weight to 500°C has been related with the content in hydrate in the metallurgical grade aluminas. Thermal conductivity of aluminas have been determined by the hot wire method. A linear relation between thermal conductivity and density was observed. If the alumina has some residual hydrate, the obtained value of the thermal conductivity will be lower than the expected according with the packing density.

9:45 AM
Study on the Relationship Between the Stress-State and the Intensity of the Overlapped Al(OH)3 Crystals: Wangxing Li[1]; Lusheng Ye[2]; Shugui Hua[1]; Zhoulan Yin[1]; [1]Zhengzhou Research Institute of Chalco, Zhengzhou, Henan 450041 China; [2]Central South University, Physl.-Chmst. Inst., Changsha, Hunan 410083 China

Using stress analysis, the stress-state between two overlapped Al(OH)3 crystals was calculated. The relationship between the intensity and the overlap of the crystals was obtained. Conclusions were made that the intensity of two overlapped Al(OH)3 crystals is reduced as the overlap area is decreased. The method can also be used to study the relationship between the stress-state and the intensity for more overlapped Al(OH)3 crystals.

10:10 AM Break

10:25 AM
Influence of Organic Additives on Particle Size and Strength of Hydrate from Carbonization of Seeded Sodium Aluminate Liquors: *Wang Zhi*[1]; Bi Shiwen[2]; Yang Yihong[2]; Yuan Zhangfu[1]; [1]Chinese Academy of Sciences, Inst. of Process Engrg., Zhong Guan Cun, Haidian Dist., Beijing 100080 China; [2]Northeastern University, Sch. of Matls. & Metall., Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

Poor strength and excessive fine grain content are the main problem to be solved in the Sintering process for sandy alumina production. A systematic study was made of the effects and mechanism of different organic compounds on product median diameter and attrition index quantitatively and on the crystal habit of gibbsite qualitatively. It has been determined that some additive at certain concentration can significantly decrease the fine particle content, improve the product strength and modify gibbsite crystal morphology as crystal habit modifier. For instance, additive CF2 increases $Al(OH)_3$ median size by 12 μm at 200 ml/l, at the same time decreases the <45 μm particle mass fraction and the attrition index by 17% and 10% respectively. The enhancement of anti-attrition ability can be linked to the changes of structure and shape of gibbsite crystals. The crystal morphologies indicate that additives can accelerate the aggregation and inter-growth of crystal units, as a result the gibbsite presents an inlaid crystal structure close to globular shape.

10:50 AM
Application of Fractal Theory in Studying Strength of Sandy Alumina: *Tan Jun*[1]; Chen Qi Yuan[1]; *Yin Zhou lan*[1]; [1]Central South University, Chmst. & Cheml. Engrg., Lushan nanlu 154#, Changsha, Hunan 410083 China

The fractal theory was introduced to study the relationship of sandy alumina between morphology and strength. Morphological characterizations of a series of alumina from seeded and carbonization precipitation were described using scanning electron microscopy (SEM). Attrition indices and fractal dimensions were determined. It showed that the attrition index of alumina from carbonization precipitation increases with increasing fractal dimension. As to alumina from seeded precipitation, it suggested that the crystal structure should be considered during analyzing the relationship between fractal dimension and strength. The attrition index of alumina with the same structure increases with increasing fractal dimension.

11:15 AM
Microstructure Analysis of Aluminum Hydroxide and Alumina: Tan Jun[1]; *Chen Qi Yuan*[1]; *YIn Zhou lan*[1]; [1]Central South University, Coll. of Chmst. & Cheml. Engrg., Lushan nanlu 154#, Changsha, Hunan 410083 China

The microstructure of aluminum hydroxide and alumina were characterized by scanning electronic microscopy (SEM). The differences between two kinds of alumina from seeded precipitation and carbonization precipitation were compared. Two methods of breakage were developed to view the interior microstructure of alumina. The microstructure of alumina is similar to that of aluminum hydroxide, while there are some cracks on the surface of alumina. The small crystals to form alumina from seeded and carbonization precipitation are hexagonal prism, short hexagonal cylindrical respectively. The growth mechanism of aluminum hydroxide can be concluded according to the interior structure of alumina.

Aluminum Reduction Technology: Fundamentals

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Thursday AM Room: 2003
February 17, 2005 Location: Moscone West Convention Center

Session Chair: Pavel Fellner, Slovak University of Technology, Inorganic Tech., Bratislava SK-812 37 Slovakia

8:30 AM
A New Modelling for Simulating Bubble Motions in a Smelter: *Michel V. Romerio*[1]; Alexei Lozinski[1]; Jacques Rappaz[1]; [1]Swiss Federal Institute of Technology, IACS, Lausanne 1015 Switzerland

A model allowing to describe motion, coalescence and escape of bubbles in the bath under the action of gravitation is proposed. The bubbles introduced under the anodes at the initial time have fixed sizes corresponding to those obtained by the detachment phenomenon. Bubble shapes are described by ellipsoids with a fixed volume. The gas motion effects inside the bubbles are neglected. The motion of a bubble is obtained in a Lagrangian form using the virtual works principle. It is thus described by the motion of the gravity center combined with a rotation and a deformation. The set of equations is numerically solved with the help of the fictitious domain technique in which the Navier-Stokes equations in the domain formed by both fluid and gas are considered. The equations governing the bubbles motion are imposed by introducing Lagrange multipliers on the bubbles boundaries. Numerical results in 2D are presented.

8:55 AM
Experimental and Numerical Studies on Bubble Removal Under Anodes by Using Ultrasound in Water Solutions and in Cryolitic Melts: Harald A. Øye[1]; *Jilai Xue*[1]; G. Servant[1]; Trygve Foosnæs[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Sem Sælands v. 14, Trondheim N-7491 Norway

Gas bubbles under the anodes during aluminium electrolysis constitute a significant part of the cell voltage drop, and large bubbles can sometimes result in unwanted anode effects. Laboratory studies on removing gas bubbles under anodes by using ultrasound were carried out in water solutions and in cryolitic melts. Numerical simulation techniques were applied in scale- up of the anode size. With 20 kHz and 28 kHz ultrasound, the gas bubbles are removed from the downward surface of the anodes, which result in a reduction in the cell voltage. The results show potential in energy saving, while the ultrasound effectiveness should be improved before possible application of this technology in aluminium reduction cells.

9:20 AM
Simulation of the Bubble Layer in Aluminum Electrolysis Cells: *Laszlo I. Kiss*[1]; Sándor Poncsák[1]; Jacques Antille[2]; [1]Universite du Quebec, Dept. des Scies. Appliquees, 555, boul. de l'Universite, Chicoutimi,

Quebec G7H 2B1 Canada; [2]Alcan Primary Metals Europe, Sierre CH 3695 Switzerland

The structure and dynamics of the bubble laden layer has an important influence on the performance of the aluminum reduction cells as the gas bubbles block the way of passage of the electric current. The extent of the gas covered portion of the anode bottom depends on the equilibrium between the rates of production and evacuation of the gas. A mathematical model was developed that predict the variation of the covering factor as a function of the design and operational parameters of the aluminum electrolysis cell. The simulator offers the time series of the fluctuations of the covering factor, its average values as well as a graphical representation of the movement of the bubbles. The influence of the size and shape of the anode on the covering factor is presented.

9:45 AM
Regimes of the Movement of Bubbles Under the Anode in an Aluminum Electrolysis Cell: *Alexandre Perron*[1]; László I. Kiss[1]; Sándor Poncsák[1]; [1]Université du Québec à Chicoutimi, Dept. des Scis. Appliquees, 555 boul. de l'Université, Chicoutimi, Québec G7H 2B1 Canada

Although the bubble layer plays and important role in the operation of an aluminum reduction cell, the details of the nucleation, detachment and movement of the bubbles are not completely understood. The direct observation of the bubble related phenomena is practically impossible, so our understanding is based on theoretical analysis, speculative models and on the observation of the behavior of bubbles in water models or in other low temperature, transparent systems. In the present paper the different regimes of the movement of the individual bubbles under a downward facing solid plate are discussed. The formation of a separating liquid layer, the kinematics of the bubbles as well as the flow field around them were observed and analyzed using low temperature physical models.

10:10 AM
A New Study on Bubble Behaviors on Carbon Anode in Aluminum Electrolysis: *Bingliang Gao*[1]; Zhaowen Wang[1]; Zhuxian Qiu[1]; Haitao Li[1]; [1]Northeastern University, Coll. of Matls. & Metall., MB 117, Shenyang, Liaoning 110004 China

In this paper, the bubble behaviors on carbon anodes during aluminum electrolysis were studied in a bench scale cell. Previous studies on anode bubble behaviors were largely based on the water models simulation and see-through electrolysis cell observation. The tests adopted same method used by R.J Aaberg to investigate the influence of anode current density and anode-cathode distance (ACD) on the anode bubble behaviors at the status of electrolysis. PLC (programmable controller) combined with a computer was applied to collect experimental data. The results of the study indicate: Increasing current density leads to increasing release frequency of bubbles and thin bubble layer thickness when current density on anode is less than 1.2A/cm2; At higher anode current density, the release frequency of the anode bubbles decreases with increasing current density; During anode effect, the release frequency of the anode bubbles goes to zero.

10:35 AM Break

10:50 AM
Chemical and Electrochemical Reactions of Sulphur Species in Cryolite Melts: *Pavel Fellner*[1]; Marta Ambrová[1]; Ján Híves[1]; Michal Korenko[2]; Jomar Thonstad[3]; [1]Slovak University of Technology in Bratislava, Dept. of Inorganic Tech., Radlinskeho 9, Bratislava SK - 812 37 Slovakia; [2]Slovak Academy of Sciences, Inst. of Inorganic Chmst., Dúbravská cesta 9, Bratislava SK-845 38 Slovakia; [3]Norwegian University of Science and Technology, Dept. of Matls. Tech., Trondheim N-7491 Norway

Data on the chemical reduction of sulphate by aluminium and by graphite in cryolite melts will be reported. Chemical reduction of sulphate by aluminium or carbon follows a similar sequence. Only SO42-, S2- and polysulphides species were detected in the solidified melts. It was found that sulphide reacts with dissolved iron(II), forming FeS, which is insoluble in cryolite melts. Because of its high density, solid FeS particles will tend to settle and react with the aluminium cathode, forming Al2S3, which is readily soluble in molten cryolite. This may be a non-electrochemical mechanism for contaminating aluminium with iron. The cryolite-rich part of the phase diagram Na3AlF6-Al2S3 was determined. By cathodic reduction of sulphate it was found that two electrons take part in the reduction of sulphate, i.e. SO42- + 2e- = SO32- + O2-. Since the sulphite species is not stable at high temperatures, it decomposes thermally into SO42-and S2-.

11:15 AM
Aluminum Electrowinning in Ionic Liquids at Room Temperature: *Mingming Zhang*[1]; Venkat Kamavaram[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., A-129 Bevill Bldg., 126 Seventh Ave., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

The electrowinning of aluminum was studied in room temperature ionic liquids prepared by mixing 1-hexyl-3-methylimidazolium chloride (C6mim[Cl]) and aluminum chloride (AlCl3). Aluminum was electrowinned at copper cathode with graphite as anode. Cathode current density and current efficiency increase with increasing temperature, molar ratio of AlCl3 and applied cell voltage. However, high current efficiencies were obtained at high voltage, intermediate AlCl3 concentration and low temperature. The deposit morphology and thickness were examined in detail using SEM and XRD techniques. The electrowinning of aluminum in C6mim[Cl]-AlCl3) and C4mim[Cl]-AlCl3 was also compared regarding the current density and current efficiency.

11:40 AM
The Aluminum Reduction Cell Closed System of 3D Mathematical Models: Gennady V. Arkhipov[1]; *Alexander V. Rozin*[2]; Alexander G. Arkhipov[1]; [1]RUSAL, Engrg.-Technologl. Ctr. Ltd., Pogranichnikov St. 37, Krasnoyarsk 660111 Russia; [2]Lomonosov Moscow State University, Inst. of Mech., Michurinsky Pr. 1, Moscow 119899 Russia

3D mathematical models to predict different processes in aluminum reduction cell have been developed by RUSAL for several recent years. Each of the models can calculate the field of some physical parameter (e.g. flow velocity) using for data fields of other parameters (current density and magnetic induction) or boundary conditions (ledge profile) produced by other models. In this study all models have been worked out to run simultaneously except for the calculation of magnetic field, which is assumed to be stationary. Natural convection and radiation are taken into account in the conjugate heat transfer problem. This closed system of mathematical models makes possible to evaluate main physical parameters in the reduction cell but it needs large computation times. To solve the optimization tasks simplified "local" models can be created based on the coarse-mesh solutions of the whole problem.

Bulk Metallic Glasses: Mechanical Behavior and Phase Transformation

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Thursday AM Room: 3006
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Yoshihiko Yokoyama, University of Hyogo, Matls. Sci. & Engrg., Himeji 671-2201 Japan; D. H. Kim, Yonsei University, Metallurgl. Engrg., Seoul 120-749 S. Korea

8:30 AM
Development and Characterization of New Ca-Mg-Zn-Cu Bulk Metallic Glasses: *Oleg N. Senkov*[1]; J. Mike Scott[1]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Several Ca-Mg-Zn-Cu bulk metallic glasses with the thickness of up to 10 mm were produced by a Cu mold casting method. The glass transition, crystallization, solidus and liquidus temperatures, as well as the heats of crystallization and melting were determined for these glasses using differential scanning calorimetry. Compression properties of these amorphous alloys were also studied at room temperature and in the temperature range of the super-cooled liquid (ΔT_x range). As-cast alloys were very brittle at room temperature; however, ~30-40% compression strain was recorded during elevated temperature deformation. After deformation in the ΔT_x range the alloys remained fully amorphous and they showed some ductility at room temperature.

8:50 AM
Modeling the Formability of Bulk Metallic Glasses: *Justin Cheney*[1]; Kenneth S. Vecchio[1]; [1]University of California, Dept. of MAE, Matls. Sci. Grp., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

Understanding the processes of glass formation in metals will allow better alloys with lower critical cooling rates to be produced. Lowering the critical cooling rate includes slowing the crystallization rates of metal alloys through kinetic and thermodynamic means. Currently no definitive method for evaluating the theoretical glass forming ability of an alloy has been produced. In the current work, liquidus surfaces are generated using ThermCalc simulations to determine deep eutectics in multi-component alloys. Three-dimensional visualization is used to examine the melting temperatures of an entire compositional range within a multi-component alloy. The thermodynamic behavior of an alloy is evaluated with this technique as the crystal nucleation temperature follows the trend of the liquidus temperature. To model the kinetics of amorphous forming ability, a short range order simulation in combination with random hard sphere packing models are used. The result gives a theoretical description of the atomic movement necessary to form a crystal structure. The aim is to predict alloys that can exist as densely packed liquids with uniformly distributed, large magnitude strain fields. 3-D visualization of the model predictions is used to evaluate the theoretical structures of an alloy, in addition to the strain fields and packing densities that exist throughout the volume. These models are compared with existing empirical data to confirm their validity. The simulations can then be used to evaluate all possible elemental combinations to determine potentially novel alloy systems with higher glass forming ability.

9:10 AM
Mg49Y15Cu36 Bulk Metallic Glass Composites Synthesized by Vacuum Hot-Pressing of Mechanically Alloyed Powders: *Pee-Yew Lee*[1]; Cheng Lo[1]; [1]National Taiwan Ocean University, Matls. Engrg., 2, Pei-Ning Rd., Keelung, Taiwan 202 Taiwan

In the present study, WC/Mg49Y15Cu36 metallic glass composite powders were prepared by mechanical alloying of pure Mg, Y, Cu, and WC powder mixtures. Mg49Y15Cu36 metallic glass composite powders were obtained after 10 h of milling as confirmed by X-ray diffraction and differential scanning calorimetry. The metallic glass composites powders were found to exhibit a supercooled liquid region before crystallization. Bulk metallic glass (BMG) composites were synthesized by vacuum hot pressing the as-milled Mg49Y15Cu36 metallic glass composite powders at 723 K in the pressure range of 0.72-1.20 GPa. BMG composite with submicron WC particles homogeneously embedded in a highly dense nanocrystalline/amorphous matrix was successfully prepared under applied pressure of 1.20 GPa. It was found that the pressure could enhance the thermal stability and promotes nanocrystallization of WC/Mg49Y15Cu36 BMG composites.

9:30 AM
Transformations Near the Glass Transition in Pd-Based Bulk Metallic Glasses: *Shantanu Vijay Madge*[1]; Gerhard Wilde[1]; [1]Forschungszentrum Karlsruhe, Inst. of Nanotech., Hermann-von-Helmholtz-Platz 1, Eggenstein-Leopoldshafen 76344 Germany

Bulk metallic glasses (BMGs) are interesting partly because of the complexity that underlies crystallisation in these alloys, which can involve various metastable intermediate phases. Phase separation in the undercooled liquid is an issue that has seen much attention in recent years in order to explain the counter-intuitive nanocrystallisation that occurs in some BMGs. In the present work, a series of Pd-based BMGs has been prepared by suction-casting. The transformations in certain glasses on heating above the glass-transition temperature are investigated by complementary techniques such as modulated-temperature calorimetry, X-ray scattering and transmission electron microscopy. The results are discussed in the light of possible amorphous phase separation that has been suggested to occur also in Pd-rich systems. The possibility of using phase separation for preparing glass-glass composites with enhanced mechanical properties is also considered.

9:50 AM
A New Criterion for Glass Forming Ability of Bulk Metallic Glasses: *E. S. Park*[1]; W. T. Kim[2]; D. H. Kim[1]; [1]Yonsei University, Dept. of Metallurgl. Engrg., Ctr. for Non-Crystalline Matls., 134 Shinchon-Dong, Seodaemun-gu, Seoul 120-749 S. Korea; [2]Chongju University, Dept. of Applied Sci., 36, Naedok-Dong, Sangdang-Gu, Chongju, Chungbuk 360-764 S. Korea

A new criterion for glass forming ability (GFA) of bulk metallic glasses (BMGs) is proposed based on the consideration of both the liquid phase stability and the resistance to the formation of competing crystalline phases. Especially, the simple rule of mixtures of melting temperatures,Tmmix has been considered for a comprehensive expression to predict GFA for various glass-forming systems. The interrelationship between this new parameter and maximum diameter, Dmax is elaborated and discussed in comparison with four other representatives, i.e. supercooled liquid region, K parameter, reduced glass transition temperature Trg, and gamma parameter. The new parameter demonstrates the highest regression coefficient value and the narrowest prediction band implying that the new parameter correlates better with the maximum diameter, Dmax than other parameters suggested so far. It is therefore considered that the presently proposed parameter has a stronger correlation with GFA in the various BMG alloy systems.

10:10 AM
Evaluation of Shear Bands Developed Under the Tensile Deformation Mode for Bulk Metallic Glasses and MMCs Reinforced by Metallic Glass: *Donghyun Bae*[1]; [1]Yonsei University, Dept. of Metallurgl. Engrg., 134 Shinchon-dong, Seodaemun-gu, Seoul 120-749 Korea

The tensile deformation/failure behaviors of a Zr-based bulk metallic glass (BMG) and metal matrix composites (MMCs) reinforced by Ni-based metallic glass fibers have been systematically investigated. The Zr-based BMG is fabricated by the suction casting method and the MMCs are synthesized by warm extrusion of gas-atomized powders. Under the tensile loading conditions, i.e. 4-point bending or uniaxial tension, the shear bands are developed significantly due to the constrained effect stemming from strain distribution for 4-point bending or from confinement of ductile matrix for MMCs. The formation and propagation of the shear bends under tension, significantly different from those observed under compression, and its structure will be discussed.

Carbon Technology: Cathode Materials and Corrosion II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Todd W. Dixon, ConocoPhillips, Borger, TX 79007 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland

Thursday AM Room: 2007
February 17, 2005 Location: Moscone West Convention Center

Session Chair: Morton Sorlie, Elkem Aluminium ANS Research, Dept. of Matls. Tech., N-4675 Kritiansand Norway

8:30 AM
Quality Evaluation of Nitride Bonded Silicon Carbide Sidelining Materials: *Egil Skybakmoen*[1]; Lisbet Stoen[1]; Jannicke H. Kvello[1]; Ove Darell[1]; [1]SINTEF Materials and Chemistry, Energy Conversion & Matls., Sem Saelandsvei 6, Trondheim N-7465 Norway

A laboratory test method to determine the chemical/oxidation resistance of commercially available SiC-based sidelining materials was developed at SINTEF in 1996. Since then more than 260 samples from around 20 suppliers world-wide have been tested, and an overview of typical - and abnormal results will be given. The tests have demonstrated the importance of checking the quality of the SiC blocks throughout the entire cross section of the blocks. Large variations were found, depending on whether samples were taken near the surface or from the central parts of the blocks. The observed degradation will be discussed in terms of a proposed reaction mechanism, and related to material quality parameters and test conditions (bath acidity and gas atmosphere).

8:55 AM
Effect of Changes in Physical-Mechanical Properties of Materials on Aluminum Reduction Cell Performance: *Gennady V. Arkhipov*[1]; Victor Yu. Buzunov[1]; Vitaly V. Pingin[1]; Vasily I. Borisov[1]; [1]RUSAL, Engrg.-Technologl. Ctr. Ltd., Pogranichnikov St. 37, Krasnoyarsk 660111 Russia

Calculation results of thermo-electric-mechanical fields with the initial lining and cathode shell material properties differ numerically from calculated results taking into account the property changes during operation. Mathematical modeling demonstrated effects of changes in the properties on thermoelectric fields, magnetic hydrodynamics and stress-strain state. To study changes in properties like electric conductivity, thermal conductivity, elasticity modulus, thermal and sodium expansion of lining materials, they have been investigated after autopsies of cells with different life. Mechanical properties of cathode shells have been investigated after different campaigns to study changes in mechanical properties of the steel.

9:20 AM
Titanium Diboride and Molybdenum Silicide Composite Coating on Cathode Carbon Blocks in Aluminum Electrolysis Cells by Atmospheric Plasma Spraying: *Huimin Lu*[1]; Ruixin Ma[1]; Wenhui Yuan[1]; Yongheng Wang[1]; [1]University of Science and Technology, Metallurg. Engrg. Sch., No. 30 Xueyuan Rd., Beijing, Beijing 100083 China

Self-propagating high-temperature synthesis with reduction process was used to fabricate the TiB2- MoS2 composite powder. The TiB2- MoS2 composite was coated on the cathode carbon blocks by atmospheric plasma spraying. Various properties of the coated carbon block such as the chemical and electrochemical stability, the corrosive resistance, thermal shock resistance, wettability and service life in molten cryolite-alumina and aluminum liquids were tested. The microstructure of the coating, the interfacial bonding between the coating and the carbon substrate, thermal expansion performance and electrical resistivity of the coating, the effect of different process parameters e.g. spray distance, additive MoS2 content on the coating were also studied. These research results indicate that the TiB2-MoS2 composite coating cathodes by atmospheric plasma spraying have the potential to give significant benefits to the aluminum electrolysis process in terms of energy coast saving and extended cell life.

9:45 AM
The Effect of Sodium-Containing Additives on the Sodium-Penetration Resistance of TiB2/C Composite Wettable Cathode in Aluminum Electrolysis: *Qingyu Li*[1]; Yanqing Lai[2]; Jie Li[2]; Jing Fang[2]; Jianhong Yang[2]; Zhu Chen[3]; [1]Guangxi Normal University, Sch. of Chmst. & Cheml. Engrg., Guilin, Guangxi 541004 China; [2]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China; [3]Aluminum Corporation of China Limited, Guangxi Branch, Pingguo, Guangxi 431400 China

TiB2/C composite material is one of the best wettable cathode materials for aluminum electrolysis. But TiB2/C composites usually contain about 30-70% carbon, which results in the expansion and failure of the composites due to sodium and bath penetration in aluminum electrolysis. In this paper, TiB2/C composites were prepared with sodium-containing additives, such as spent potlining powder, as carbon component hoping to improve the sodium-penetration resistance of TiB2/C composites in aluminum electrolysis. Results showed that the sodium-penetration resistance of TiB2/C composites in aluminum electrolysis was improved effectively by adding sodium-containing additives.

10:10 AM Break

10:25 AM
Cells Incorporating Cathode Blocks Impregnated with Boron Oxide: *Rudolf Keller*[1]; [1]EMEC Consultants, 4221 Roundtop Rd., Export, PA 15632 USA

Cathode blocks of aluminum reduction cells were impregnated with boron oxide. The purpose was to promote the wetting of the carbon surface by aluminum metal with the formation of titanium diboride through the reaction of the boron oxide with titanium being added to the metal pool. An 11-month test with a cell fully equipped with impregnated amorphous blocks (containing 30% graphite) resulted in excellent, minimal cathode wear, but it did not yield operational benefits. It is projected that the approach is very promising for use with graphitized blocks, particularly to improve cathode life. This presentation is based on work conducted in cooperation with industrial partners (Century Aluminum, Northwest Aluminum, and SGL Carbon) and supported by the Department of Energy under Cooperative Agreement DE-FC36-98ID13664.

10:50 AM
Effect of Porosity Structure on Penetration and Performance of Lining Materials: *Sergey A. Khramenko*[1]; Peter V. Polyakov[2]; Alexander V. Rozin[3]; Alexander P. Skibin[4]; [1]RUSAL Engineering & Technology Centre, Pogranichnikov St. 37, Krasnoyarsk 660111 Russia; [2]State University for Non-Ferrous Metals and Gold, Krasnoyarskiy Rabochiy St.95 66025 Russia; [3]Lomonosov Moscow State University, Inst. of Mech., Michurinsky Pr. 1, Moscow 119899 Russia; [4]Bauman Moscow State Technical University, Profsouznaya St.100-4-40, Moscow 117437 Russia

The characteristic property of cathode materials is porosity that causes bath penetration in the cathode blocks. As a result, internal stresses and micro cracks may appear that increase the wear of the reduction cell. The pore distribution in the cathode blocks materials from different manufactures is investigated. It is found that pore distributions vary from homogeneous monopore normal distribution of pore size to bipore distribution and even nonuniform structure with continuous distribution of pores. The mechanism of pores formation is investigated experimentally on the model patterns of cathode carbon materials. The dependences of their physical and engineering attributes on pore structure are obtained. The mathematical model of the bath filtration in the cathode blocks during start up operations is created. The model can estimate the performance attributes of the cathode materials with different pore structure and its influence on cell lifetime.

Cast Shop Technology: Foundry

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Thursday AM — Room: 2001
February 17, 2005 — Location: Moscone West Convention Center

Session Chair: Arne K. Dahle, University of Queensland, CRC for Cast Metals Mfg., Brisbane Qld 4072 Australia

8:30 AM
Simulation of Microstructures and Yield Strength of a High Pressure Die Cast A380 Aluminum Alloy Component: *Mei Li*[1]; Jake Zindel[1]; Larry Godlewski[1]; Brian Schneider[1]; Adam Olukalns[1]; Christopher M. Wolverton[1]; John Allison[1]; [1]Ford Motor Company, Ford Rsch. & Advd. Engrg. Lab., 2101 Village Rd., Dearborn, MI 48124 USA

Cast aluminum alloys are increasingly being utilized by automotive industry for manufacturing chassis and powertrain components to reduce vehicle weight and consequently increase fuel economy and reduce emissions. High pressure die casting process provides tremendous cost saving opportunities in manufacturing these components. Development of Virtual Casting tools for HPDC aluminum will lead to fast prototyping and tooling, thus fast final products with reduced cost. This talk describes the development of the interfacial heat transfer coefficients between casting and die with in-cavity and in-die thermal couple measurements in a lab HPDC component, the simulation of microstructure evolution during casting and the prediction of yield strength of an engine block. The prediction can be used to design and optimize casting component and process.

8:50 AM
Preliminary Results on the Effects of Modification During Semi-Solid Processing of Al-Si Alloys: *Shahrooz Nafisi*[1]; [1]University of Quebec, CURAL, 555, Univ. Blvd., Chicoutimi, Quebec G7H 2B1 Canada

Silicon is one of the most important alloying elements incorporated in aluminum alloys. Its addition is to improve castability, fluidity, reduce shrinkage as silicon expands on solidification and renders superior mechanical properties. The morphology of silicon however plays an important role on the properties of finished products. Therefore, the usual practice is to modify the as-cast flake or acicular silicon morphology employing special heat treatment or addition of certain modifiers. As a result, the silicon morphology changes to fibrous form and thus the mechanical properties of the as-cast parts improve. For conventional casting, this treatment has been investigated widely by so many researchers but in the SSM processing, few published papers could be found. In this article, the addition of Sr as a modifier for Al-Si binary alloy is investigated for semi-solid alloy using the patented SEED process, "Swirled Enthalpy Equilibration Device."

9:10 AM
Effects of Sr-Na Interactions in Hypoeutectic Foundry Alloys: *Liming Lu*[1]; Malcolm J. Couper[2]; Arne K. Dahle[1]; [1]University of Queensland, CRC for Cast Metals Mfg., Sch. of Engrg., Brisbane, QLD 4072 Australia; [2]Comalco Research & Technical Support, 15 Edgars Rd., Thomastown, VIC 3072 Australia

From recent published data, it is still unclear whether combined additions of Na and Sr have synergistic effects or deleterious interactions. This paper clarifies the interactions and the effects on alloy solidification and castability. Combined additions of Sr and Na do not appear to cause improvement of the modification of the eutectic microstructure even after only a short period after addition. Na addition may promote Sr vaporization and/or oxidation kinetically, leading to a quicker loss of both modifiers, which is blamed for the disappearance of the modification effect during holding. Quenching trails during the eutectic arrest indicate that addition of Sr into Na-modified melts does not alter the eutectic solidification behaviour. The effect of

Na on eutectic solidification dominates, and the eutectic is observed to evolve with a significant dependency on the thermal gradient. Combined Sr and Na additions produced no beneficial effects on porosity and casting defects.

9:30 AM
Dissolution, Recovery and Fade of Sr Master Alloys in Al-7Si-0.5Mg Casting Alloy: *John A. Taylor*[1]; Malcolm J. Couper[2]; Catherine L. Smith[2]; Darius P.K. Singh[3]; [1]University of Queensland, CRC for Cast Metals Mfg. (CAST), Sch. of Engrg., Brisbane, QLD 4072 Australia; [2]Comalco Research and Technical Support, 15 Edgars Rd., Thomastown, VIC 3074 Australia; [3]Ion Automotive, 100 Plunkett Ave., Manukau City, Auckland New Zealand

A number of commercial Al-Sr master alloys of differing Sr content and product form have been added to Al-7Si-0.5Mg casting alloy melts and held at constant temperature for periods of at least 6 hours following the addition. The master alloys were added to achieve specific Sr target levels (mostly 200 ppm, but also 500 and 1,000 ppm) and the melts were held at various temperatures (most at 710ºC, but also 670, 690, 740 and 770ºC). A total of thirty six melt trials were conducted and during each trial chill-cast disc samples were taken throughout for subsequent chemical analysis. The Sr concentration versus time data of each trial has been considered in terms of Sr dissolution/recovery behaviour, as well as Sr loss/fade. Trends in the data are identified and discussed, and implications for industrial practices are suggested.

9:50 AM
Effect of Process and Design Variables in the Production of Expandable Polystyrene Patterns for Lost Foam Casting: *Rohan Bhat*[1]; Sushil Bhavnani[1]; Ruel Overfelt[1]; David Sheldon[2]; [1]Auburn University, Mechl. Engrg., 201 Ross Hall, Auburn, AL 36832 USA; [2]Vulcan Engineering Company, Helena, AL 35080 USA

The defects caused in Lost Foam Casting (LFC) are believed to be due to inconsistencies in the Expandable Polystyrene (EPS) pattern injection process. It is believed that fairly small localized density variations in EPS patterns lead to folds and other casting defects. The focus of this study is to understand the cause of these inconsistencies. The packing of EPS beads into the mold during a pattern-making is affected by system parameters such as the fill pressure and the pressure in the bead supply canister, geometric features such as distance between the tip of injector and the surface on which beads impinge, and the venting configuration. High speed video confirms that the time required for the mold to get packed with beads affects the density distribution; especially when used with a pressurized bead canister. For injector to opposing wall (IOW) distance less than 3.5mm, the injector performance becomes erratic. It was observed that for IOW distance less than 8mm the density gradient within the mold was about 12% for a vented bead canister and was about 16% for pressurized canister operation. As the IOW distance increases the density distribution throughout the mold gets better. The bead mass dispensed increased with increase in both fill pressure and canister pressure leading to an increase in the packing ratio (defined as the ratio of volume occupied by beads to total mold volume). Pressure measurements at the injector tip and the vent locations are also reported. The fill time decreased as the pressure increased but was independent of fill pressure at high canister pressure. With this better understanding of the blowing process in LFC, mold designers can design molds which produce better patterns leading to the production of sound quality castings.

10:10 AM Break

10:20 AM
Effect of Flux Compositions on Grain Refinement in Al-Si-Mg Alloy: *Chaowalit Limmaneevichitr*[1]; Witthaya Eidhed[2]; [1]King Mongkut's University of Technology Thonburi, Production Engrg. Dept., 91 Pracha-u-tit Rd., Bangmod, Tungkhru, Bangkok 10140 Thailand; [2]King Mongkut's Institute of Technology North Bangkok, Faculty of Engrg., Bangsue, Bangkok 10800 Thailand

There are many compositions in fluxes and each composition has different effects on melt treatment. However, the effect of flux compositions on grain refinement fading has not yet been well established. This research is to study effect of different typical chemical compositions in various ratios on grain refinement fading after recycling of return scraps. Al-Si-Mg alloy (A356) was selected for this research and grain refined using 0.2 wt. % of Al-5Ti-1B grain refiners. Each molten alloy was treated with three different fluxes at 1% of aluminum weight. The work was undertaken by recycling the grain-refined castings for four cycles. No grain refiner was added but the same flux was added in each melting process at the same weight ratio. It was found that flux compositions have strong effect on grain size and fading phenomenon of grain refinement. Based on this result, it was found that MgCl2 in flux composition can reduce the fading phenomenon substantially.

10:40 AM
Effect of Stirring on the Morphological Evolution of Silicon in Al-Si Alloys: *Shahrooz Nafisi*[1]; [1]University of Quebec, 555, Univ. Blvd., Chicoutimi, Quebec G7H 2B1 Canada

SemiSolid Metal Processing (SSM) is a relatively new technology for metal forming different from the conventional forming process which use either solid or liquid metals as started materials. In SSM processing of AlSi alloys, there are two important features that should be considered, the size and shape of the primary á-Al particles and also the morphology of silicon in the eutectic phase. It is quite well known that the irregular growth of the Si eutectic can be modified by addition of modifiers, i.e., Strontium. In this paper, it will be shown that in the SSM processing with stirring application, not only á-Al particles took the rosette or globule shape, but also the silicon in the eutectic is broken and become much smaller in size.

11:00 AM
Impact of Cu Additions on a Structure and Mechanical Properties of Near and Hypoeutectic Silumins: *Tomasz Stuczynski*[1]; Zbigniew Zamkotowicz[1]; Marzena Lech Grega[1]; [1]Institute of Non-Ferrous Metals, Light Metals Dept., Pilsudskiego 19, Skawina 32-050 Poland

Paper presents results of investigations defining the role of Cu in forming macro and microstructure of near and hypoeutectic silumins. The following parameters acted as criteria of evaluation: size of grain and dendrites of solid solution á as well as the mechanical properties of tested alloys. Achieved results were presented to forecast the properties of alloys in a function of chemical composition.

11:20 AM
Study on Method of Increasing Viscosity in Fabricating Aluminum Foam: Hong-jie Luo[1]; G.C Yao[1]; [1]Northeastern University, Sch. of Matls. & Metall., Wenhua Rd., No.11, Heping Dist., Shenyang, Liaoning Province 110004 China

The method of directly foaming in molten Al to prepare closed-cell aluminum foam is described in this article. Different kinds of ingredients are put into the molten Al alloy to make its viscosity increased. The TiH2 as foaming agent is also added into these molten Al to fabricate aluminum foam. The effectiveness of the method to increase the viscosity of molten Al on foam structure is researched by macroscopic observation and microstructure analysis. The results are showed as follow. After these ingredients, such as calcium, magnesium, Al2O3 and coal ash, etc, had been added into molten Al, the phase component of Al alloy changed. However the phase component and the mechanism of its increasing viscosity is different slightly. Meanwhile, the obtained Al foam also has great difference in its structure. The foams obtained by calcium and coal ash hold thick cell wall and high intensity, while the foams obtained by magnesium and Al2O3 hold thin cell wall and low intensity.

Computational Thermodynamics and Phase Transformations: Phase Field Models and Related Methods

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Thursday AM Room: 3005
February 17, 2005 Location: Moscone West Convention Center

Session Chair: Christopher Mark Wolverton, Ford Motor Company, Scientific Rsch. Lab., Dearborn, MI 48121-2053 USA

8:30 AM Invited
Bridging the Scales Between Thermodynamic, Microscopic and Macroscopic Calculations When Simulating Solidification Microstructures: *Peter D. Lee*[1]; Robert C. Atwood[1]; [1]Imperial College London, Matls., Prince Consort Rd., London SW7 2BP UK

Models of phase transformations have proved very beneficial when investigating microstructural features when the representative volume element (RVE) is only one or two orders of magnitude larger then the

spatial discretization required to encapsulate the relevant physics. However, during solidification in particular, microstructural features are formed through phenomena occurring over multiple length scales due to the interaction of crystallographic, thermodynamic, and diffusion processes. This limits the applicability of RVE techniques. Therefore other approximations must be made to bridge the scales and allow the influence of microstructural features on the final performance of engineering components to be simulated. This paper presents an approach to bridging these scales by combining thermodynamic data into a microscale model and then weakly coupling these results into macroscopic heat transfer models.

9:00 AM
Phase-Field Modeling of the Irregular Interface Morphology During Directional Solidification: *Taiming Guo*[1]; Guoxiang Wang[1]; [1]University of Akron, Dept. of Mechl. Engrg., Akron, OH 44325-3903 USA

Depending on the relative strength of the anisotropy of the surface tension, a growing solidification interface may develop into various distinguished patterns. This paper employs the phase-field model with both the anisotropy of the surface tension and the anisotropy of the kinetic attachment to simulate such interface patterns observed in the directional solidification experiments. Not only the regular interface patterns (planar, cellular and dendritic patterns), but also the irregular interface patterns (tilted dendritic, degenerate, seaweed, doublon and doublet) have been successfully reproduced. The parameters of the temperature gradient, the anisotropy of the surface tension, the anisotropy of the kinetic attachment and the tilt angle are studied in the model. It has been found that the irregular interface patterns occur with a weak anisotropy of the surface tension and is strongly affected by the anisotropy of the kinetic attachment. On the basis of the simulation results, a morphology diagram is constructed with respect to the anisotropy of the surface tension and the anisotropy of the kinetic attachment.

9:20 AM
Online-Coupling of Thermodynamic Databases to a Multi-Phase-Field Model - Application to Hypereutectic Aluminum Casting Alloys: *Bernd Böttger*[1]; Ingo Steinbach[1]; [1]ACCESS e.V., Intzestr. 5, Aachen 52072 Germany

Due to the increasing computer performance phase-field methods allow more and more realistic microstructure simulations for practical foundry problems. If many alloy components and different phases are involved the exact description of the thermodynamic properties becomes absolutely critical for getting realistic simulation results. Ideal solution approximations, linear phase diagrams or tie-line tables are helpful only in special cases. In this conference paper the online-coupling of CALPHAD-databases to the phase-field software MICRESS using the Thermo-Calc Fortan interface is presented. The phase boundary conditions are described by a quasi-equilibrium model which uses the driving-force as an additional degree of freedom and also includes metastable interfaces. This general approach can be applied to various types of alloys for which databases are available and for an arbitrary number of alloy components and phases. The model is applied to hypereutectic aluminum casting alloys, which exhibit a rather complex solidification sequence and microstructure. Systematic variation of local solidification parameters is used to obtain a general knowledge about microstructural parameters, which can be later used in process optimization.

9:40 AM
Kinetics of Primary Crystallization Studied by Phase-Field Simulations: Pere Bruna[1]; Eloi Pineda[2]; *Daniel Crespo*[1]; [1]Universitat Politecnica de Catalunya, Dept. Fisica Aplicada, Escola Politecnica Superior de Castelldefels, Avda. del Canal Olimpic s/n, Castelldefels 08860 Spain; [2]Universitat Politecnica de Catalunya, Dept. de Fisica i Enginyeria Nuclear, ESAB, Urgell 187, Barcelona 08036 Spain

Primary crystallization of metallic glasses usually results on a high density of nanocrystallites of limited size; this fact usually leads to an improvement in the macroscopic properties of the glass. The diffusion of those species lacking or exceeding in the crystallized phase is known to stabilize the precursor phase, inducing a non-random nucleation; besides, the overlapping of the concentration profiles around neighbor growing grains reduces the growth rate (soft impingement). Although calorimetric data of these transformations was modeled by introducing empirical descriptions of soft-impingement on the Avrami framework, realistic phase-field simulations with constant diffusion coefficient cannot account for the observed experimental delays in the transformation rate. Thus, the change in the relaxation properties of the amorphous phase due to the local varying composition, usually neglected, may modify the local diffusion properties. In this work, a 3D phase-field simulation of a primary crystallization with a composition dependent diffusion is presented.

10:00 AM
2D and 3D Phase Transformations in Ternary Polymeric Membrane Systems with Variable Mobilities and Viscosity: *Bo Zhou*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-043, Cambridge, MA 02139 USA

Most commercial polymeric membranes are made from nonsolvent/solvent/polymer ternary systems by immersion precipitation. In this work, a ternary Cahn-Hilliard formulation incorporating a Flory-Huggins homogeneous free energy function is used to study the kinetics and the phase behavior of the immersion precipitation process. The Water/DMF/PVDF ternary system with a two-layer polymer-solvent/nonsolvent initial condition is used to simulate actual membrane fabrication conditions. 2D and 3D simulation results show the membrane morphology evolution during the spinodal decomposition. The simulated final morphologies show an asymmetric structure of membranes, which strongly agrees with the experimental observation. Simulations with different initial compositions show membrane morphology changes from isolated droplets to bicontinuous patterns. Furthermore, the effects of concentration-dependent polymer mobility are studied. In addition, the Navier-Stokes equations are coupled with this ternary system to model hydrodynamics with concentration-dependent viscosity in 2D and 3D. The results show that fluid flow destabilizes the top layer of membrane, with larger surface tension and smaller viscosity making the top layer more unstable.

10:20 AM Break

10:30 AM Invited
Multi-Scale Phase Field Modeling of Solute Segregation at Grain Boundaries: Ning Ma[1]; Ken R. Elder[2]; Suliman A. Dregia[1]; *Yunzhi Wang*[1]; [1]Ohio State University, Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA; [2]Oakland University, Dept. of Physics, Rochester, MI 48309 USA

We investigate solute segregation at grain boundaries using both mesoscale and atomic scale phase field models. The mesoscale phase field model is based on gradient thermodynamics, and it differs from much previous work because it takes into account self consistently the effect of concentration gradient, spatial variation of gradient-energy coefficient and concentration dependence of solute-boundary interactions. The model predicts a sharp transition of grain boundary mobility as a function of temperature, which is related to the sharp transition of solute concentration of grain boundary as a function of temperature. The mesoscale model is, however, limited by its assumption of a uniform solute-boundary interaction potential along a boundary. The atomic scale phase field model is based on the phase field crystal model, which implements the phase field kinetic equations using atomic-resolution, time-averaged particle density as one of the field variables. The model accounts self-consistently for defect core structures at different types of grain boundaries. Different characteristics of solute distribution at different types of grain boundaries and its effect on subsequent spinodal decomposition are discussed, and results are compared with those obtained from the mesoscale phase field model.

11:00 AM Invited
Lattice Mismatch and Microstructure Evolution in Ni-Base Alloys: T. Wang[1]; J. Zhu[1]; Y. Wang[1]; S. H. Zhou[1]; Z. K. Liu[1]; *L. Q. Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 102 Steidle Bldg., Univ. Park, PA 16802 USA

Ni-based superalloys consist of ordered intermetallic gamma-prime(Ni3Al) precipitates embedded in a disordered face-centered cubic (fcc) gamma matrix. The morphology of gamma-prime precipitates is critically dependent on the lattice parameter differences between the precipitates and matrix. This presentation will describe our recent efforts for studying the compositional dependence of lattice parameters of gamma and gamma-prime within the CMSX series of superalloys using a combination of first-principles calculations and the CALPHAD approach as well as for extending our phase-field simulations of gamma-prime precipitate microstructure evolution beyond binaries. In particular, the effect of various solute substitutions on the lattice parameter will be studied and compared with existing experimental measurements. The compositional dependence of gamma-prime precipitate morphologies and coarsening kinetics is systematically studied. Finally, the possibility of further extending the phase-field models to high-order multicomponent alloys will be discussed.

11:30 AM
A Phase-Field Model of Microstructural Evolution in Ferromagnetic Shape Memory Alloys: *Todd M. Heil*[1]; William T.

Reynolds[1]; [1]Virginia Tech, Matls. Sci. & Engrg. Dept., Ste. 302 Collegiate Sq., MC 0286, Blacksburg, VA 24061 USA

A three-dimensional computational model is employed to simulate a proper martensitic transformation and a magnetic transition in ferromagnetic shape memory alloys. The model's free energy functional is based upon phase field microelasticity and micromagnetic theories; terms are included to account for energy contributions from composition, temperature, variant boundaries, elastic strain, magnetocrystalline anisotropy, magnetic domain walls, magnetostatic potential, and external applied magnetic fields. The model tracks the microstructural and magnetic responses to applied temperature, stress, and/or magnetic fields. Model parameters are fitted to the physical properties and transformation temperatures from a series of ferromagnetic shape memory alloys in the Ni-Fe-Ga system. Magnetization and microstructural features predicted by the computational model are compared with corresponding data obtained from the Ni-Fe-Ga alloys to test the validity of the model.

11:50 AM

Simulation of Cooperative Growth of Pearlite Using Multi-Phase Field Method: *Katsumi Nakajima*[1]; Markus Apel[2]; Ingo Steinbach[2]; [1]JFE Steel Corporation, Steel Rsch. Lab., 1 Kokan-cho, Fukuyama, Hiroshima 721-8510 Japan; [2]RWTH-Aachen, Access e.V., Intzestr. 5, Aachen D-52072 Germany

The phase field method has proved to be a useful numerical tool to calculate the lamellar microstructure during eutectic solidification process. In this paper, this method is applied to eutectoid transformation in solid state. A cooperative growth of pearlite is simulated for eutectoid steel by using multi-phase field model coupled with diffusion equation, taking into consideration the diffusion of carbon not only in γ-phase, but also in α-phase. A stable lamellar spacing is estimated in conditions of some undercoolings and compared with experimental results from literature and analytical models.

12:10 PM

Phase Transformations and Microstructure Evolutions in Small Confined Systems: *Yongmei M. Jin*[1]; Yu U. Wang[2]; Armen G. Khachaturyan[1]; [1]Rutgers University, Ceram. & Matls. Engrg., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]Virginia Tech, Matls. Sci. & Engrg., Blacksburg, VA 24061 USA

Novel microstructures formed in finite volumes during decomposition, ordering and martensitic transformation in thin films, precipitate particles and polycrystal grains have been investigated by using Phase Field Microelasticity model and computer simulation. It is found that the effect of free surfaces on the micromechanics of the ordering producing L10 tetragonal phase domains in free-standing thin films changes the morphology of the domain microstructures and the structure-sensitive physical properties with respect to those in bulk. The mechanism of formation of highly dense twin-related hexagonal microdomains in non-stoichiometric Al-Mg spinel providing extraordinary hardness is investigated. It is shown that the microstructure is formed due to the isostructural spinodal decomposition into two cubic phases followed by the cubic-hexagonal transformation in small Al-rich precipitates and presumably is responsible for the advanced mechanical properties of this material. The effect of finite-sized grains of a polycrystalline body on the morphology of self-accommodating multi-variant martensitic microstructure is investigated. It is shown that the elastic grain coupling influences the thermodynamics and microstructure. The long-range elastic interaction and mechanical confinement play the key role in all these microstructure formations.

General Abstract Session: Mechanical Behavior—Quasi-Static Loading

Sponsored by: TMS

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Thursday AM — Room: 2006
February 17, 2005 — Location: Moscone West Convention Center

Session Chair: Peter N. Kalu, FAMU-FSU College of Engineering, Mechl. Engrg., Tallahassee, FL 32310 USA

8:30 AM

The Room Temperature Ductility of Molybdenum with Spinel ($MgAl_2O_4$) Particles: *Joachim H. Schneibel*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA

In 1967, D. M. Scruggs published a patent on the improved room temperature ductility of Mo-spinel (Mo-$MgAl_2O_4$; Mo-$NiAl_2O_4$), as compared to nominally pure Mo. To verify this effect, molybdenum specimens containing 0, 5, 10, and 15 vol. % $MgAl_2O_4$ spinel powder were processed by powder metallurgy. Room temperature tensile tests were performed at different strain rates. The measured ductility values showed substantial scatter. Regardless of the spinel volume fraction, the ductility increased with decreasing fraction of intergranular fracture. The spinel ductilization effect, if it exists, was therefore overwhelmed by variations in intergranular strength. However, the fracture surface of one spinel-containing specimen showed local evidence for substantial ductility. In order to verify the spinel ductilization effect, work is in progress to minimize intergranular fracture, and thus the scatter in the ductility values, by controlling the trace element concentrations. This work was sponsored by the Office of Fossil Energy, Advanced Research Materials (ARM) Program, U.S. Department of Energy, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory managed by UT-Battelle, LLC.

9:00 AM

Deformation Behavior of Coarse Grained and Ultrafine Grained 5083 Al At 77 K and 298 K: *Kyung-Tae Park*[1]; Jun-Hwan Park[1]; Yong-Shin Lee[2]; Won-Jong Nam[3]; [1]Hanbat National University, Div. of Advd. Matls. Sci. & Engrg., San 16-1, Dukmyung-Dong, Yuseong-Gu, Taejon 305-719 S. Korea; [2]Kookmin University, Sch. of Mechl. & Auto. Engrg., 861-1, Chongneung-Dong, Songbuk-Gu, Seoul 136-702 S. Korea; [3]Kookmin University, Sch. of Advd. Matls. Engrg., 861-1, Chongneung-Dong, Songbuk-Gu, Seoul 136-702 S. Korea

Compression tests were conducted on ultrafine grained (UFG) 5083 Al alloy processed by ECAP and its coarse grained(CG)counterpart at 77 K and 298 K. Deformation of the CG alloy was dominated by the stage II and III hardening at 77 K and 298 K, respectively. The microstructure of the CG alloy deformed at 298 K consisted of well-defined dislocation cells but the blurred ill-defined cells were formed at 77 K. By contrast, the UFG alloy exhibited the elastic-near perfect plastic behavior at both temperatures. No dislocation cells were formed in the UFG alloy. Instead, localized shear bands were formed at the onset of plastic deformation at both temperatures. Based on the above findings, the effect of the grain size and temperature on the deformation mode of the alloy was analyzed.

9:30 AM

Low Temperature Creep Anomalies in Titanium Aluminum Alloys: *M. C. Brandes*[1]; M. J. Mills[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

In recent studies, several creep related anomalies, particularly a marked recovery of strain hardening ensuing creep deformation at ambient temperature, were documented in metallic Ti-Al alloys. These were observed in specimens that had been loaded in compression at stresses less than the yield stresses of the materials, crept to a given level of plastic strain, unloaded, naturally aged for a period of days, and reloaded at the initial stress levels. Unexpectedly, the instantaneous strain rate upon reload was found to be higher, orders of magnitude in some cases, than that prior to the initial unload. This work presents the most current observations of the variations in the recovery re-

sponses of two Ti-6 wt% Al alloys with respect to time spent in the unloaded state, exposure temperature, accumulated plastic strain, loading geometry, and microstructure, proposes thermally activated, dislocation level recovery processes that give rise to the effect, and details planned experiments and analyses that will allow for the characterization of both the phenomenology and mechanisms of the behavior.

10:00 AM Break

10:20 AM
Effects of Initial Powder Size on the Mechanical Properties and Microstructure of As-Extruded GRCop-84: *Chika L. Okoro*[1]; Peter N. Kalu[1]; David L. Ellis[2]; [1]FAMU-FSU College of Engineering, Mechl. Engrg., Tallahassee, FL 32310 USA; [2]NASA Glenn Research Center, 21000 Brookpark Rd., Cleveland, OH 44135 USA

GRCop-84 is a ternary Cu-Cr-Nb alloy having approximately 8 at% Cr and 4 at% Nb. This work focuses on characterizing the effect of varying starting powder size on the microstructural evolution and mechanical properties of as-extruded GRCop-84. Tensile tests and constant load creep tests were performed on extrusions of four powder meshes: +140 mesh (>105 •m powder size), -140 mesh (• 105 •m), -140/+270 mesh (53 - 105 •m), and -270 mesh (•53 •m). Samples were tested in tension at room temperature and at 500ºC (932°F). Creep tests were performed under vacuum at 500ºC (932°F) using a stress of 111 MPa (16.1 ksi). The fracture surfaces of selected samples from both tests were studied using a Scanning Electron Microscope (SEM). Both optical microscopy and SEM analysis were used to characterize changes within the microstructure of the as-extruded materials due to the powder size variation.

10:50 AM
The Effect of Flash Annealing on the Mechanical and Electrical Properties of Previously Used AM2 Mats Composed of Al 6061-T6: *N. Aizpuru*[1]; D. Le[1]; J. McDonald[1]; L. McLennan[1]; S. Tewfik[1]; E. W. Lee[2]; D. Piatowski[3]; J. Foyos[1]; J. Ogren[1]; J. McLennan[1]; O. S. Es-Said[1]; [1]National Science Foundation, Loyola Marymount University, Rsch. Experience for Undergrads. Prog., Mechl. Engrg. Dept., Los Angles, CA 90045-8145 USA; [2]Naval Air Systems Command, Naval Air Warfare Ctr., Patuxent River, MD 20670-1908 USA; [3]Naval Air Systems Command, Naval Air Warfare Ctr. Aircraft Div., Lakehurst, NJ 08733 USA

Used AM2 mats (aircraft landing platforms) composed of Aluminum 6061-T6 were machined into tensile and conductivity bars. Samples were then subjected to five different temperatures: 315.6, 371.1, 426.7, 482.2 and 537.8°C (600, 700, 800, 900 and 1000°F), seven different time intervals (10, 20, 30, 70, 90, 120 and 150 seconds), and eight different exposure times (1, 2, 3, 5, 7, 10, 15 ad 20 times). The ultimate strength, yield strength, and percent elongation of the samples were determined. Conductivity and hardness tests were also performed to assess the effect of thermal exposure. Used AM2 mats are available in large quantities. It would be economical if they could be used by modern aircraft with vertical take off and landing (VTOL) capabilities that expose the mats to high temperature gaseous exhausts. The objective of this work is to evaluate the mechanical properties of the mats after several simulated take off and landing exposures. These exposures are simulated by flash annealing samples taken from the AM2 mats. Another set of AM2 mats were subjected to takeoff and landing tests performed by aircraft with Vertical Takeoff and Landing (VTOL) capabilities. The hot exhaust gases from these aircraft caused discolorations on the mat surfaces. Samples were selected radially out from the heat-affected region to examine the change of mechanical and electrical properties as a function of distance from these regions and discoloration. All samples were tested for their tensile properties as well as their conductivity (IACS %) and hardness (15-T) scale. All results were plotted according to their position from an arbitrary origin common to each of the mats.

Magnesium Technology 2005: Corrosion and Surface Finishing - Magnesium Alloys

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Thursday AM Room: 2004
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Per Bakke, Hydro Aluminium Hycast AS, Porsgrunn 3907 Norway; Naiyi Li, Ford Motor Co, Ann Arbor, MI 48103 USA

8:30 AM
Corrosion Properties of Secondary AZ91 Alloys: *Carsten Blawert*[1]; Emma Morales[1]; Wolfgang Dietzel[1]; Norbert Hort[1]; Karl Ulrich Kainer[1]; Christiane Scharf[2]; Andre Ditze[2]; Frank Endres[2]; [1]GKSS Forschungszentrum Geesthacht GmbH, Ctr. for Mg Tech., Max-Planck-Str. 1, Geesthacht 21502 Germany; [2]TU Clausthal, Inst. für Metallurgie, Robert-Koch-Str. 42, Clausthal-Zellerfeld 38678 Germany

The corrosion properties of AZ91 alloys are determined by the amount of impurities, enriching in the alloy during the recycling process. However within reasonable costs the recycling of Mg scrap metal results only in AZ91B quality and respectively poor corrosion behaviour can be expected. This influence on the corrosion resistance was studied by controlled additions of Fe, Ni, Cu, and Si to AZ91D alloy. The corrosion properties of the gravity permanent mould castings were studied by various corrosion tests and correlated to the observed microstructure and phase composition. Most of the impurities were found as or in intermetallic phases. The influence on the corrosion resistance was found to be depending on the solidification behaviour of the particular intermetallic phase. Altogether the corrosion test results indicate a much higher tolerance against the impurities than expected and effects on the possible use of secondary AZ91 alloys are discussed.

8:50 AM
Intermetallic Morphology Development in AM60 Alloy: *Christopher Patrick Corby*[1]; Nigel Jeffrie Ricketts[2]; Ma Qian[3]; John Andrew Taylor[1]; [1]University of Queensland, CRC for Cast Metals Mfg., Div. of Matls., Sch. of Engrg., St. Lucia, Brisbane, QLD 4072 Australia; [2]CRC for Cast Metals Manufacturing, CSIRO Mfg. & Infrastruct. Tech., 2643 Moggill Rd., Pullenvale, QLD 4069 Australia; [3]Brunel University, Brunel Ctr. for Advd. Solidification Tech., Uxbridge, Middlesex UB8 3PH UK

It has been established that entrapped Al-Mn-Fe intermetallics can act as microgalvanic cathodes in magnesium alloys. Many studies present evidence suggesting these particles are Al8(Mn,Fe)5 or at least Al-Mn rich. However, the morphological development of these particles has not been documented before. This study investigates the development of these Al-Mn-Fe phases and presents evidence suggesting that two types of particle form in an AM60 melt. There are nearly spherical Al8(Mn,Fe)5 particles which sink in the melt and collect as sludge, and there is an Al-Mn rich phase, which may also be Al8(Mn,Fe)5, that grows over long holding times into floating intermetallic particles. These suspended particles are quite different in appearance to, and have lower Fe contents than the sludge particles. They initially form as faceted particles and then grow into complex floral structures after many hours holding time. These floral structures develop with more branching when holding temperature is higher.

9:10 AM
EIS Study of Corrosion Behaviour of AZ91 and AM50 Alloys: *Emmanuel Rocca*[1]; *Joseph Hazan*[1]; [1]University Henri Poincaré, LCSM UMR7555, BP239, Vandoeuvre-Les-Nancy 54506 France

The corrosion and electrochemical behaviour of AZ91 and AM50 magnesium alloys in aerated, unbuffered ASTM D13847 water have been investigated using steady-state polarization curves and mainly electrochemical impedance spectroscopy. Two relatively well separated capacitive loops can be generally observed in the complex plot for the stationary electrodes used which indicate that some protective behaviour is reached due to a film formed on the surface of the alloys. This passivation is stimulated by a pH increase accompanied by an increase of potential resulting in an anodic control. The second time constant seems related to a diffusion process through the film. So the corrosion resistance of the alloy containing a higher content of aluminium (AZ91) is better in comparison to the other alloy studied

(AM50). The RHF resistance value is a good parameter to determine more accurately the corrosion rates for magnesium alloys.

9:30 AM
Evaluation of Corrosion Protection Methods for Magnesium Alloys in Automotive Applications: *Gregory T. Bretz*[1]; Patrick J. Blanchard[1]; David J. Hill[1]; Robert C. McCune[1]; [1]Ford Motor Company, Rsch. & Advd. Engrg., PO Box 2053, MD3135 SRL, Dearborn, MI 48121 USA

Magnesium alloys are susceptible to galvanic corrosion. Consequently, it is often necessary to apply coatings to components for isolation purposes. However, previous publications suggest the effectiveness of commercial coatings can vary widely. Therefore, a screening study was performed to evaluate pre-treatment and coating systems currently available for use within the automotive industry. This paper focuses on a selection of conversion and anodized coatings. In many instances, these coatings were used in conjunction with either powder coat or an electro-coat to assess the additional protection offered by a supplemental barrier. Results from the study include SEM micrographs to highlight coating thickness and morphology. A correlation is then made between the SEM analysis and performance of specimens subjected to accelerated corrosion testing. Finally, sample sections are examined to show the nature of the corrosive attack, and highlight the robustness and physical protection offered by respective coating systems.

9:50 AM Break

10:05 AM
Corrosion Inhibition of Magnesium Alloys in Coolants: Guangling Song[1]; *David H. StJohn*[1]; [1]University of Queensland, Matls., St. Lucia, Brisbane 4071 Australia

A number of magnesium alloys show promise as engine block materials. However, corrosion of magnesium alloy engine components by coolant is an important issue in the automotive industry. This paper shows that the corrosion rate of magnesium was increased by dilution and contamination of ethylene glycol. Fortunately, the corrosion of magnesium in ethylene glycol can be effectively inhibited by addition of fluorides. This finding was further verified by assessing the corrosion performance of AZ91D and a recently developed engine block magnesium alloy AM-SC1 in several commercial coolants. Generally speaking, the tested commercial coolants were corrosive to the magnesium alloys in terms of general and galvanic corrosion, but an organic-acid based long-life coolant appeared to be less corrosive than a traditional coolant. It was found that both general and galvanic corrosion rates were significantly decreased by addition of KF, and there were no evident side effects on the other engine block materials, such as copper, solder, brass, steel and aluminium alloys, in terms of their corrosion performance. The ASTM D 1384 test further confirmed these results and suggested that Toyota long life coolant with a KF addition is a promising coolant for magnesium engine blocks.

10:25 AM
Characterization of Die Skin Structure and its Effects on the Corrosion Properties of the Hot-Chamber Die Casting AZ91D Thin Plate: *Jun-Yen Uan*[1]; Bing-Lung Yu[1]; [1]National Chung Hsing University, Dept. of Matls. Engrg., 250 Kuo Kuang Rd., Taichung 402 Taiwan

The corrosion of hot-chamber die cast AZ91D thin plates was investigated with reference to their microstructures, to elucidate the role of die chill skin in the corrosion. At pH 2.7 in a chloride solution, the sample with die skin corrodes at 200~250 miles per year (mpy), whereas the sample without the die skin exhibits a corrosion rate of 100~150 mpy. The two kinds of samples have similar Ecorr values of -1.45 V. However, the mean Icorr value of the sample with die skin is 150~250 ìA/cm2, whereas that of the sample without die skin is only 40~60 ìA/cm2. Immersion tests and polarization experiments reveal the inferior corrosion performance of the specimen with the die skin on the surface. The die skin structure was explored. The corrosion performance of the hot-chamber die cast thin plate with die skin is closely related to the morphology and distribution of the Al12Mg17 â phase in the matrix.

10:45 AM
Microstructure and Wear Characteristic of Laser Clad Al-12% Si, Al-30% Si and ALSI/WC on AS21 Magnesium Alloy: *Meity Natasya Mandagie*[1]; Milan Brandt[1]; Yvonne Durandet[1]; Mahnaz Jahedi[2]; [1]Swinburne University of Technology, IRIS, PO Box 218, Hawthorn, Melbourne, Victoria 3123 Australia; [2]CSIRO, Mfg. & Infrastruct. Tech., Locked Bag 9, Preston, Melbourne, Victoria 3072 Australia

Although magnesium alloys possess a set of desirable properties such as low specific weight and high specific strength that make them attractive to automotive and aerospace industries, they have poor wear and corrosion resistance compared to that of steel or aluminium alloys. This is due to their relatively low surface hardness and high chemical affinity for numerous elements. This study investigates the microstructure and wear characteristics of clad layers made of Al-12% Si, Al-30%Si, and a mixture of Al-12%Si (40%) and WC (60%). The claddings were deposited on creep resistant AS21 magnesium alloys using a high power Nd: YAG laser. The results indicate that the clad layers have better wear resistance than the substrate.

11:05 AM
The Electropolishing and Anodic Coating of AZ31 Magnesium Alloy in Anhydrous Electrolyte: *Qun Zhao*[1]; Yuanfu Zhou[1]; Ying Zhang[1]; Chunfang Zhao[1]; Yonghen Guo[1]; Xianghui Cang[1]; [1]Zhengzhou Research Institute of Chalco, Aluminum Corporation of China Limited, No.82, Jiyuan Rd., Shangjie Dist., Zhengzhou, Henan 450041 China

The metallic shining surface of AZ31 magnesium alloy strip was obtained by electropolishing process in anhydrous electrolyte, in which a thick phosphate chemical conversion coating was formed by anodic polarization. The influence of water concentration on the properties of electrolyte and the electrochemical process were investigated at the same time. The microstructure of the bright surface and anodic oxidizing coating were observed using SEM and XRD.

11:25 AM
Corrosion Protection and Repassivation After the Deformation of Magnesium Alloys Coated With a Protective Magnesium Fluoride Layer: Friedrich Wilhelm Bach[1]; *Thomas Hassel*[1]; Christian Krause[1]; Peter Wilk[1]; [1]University of Hannover, Dept. of Matls. Sci., Schoenebecker Allee 2, PZH, Garbsen, Low Saxonia D-30823 Germany

The development of a biodegradable, cardiovascular implant (stent) made from a resorbable magnesium alloy demands an accurately defined degradation profile. The corrosion protection of the stent for the first 4-6 weeks after implantation is only obtainable by usage of a surface protective coating. During this time the implant is able to grow into the vessel. After the steady solution of the coating the base material is resorbed by a normal corrosion process in chloride media. During the implantation the material has to tolerate partial deformations. The influence of the induced micro-cracks during the deformation process has been analysed in a 4-point bending test combined with an electrochemical corrosion test. The material system consists of a Mg(Ca<1wt%)-alloy and a dense MgF2-coating. The coating process is based on the conversion of the natural layer by a treatment with hydrofluoric acid. The specimens show micro-cracks after the deformation but no increased corrosion activity. The measurement of the corrosion current indicates a repassivation of the surface. Detailed EDX and REM analyses prove the regeneration of Mg(OH)2/MgO on the crack ground.

11:45 AM
Mg2Si Coating Technology on Magnesium Alloys to Improve Corrosion and Wear Resistance: *Takashi Yamaguchi*[1]; Katsuyoshi Kondoh[2]; Tadashi Serikawa[2]; Momoko Henmi[2]; Hideki Oginuma[2]; [1]Gifu Prefectural Science and Technology Promotion Center, 4-179-1, Sue-Cho, Kakamigahara-City, Gifu pref. Japan; [2]University of Tokyo, Rsch. Ctr. for Advd. Sci. & Tech., 4-6-1 Komaba, Meguro-ku, Tokyo Japan

Magnesium silicide (Mg2Si) bulky material has a possibility to improve the surface function of light metals such as magnesium and aluminum alloys due to its superior corrosion resistance to the conventional stainless steel and high mechanical properties. In this study, Mg2Si thin film coated on AZ31 magnesium alloys by using a high frequency sputtering method was examined. Salt spray test to evaluate the corrosion resistance indicated that AZ31 substrate with Mg2Si coating was hardly damaged after 240h. On the other hand, non-treated one was corroded in only 1h. Concerning the wear resistance under oil lubricant test, a friction coefficient, ¯¬ of the AZ31 alloy with Mg2Si film is remarkably stable in employing S35C steel as a counter specimen. In the combination of AZ31 alloy disc and S35C pin specimens, seizure and sticking phenomena occurred and ¦Ì value suddenly increased. Accordingly, Mg2Si coating technology is a suitable surface modification processing to improve corrosion and wear resistance of magnesium alloys.

Materials Issues for Advanced Nuclear Systems: Materials Compatibility

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Robert J. Hanrahan, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA

Thursday AM Room: 3012
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Sean M. McDeavitt, Purdue University, Nucl. Engrg., Purdue, IN USA; Robert J. Hanrahan, Los Alamos National Laboratory, NNSA, Washington, DC 22209 USA

8:30 AM
SiC/SiC for Advanced Reactors: *Russell H. Jones*[1]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., PO Box 999, Richland, WA 99354 USA

Composite materials have the potential for their properties to be tailored to specific applications by engineering the combination of fibers and matrices. Ceramic matrix composites are attractive because of their excellent high-temperature properties and corrosion resistance. In particular, ceramic composites made from silicon carbide fibers and silicon carbide matrices (SiCf/SiC) are promising for nuclear applications because of the radiation resistance of the b phase of SiC, their excellent high-temperature fracture, creep, corrosion and thermal shock resistance. The b phase of SiC has been shown by numerous studies to have a saturation swelling value of about 0.1 to 0.2% at 800 to 1000 C. This suggests that composites of SiC/SiC have the potential for excellent radiation stability. The continuous fiber architecture, coupled with engineered interfaces between the fiber and matrix, provide excellent fracture properties and fracture toughness values on the order of 25 MPa m 1/2. The strength and fracture toughness are independent of temperature up to the limit of the fiber stability. Also, these fiber/matrix microstructures impart excellent thermal shock and thermal fatigue resistance to these materials so start-up and shut-down cycles and coolant loss scenarios should not induce significant structural damage.

9:00 AM
Fuel-Cladding Compatibility in Metallic Nuclear Fuels: *Dennis D. Keiser*[1]; James I. Cole[1]; [1]Argonne National Laboratory-West, Engrg. Tech. Div., PO Box 2528, Idaho Falls, ID 83403-2528 USA

In advanced nuclear systems, the chemical compatibility of the fuel and cladding is imperative for safe operation of a reactor. Interactions between fuel and cladding during irradiation can result in the formation of strength reducing zones in the cladding and the formation of compositional zones with melting points below the anticipated operating temperatures. Therefore, it is essential to limit the interdiffusion between the fuel and cladding. To better understand the compatibility of metallic fuel and cladding, diffusion experiments were conducted at prototypic temperatures using fuel alloys and various stainless steel claddings. Additionally, diffusion experiments were performed using fuel, cladding, and materials that may act as barriers to fuel-cladding interaction (viz., V, Zr, and Ta). The various annealed diffusion couples were analyzed using scanning electron microscopy and transmission electron microscopy to investigate the kinetics of the fuel-cladding interactions and to identify the types of phases that formed. The results of these analyses will be described, along with the effectiveness of the barrier materials at impeding interdiffusion between the fuel and cladding alloys.

9:30 AM
Effects of Irradiation on Materials for Advanced Gas Cooled Reactors: *Wolfgang Hoffelner*[1]; Manuel Alexandre Pouchon[1]; Jiachao Chen[1]; [1]Paul Scherrer Institute, Nuclear Energy & Safety, Villigen PSI CH-5232 Switzerland

Gas cooled reactors are considered as future plants for sustainable co-generation of electric energy and heat (Generation IV initiative). For advanced plant designs high temperature materials like ODS or intermetallics become important. The interaction of irradiation induced defects, transmutation products and eventual grain boundary voids with diffusion controlled processes like creep are life-limiting factors. Samples of the ferritic ODS alloy PM 2000 (Plansee) and of a lamellar, W-containing titanium aluminide (ABB-2) were subjected to He-implantation, in order to investigate changes in microstructure and swelling behaviour of the material. The implantations were performed at different temperatures (room temperature up to 1000 C). The ODS material was irradiated with 1.5 MeV under four different angles to get a homogeneous damage profile. The investigated damage range was 0.25 to 2 dpa. Swelling was measured with atomic force microscope (AFM). Irradiation of the TiAl was done at an energy of up to 24 MeV. Microstructural damage analysis was performed with TEM. The results are discussed with respect to expected changes of mechanical properties.

10:00 AM Break

10:20 AM
Selection of a Canister Crucible Material for the Mobile Melt-Dilute Process: *Brian Robert Westphal*[1]; Dave A. Sell[1]; Dennis D. Keiser[1]; [1]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403-2528 USA

As part of the mobile melt-dilute project for the processing of spent nuclear fuel in the Former Soviet Union (FSU), the selection of a canister crucible is of considerable importance for containment of a molten fuel matrix. During the process, a highly enriched aluminum-uranium fuel is diluted isotopically to satisfy proliferation concerns. The crucible material must sustain the high temperature dilution operation as well as be compatible with either storage or subsequent processing schemes. In addition, the crucible material must be readily available in the FSU and easily fabricated into a canister design. A materials testing program was initiated to identify the compatibility of potential crucible materials with the mobile melt-dilute process. Following a series of preliminary screening tests, scale-up testing was performed with the crucible material of choice and an aluminum-uranium feed. Results from the testing program are presented along with a justification for the material selection.

10:50 AM
Systematical Investigation on the Luminescence Enhancement of PbWO4 Crystals by Doping and Annealing: *Yanlin Huang*[1]; [1]Zhong Yuan Institute of Technology, Dept. of Mechl. Engrg., 41 Zhongyuan W. Rd., Zhengzhou, Henan 450007 China

PbWO4 crystal has been chosen for a scintillating detector at the Large Hadron Collider (LHC) in CERN due to its high density, short radiation length and fast decay time. Extensive investigations have been done around the world to improve the scintillation performance by annealing treatment and aliavalent ion doping in the crystals. The works in this report are focused on the enhancement of light yield in PbWO4 crystals by doping and annealing, and excellent scintillation performance still are kept for this material. The doping ions in the crystal include monovalent ions, trivalent ions and co-doping between different aliavalent ions. Many results were first report in the material. Meanwhile, the annealing mechanism and aliavalent ion doping mechanism from viewpoint of microstructure were also discussed. Besides, these results reveal also that PWO might have potential use in the PET material, even in the optoelectronic application.

11:20 AM
Oxide Layers on the High Cr Steels After SCW Corrosion: *Jinsung Jang*[1]; Chang Hee Han[1]; Yong Sun Yi[1]; Seong Sik Hwang[1]; Yongbok Lee[2]; [1]KAERI, Nucl. Matl. Tech. Dvlp. Div., 150 Dukjin-dong, Yuseong-gu, Daejeon 305-353 S. Korea; [2]KRISS, Ctr. for CMR Matls., 1 Doryong-dong, Yuseong-gu, Daejeon 305-340 S. Korea

Among the candidate materials for Generation IV SCWR (Supercritical Water-cooled Reactor) four high Cr steels and one Fe-based O.D.S alloy specimens were investigated. After the SCW corrosion tests in the temperature from 400 to 627 C under 25 MPa and the weight change measurement, the oxide layers were analyzed using a grazing incidence X.R.D, S.E.M and T.E.M. Cross sectional view of the oxide scale on the 9Cr steel specimen after the corrosion test was found to consist of three distinctive layers. The outermost layer with about a 35 micron thickness after 200 hr at 627 C was identified to be Fe3O4, and the intermediate layer of about 25 micron thickness was revealed to be Cr partitioned (Fe,Cr)3O4. The innermost layer next to the matrix was the internally oxidized zone. Oxygen atoms apparently diffused along the grain boundaries and the lath boundaries, forming oxide phase of (Fe,Cr)3O4 or (Fe,Cr)2O3 along the boundaries.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Thin Films and Multilayers

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Thursday AM Room: 3000
February 17, 2005 Location: Moscone West Convention Center

Session Chair: H. Y. Yu, US Army International Technology Center - Pacific, Asian Rsch. Office, Minato-Ku, Tokyo 106-0032 Japan

8:30 AM Invited
Influence of Cathode Oxide Films on Copper Nucleation During Electrodeposition: Hyunku Chang[1]; Byung-Hak Choe[2]; *Jong K. Lee*[3]; [1]Sungkyunkwan University, Sch. of Applied Matls. Engrg., Suwon 440-746 Korea; [2]Kangnung National University, Dept. of Metallurgl. Engrg., Kangnung 210-702 Korea; [3]Michigan Technological University, Dept. of Matl. Sci. & Engrg., Houghton, MI 49931 USA

Copper electrodeposition has an important industrial role because of various interconnects used in microelectronic devices. A typical deposition process utilizes a titanium cathode as the finished copper foil is easily separable from the thin passive TiO2 oxide layer, 2 to 3 nm thick, of the cathode. In this work, the effect of the oxide layer on copper nucleation was studied through both "ex-situ" and "in-situ" nano-scratch tests. In the case of ex-situ tests, an MTS-XP indenter was employed to induce scratches onto the cathode surface as a function of a load up to 0.6 Newtons. For "in-situ" experiments, the cathode surface was, while immersed in the electrolyte cell, scratched with a blade. In both cases, higher copper cluster densities, by a factor of 10 to 100, were observed along the scratch lines, indicating that dislocations were favored nucleation sites for copper deposition. A pipe tunneling mechanism along dislocation cores was proposed to account for the enhanced nucleation rate. With both a reduced energy barrier and a reduced barrier thickness, a dislocation line is considered to be a high electron tunneling path within an oxide layer of a large band energy gap.

8:55 AM Invited
Microbridge Testing of Thin Films: *Tong-Yi Zhang*[1]; [1]Hong Kong University of Science and Technology, Dept. of Mechl. Engrg., Clear Water Bay, Kowloon Hong Kong

In the present work, we summarize the novel microbridge testing method for thin films including single-layer, bilayer, and trilayer thin films. The samples for microbridge tests were prepared with the microelectromechanical fabrication technique such that they were easy to be handled with. The microbridge test was conducted with a load and displacement sensing nanoindenter system equipped with a microwedge probe. In mechanics analysis of the microbridge deflection versus load under large deformation, we modeled the substrate deformation with three coupled springs and considered residual stress in each layer, thereby resulting in a closed-form formula. The microbridge testing method allows us to simultaneously evaluate the Young's modulus, residual stress and bending fracture strength of single-layer films from an experimental load-deflection curve. For bilayer and trilayer films, the analysis shows that bending of a bilayer or trilayer beam is equivalent to the bending of a single-layer beam with an equivalen t bending stiffness, a residual force and a residual moment. Therefore, the closed formula is able to simultaneously evaluate the equivalent bending stiffness, the residual force and the bending strength from the microbridge test. In general, one can estimate the Young's modulus and residual stress in one layer if the corresponding values in the other n-1 layers are known. Alternately, we may first take the slope of a load-deflection curve under small deformation, which gives the relationship between the bending stiffness and the residual force of a bilayer or a trilayer microbridge. Then, using this relationship, we were able to evaluate the Young's modulus of two kinds of materials composing the bilayer or trilayer film and the average residual stress of the film simultaneously. All the theoretical formulas have been verified experimentally on single-layer, bilayer and trilayer films.

9:20 AM Invited
Piezoelectric Polarization Induced Two Dimensional Electron Gases in AlGaN/GaN Heteroepitaxial Structures: An Application for Micro-Pressure Sensors: *S. N.G. Chu*[1]; F. Ren[2]; S. J. Pearton[3]; B. S. Kang[2]; S. Kim[2]; B. P. Gila[3]; C. R. Abernathy[3]; J. Lin[4]; [1]Multiplex Inc., S. Plainfield, NJ 07080 USA; [2]University of Florida, Dept. of Cheml. Engrg., Gainesville, FL 32611 USA; [3]University of Florida, Matls. Sci. & Engrg., Gainesville, FL 32611 USA; [4]University of Florida, Electl. Engrg., Gainesville, FL 32611 USA

The wurtzite group-III nitrides exhibit piezoelectric polarization along their c-axis. Differential piezoelectric and spontaneous polarizations in strained AlGaN/GaN heterostructure grown on [0001] sapphire substrates induce two-dimensional electron gas (2DEG) at the AlGaN/GaN hetero-interface. AlGaN/GaN high electron mobility transistors (HEMT) based on electrical polarization induced 2DEG are therefore sensitive to the applied stresses. We demonstrate the feasibility of fabrication of such devices and their response to the applied stresses. Since these devices can be made onto small thin GaN membranes by removing locally the sapphire substrate using the present state-of-the-art micro-fabrication technologies, pressure sensors in the micrometer scale are possible. AlGaN/GaN/sapphire material system is ideal for high temperature applications as well as for hush chemically environments. It can also be a potential micro-sensor for medical implant applications. A detailed theoretical analysis of the dependence of trans-conductance of HEMT device on the applied stresses is provided to give insight to the stress response mechanism of the device.

9:45 AM Invited
Layer Piling Effect on Mechanical Properties of Triple-Layer Disc: *Der-Ray Huang*[1]; Tzuan-Ren Jeng[1]; Huei-Wen Yang[1]; [1]Industrial Technology Research Institute, Opto-Elect. & Sys. Labs., Chutung, Hsinchu Taiwan

To approach higher storage density, multi-layer optical disc that fabricated by photo-polymer UV curing method has become popular subject recently. In this paper, a triple layer optical disc made by new piling method has been developed. A 0.6mm thick polycarbonate substrate with first layer data (L0) is obtained from the typical injection mold. Then a silicon film was sputtered on it as first reflection layer. For second data layer (L1), one special kind of duplication method is introduced. A stamper with L1 data pit is applied as the replication source. In the next step, an UV cured acrylic resin was dispensed and spin-coated on L0. After pressing and UV curing, the stmaper could be easily separated from L0 substrate. L1 is found to stack on the L0. AgTi film is sputtered on the top of L1 as second reflection layer. As for third data layer (L2) the process is similar to L1. AgTi film is sputtered on L2 surface as third reflection layer. After bonding with a 0.6mm dummy substrate, a triple-layered disc is obtained. The surface roughness of each layer can be measured with atomic force microscope. The first surface roughness of L0 comes from ultra precision injection molding process. The data jitter of the first layer L0 is easy to be less than 7%. However, for L1 and L2, the roughness arises from the combination of photo-polymer molecular size and layer piling effect. Thus L1 and L2 with worse data jitter are expected. And from the dish measurement, we can learn the effect of stress created from layer adhesive process. The detail experimental data of the static and dynamic measurement result will be discussed.

10:10 AM Invited
Residual Stress in DVDR: *Hsueh-Lung Cheng*[1]; Der-Ray Huang[2]; Tzuan-Ren Jeng[2]; Wen-Yih Liao[1]; Sanboh Lee[1]; [1]National Tsing Hua University, Matls. Sci. & Engrg., Hsinchu Taiwan; [2]Industrial Technology Research Institute, Opto-Elect. & Sys. Labs., Chutung, Hsinchu Taiwan

Digital Versatile Disc Recordable (DVDR) consists of dye layer, reflective layer and protective layer deposited on polycarbonate (PC) substrate of thickness 0.6mm. First, the dye layer was coated on PC substrate using a spin coater. Second, a layer of silver as a reflective layer is deposited on dye layer using a DC sputter. Third, a new PC substrate as a protective layer was bonded on the reflective layer using UV curable glue. The curvatures before and after deposition of each process were measured. The residual stresses in each layer were calculated when curvature and elastic constants of each layer are given. It is found that after the protective layer was bonded on the reflective

layer, the residual stresses in protective layer increases with increasing thickness of reflective layer. The effect of dye thickness on residual stresses is also included.

10:35 AM
Thermoelastic Bending of Multilayer Structures: *Xinzhong Zhang*[1]; J. C.M. Li[1]; [1]University of Rochester, Dept. of Mechl. Engrg., Rochester, NY 14627 USA

The bending curvature due to strain mismatch in a multilayer strip or plate structure is analysed. The analytical results are compared with finite element computations. Early results of Stoney and Timoshenko are confirmed. For multilayer plates there is a critical curvature after which bifurcation takes place, namely, the plate will bend in two curvatures. This bifurcation limit is shown also by finite element analysis. Work supported by NSF through DMR-9623808 monitored by Bruce MacDonald.

10:55 AM Break

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Shock Compression

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Thursday AM Room: 3000
February 17, 2005 Location: Moscone West Convention Center

Session Chair: J. C.M. Li, University of Rochester, Mechl. Engrg. Dept., Rochester, NY 14627 USA

11:00 AM Invited
Effect of Shock Compression Method on the Defect Substructure in Single Crystal Cu: *Bu Yang Cao*[1]; Marc Andre Meyers[1]; David H. Lassila[2]; Yong Bo Xu[3]; Bruce A. Remington[2]; Chongxiang Huang[3]; Matt S. Schneider[1]; Daniel H. Kalantar[2]; [1]University of California, Matls. Sci. & Engrg., 9500 Gilman Dr., UCSD 0411, La Jolla, CA 92093 USA; [2]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; [3]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal, Shenyang, Liao Ning 110016 China

Monocrystalline copper samples with orientations of [001] and [221] were shocked at pressures ranging from 20 GPa to 60 GPa using two techniques: direct drive lasers and explosively driven flyer plates. The pulse duration for these techniques differed substantially: 2 ns for the laser experiments and 1 us for the flyer plate experiments. Defects were investigated by transmission electron microscopy and an analytical model of homogeneous dislocation nucleation is proposed. The residual microstructures were dependent on orientation and pressure. The samples shock compressed by flyer plate showed ample evidence of recrystallization and major microstructural reorganization at amplitudes of 47 GPa, whereas the laser shock compressed specimens retained shock induced microstructures up to 60 GPa. The post-shock cooling rates in laser shock compression are orders of magnitude higher than in the flyer-plate shock compression. Because the pulse duration is short in laser shock experiments, the specimens are rapidly quenched and only limited dislocation motion and post shock recovery processes occur. These differences are studied using an analytical model of thermal diffusion. This study demonstrates the unique advantage of laser shock compression in recovery experiments. Research funded by DOE and LLNL.

11:25 AM
Characterization and Modeling of Laser Induced Shock Compression of NiAl Single Crystals: *Chyi Hwang Lim*[1]; [1]Arizona State University, Mechl. & Aeros. Engrg., PO Box 876106, Tempe, AZ 85287-6106 USA

Direct drive laser irradiation tests have been performed on monocrystalline NiAl samples 150 to 300 microns thick and 5 mm in diameter. The samples were subjected to shock pressures of 10.3 GPa for the <100> loading direction and 19.4 GPa for <110> and <111> loading directions to study anisotropic material response under high strain rate deformation. Analyses of recovered samples were performed using Orientation Imaging Microscopy (OIM) revealing a region of high lattice rotation spanning a thickness of about 20 microns from the shocked surface. It was found that the magnitude of the lattice rotation measured correlates to the total number of potentially active slip systems of the {110}<001> and{100}<010> type with <001>, <011> and <111> loading axes having zero, six and nine potentially active slip systems, respectively. An incremental single crystal plasticity model was used to model NiAl behavior, including lattice rotation, for the simple case of uniaxial compression.

11:45 AM Closing Remarks: Dr. J. C.M. Li

Microstructural Processes in Irradiated Materials: Mechanical Behavior of Irradiated Materials

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Thursday AM Room: 3011
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Hideki Matsui, Tohoku University, Inst. for Matls. Rsch. Japan; G. Robert Odette, University of California, Dept. of Matls., Santa Barbara, CA 93106 USA; Yoshitaka Matsukawa, Oak Ridge National Laboratory, Metals & Ceram., Oak Ridge, TN 37831-6138 USA

8:30 AM
Microstructural Analysis of Deformation in Neutron-Irradiated FCC Materials: *N. Hashimoto*[1]; T. S. Byun[1]; K. Farrell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, Oak Ridge, TN 37831-6136 USA

Plastically deformed microstructures in neutron-irradiated face centered cubic (fcc) materials, copper, nickel and 316 stainless steel, were investigated by transmission electron microscopy (TEM). Particular emphasis is placed on the deformation microstructure responsible for the changes in mechanical behavior. Neutron irradiation at low temperature up to 1 displacement per atom (dpa) induced a high number density of small loops, stacking fault tetrahedra (SFT) and Frank loops, which resulted in irradiation-induced hardening. Deformation of irradiated fcc materials induced various microstructures, such as dislocation pileups, stacking faults, twins, and dislocation channels. At higher irradiation doses (0.1~12 dpa), dislocation channeling became the dominant deformation mode in fcc materials. In the 316 stainless steels irradiated to 0.1~0.8 dpa, the deformation microstructure consisted of a mixture of dislocation bands, tangles, twins, dislocation channels, and also martensite phase. Deformation-induced martensite transformation tends to occur with dislocation channeling, suggesting that localized deformation could lead to transformation of martensite at a high stress level.

8:50 AM
Direct Observation of SFT-Dislocation Interaction Process at Low Temperature: *Yoshitaka Matsukawa*[1]; Yuri N. Osetsky[1]; Roger E. Stoller[1]; Steven J. Zinkle[1]; [1]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, Oak Ridge, TN 37831-6138 USA

The formation mechanism of localized defect-cluster-free zones (dislocation channeling) during deformation is of interest for understanding ductility reduction in neutron-irradiated metals. The stacking fault tetrahedron (SFT) is a major vacancy cluster produced by neutron irradiation in fcc metals; however, the SFT annihilation mechanism is hard to predict due to its complicated defect geometry. We

have recently reported SFT annihilation in quenched gold during TEM in-situ straining experiments at room temperature. Here we present the results at low temperature, where vacancy migration is suppressed in gold. A large SFT (¡Ö50nm) was collapsed by direct interaction with moving dislocations at 173K, in a manner similar to previously reported room temperature results. Possible mechanisms for the SFT collapse by dislocations will be discussed: the present results indicate that the vacancy migration is not a crucial factor for the SFT collapse mechanism.

9:10 AM
Understanding Radiation Hardening and the Conditions Promoting Localized Deformation in Neutron-Irradiated Copper: *Dan Edwards*[1]; Bachu N. Singh[2]; [1]Pacific Northwest National Laboratory, Matls. Struct. & Performance Grp., PO Box 999, MSIN P8-16, Richland, WA 99354 USA; [2]Risø National Laboratory, Matls. Rsch. Dept., Rosklide DK-4000 Denmark

The phenomenon of radiation hardening and plastic flow localization in the form of dislocation channels has been observed for more than 40 years in neutron irradiated materials, but a clear understanding of the processes the control these two phenomena elude us. This presentation will highlight the results of several recent experiments investigating the microstructural features that control the deformation behavior of irradiated copper. These experiments include in-situ tensile experiments studying the microstructural evolution in materials subjected simultaneously to neutron irradiation and tensile loading at 90°C, post-irradiation annealing experiments on neutron-irradiated copper, and finally a series of interrupted tensile tests on irradiated copper evaluating the strain-dependent evolution of localized deformation. These experimental results lead to the conclusion that the radiation hardened matrix becomes increasingly unable to activate dislocation sources within grain interiors and deform homogenously, instead, dislocation channels are initiated at high stress concentrations at interfaces.

9:30 AM
Microstructure of Neutron-Irradiated Iron Before and After Tensile Deformation: *Steven J. Zinkle*[1]; Bachu N. Singh[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6138 USA; [2]Risoe National Laboratory, Matls. Rsch. Dept., Postbox 49, Roskilde DK-4000 Denmark

Tensile specimens of pure Fe were neutron irradiated at ~70 C in the HFIR and HFR test reactors to displacement dose levels of 0.0001 - 0.72 dpa. Irradiated specimens were characterized using transmission electron microscopy (TEM). Visible defect clusters were not detectable by TEM for doses below ~0.001 dpa. Both the density and average size of the dislocation loops increased with increasing dose level. Heterogeneous rafts of dislocation loops were visible for doses above ~0.2 dpa. The irradiation led to an increase in the yield stress and a decrease in the uniform elongation as a function of increasing dose. Examination of specimens after tensile deformation revealed localized deformation in the form of cleared dislocation channels. Relatively few channels were formed in the uniform elongation region of the gage section. Dislocation channels on multiple slip systems were observed in the deformation region near the fracture surface, presumably due to multiaxial stress state.

9:50 AM Break

10:20 AM
Molecular Dynamics Simulations of Dislocations Interacting with Vacancy Clusters in Face-Centered Cubic Metals: *Erik Bitzek*[1]; Daniel Weygand[1]; Peter Gumbsch[2]; [1]University of Karlsruhe, IZBS, Kaiserstr. 12, Karlsruhe 76131 Germany; [2]Fraunhofer Institut fuer Werkstoffmechanik IWM, Wöhlerstraße 11, Freiburg 79108 Germany

To study the hardening effect of vacancy clusters we performed atomistic simulations of straight edge and screw dislocation segments interacting with nano-scale voids. Static simulations with embedded atom potentials for nickel and aluminum were used to determine the obstacle strength of vacancy clusters. Molecular dynamics (MD) simulations were performed to study the dynamics of the dislocation - vacancy interaction. Depending on the temperature, the stress required for the approaching dislocation to pass the obstacles can be significantly lower compared to the static simulations. For the low temperature regime this dynamical effect can be attributed to the dislocation inertia. To asses the importance of dynamical effects for various dislocation - obstacle configurations, inertial effects were implemented in a discrete dislocation dynamics (DDD) simulation. With the required parameters (dislocation mass, drag coefficient and obstacle strength) determined from MD simulations, the DDD model reproduced the atomistic results of the dislocation - void interaction.

10:40 AM
Dynamics of Edge Dislocation Interaction with Self-Interstitial Clusters in Iron: Zhouwen Rong[1]; *David J. Bacon*[1]; Yuri N. Osetsky[2]; [1]University of Liverpool, Dept. of Engrg., Brownlow Hill, Liverpool L69 3GH UK; [2]Oak Ridge National Laboratory, Computer Scis. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA

Interaction between dislocations and defects plays an important role in mechanical properties. For example, self-interstitial clusters may move to decorate dislocations, thereby lowering their mobility, and dislocations can be hindered by direct intersection with clusters. We use atomic-scale computer simulation to investigate the dynamic interaction between an edge dislocation and glissile clusters with Burgers vector b = ½<111> in iron. When b is parallel to the dislocation glide plane and direct intersection does not occur, clusters can be dragged at high speed by a moving dislocation, and we present a model for drag and dislocation break-away based on the 1-D mobility of clusters. When b is inclined to the glide plane, the cluster can slip to intersect the dislocation, resulting in <100> line segments and hence restriction of slip. The implications of these results for mechanisms of irradiation effects are discussed.

11:00 AM
Cu-Precipitates Hardening in Iron Studied by Atomic-Scale Modeling: *Yuri N. Osetsky*[1]; Roger E. Stoller[2]; David J. Bacon[3]; [1]Oak Ridge National Laboratory, Computer Sci. & Math., PO Box 2008, Oak Ridge, TN 37831 USA; [2]Oak Ridge National Laboratory, Metals & Ceram., Oak Ridge, TN 37831 USA; [3]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

Copper precipitates are formed in Fe-Cu ferritic alloys under irradiation and ageing and cause a significant hardening. Comprehensive discrete dislocation dynamics model to predict mechanical property changes due to microstructure evolution requires a set of rules or reactions which should be defined a priori using detailed knowledge of each dislocation-obstacle reaction. For example, in the particular case of ferritic steel with Cu precipitate hardening the Russell-Brown model is widely used. However, this model, based on simple modulus hardening and constant line tension approximations, is oversimplified. In this paper we present results of atomic-scale modeling of a moving dislocation crossing coherent Cu-precipitates of up to 5nm diameter. We demonstrate the existence of several mechanisms, such as dislocation climb, phase transformation, temperature dependence of interaction mechanism, which affect the critical resolved shear stress but cannot be resolved within the continuum approach. The implementation of atomic-scale mechanisms into continuum dislocation dynamics is discussed.

11:20 AM
Effects of Irradiation of the True Stress-Strain Constitutive Behavior of RPV Steels: *Takuya Yamamoto*[1]; G. Robert Odette[1]; [1]University of California, Dept. of Mechl. Engrg., Santa Barbara, CA 93106 USA

Effects of ˜290°C <0.05 dpa irradiation on true stress-strain s(e) properties of RPV steels were assessed. The database covers a wide range of compositions (Cu, Ni, Mn,..) and irradiation conditions (flux, fluence and temperature). The analysis separated the Luder's-type non-hardening region, with slightly higher eL after irradiation, from the strain hardening ssh(e) = s(e) - the yield stress (sy). The ssh(e) data was fit to a Kock-Mecking saturation dislocation storage and annihilation model. RPV irradiation has only a modest effect on ssh(e), with maximum decreases in the saturated ssh less than 50 MPa. The ssh(e) decreases with larger hardening (Dsy) and irradiation primarily reduces the dislocation storage term, perhaps by suppressing cross slip. In contrast to the effects of much higher dose irradiations, RPV conditions do not produce severe flow localization and reductions in tensile ductility can be predicted based on the combination of Dsy and modest decreases in ssh(e).

11:40 PM
Modeling the Constitutive Behavior of Irradiated BCC Alloys: *Athanasios Arsenlis*[1]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci. Direct., PO Box 808, L-371, Livermore, CA 94550 USA

Performance degradation of bcc alloys in nuclear environments results from the formation of a high number density of nanometer scale irradiation-induced defects. An internal state variable model for the mechanical behavior of such irradiated materials has been developed. The plasticity model includes mechanisms for dislocation density growth and multiplication and for irradiation defect density evolution with dislocation interaction. The model has been modified specifically for bcc alloys by including a temperature dependent dislocation velocity law to account for the inherent lattice resistance, the

material's response to strain localization and the formation of adiabatic shear bands. The model is compared to available experimental data for Mo.

Neutron Diffraction Characterization of Mechanical Behavior: Phase Transformation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Thursday AM Room: 3004
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Mark Daymond, Queen's University, Mechl. & Matls. Engrg., Kingston, Ontario K7L 3N6 Canada; Donald W. Brown, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

8:30 AM Invited
Accelerated Aging in Uranium Niobium Alloys: *Donald W. Brown*[1]; David L. Teter[1]; Daniel J. Thoma[1]; Robert D. Field[1]; Thomas A. Sisneros[1]; Mark A.M. Bourke[1]; [1]Los Alamos National Laboratory, Matl. Sci. & Tech., MS H805, Los Alamos, NM 87544 USA

Uranium 6 weight percent niobium (U6Nb) plays an important role in our aging nuclear stockpile. Niobium is soluble in uranium at high temperature, in the body-centered cubic phase, but not at room temperature. The diffusion of niobium in uranium is rather slow, and if the alloy is quenched at moderate rates a metastable monoclinic phase is produced at room temperature. The properties that make U6Nb attractive are a strong function of the niobium content and are optimized in this metastable phase. Thus, the aging kinetics of the metastable alloy are important to the development of its mechanical behavior with time. This study was aimed at understanding the stability of the alloy through in-situ neutron diffraction measurements during accelerated aging. Samples were heated in-situ to temperatures between 100°C and 400°C and the development of the interatomic spacings monitored over roughly one-day aging times by taking diffraction patterns at 5-20 minute intervals. The observed changes in the lattice parameter are related to the decreased niobium in solution with time at temperature.

8:50 AM
In Situ Neutron Diffraction Measurement of Phase Transformation and Stress Evolution in Al/Al-Cu-Fe Composites During Vacuum Hot Pressing: *Fei Tang*[1]; Iver Eric Anderson[2]; Don W. Brown[3]; Thomas A. Sisneros[3]; Bjorn Clausen[3]; Mark A.M. Bourke[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4515, MS6064, Oak Ridge, TN 37831 USA; [2]Ames Laboratory, Matls. & Engrg. Physics, Rm. 222, Metals Dvlp. Bldg., Ames, IA 50011 USA; [3]Los Alamos National Laboratory, LANSCE, Los Alamos, NM 87545 USA

Unlike the typical tensile residual stresses in the Al matrix of many particulate reinforced Al composites, a significant compressive residual stress was found in the Al matrix of Al/Al-Cu-Fe composites, consolidated by powder metallurgy routes. This unusual compressive stress state appeared to be related to volume expansion of the reinforcement particles that transformed from Al63Cu25Fe12 (quasicrystalline) i-phase to a less-dense (crystalline) omega-phase, during high temperature consolidation. For verification, a through-thickness neutron diffraction technique, including Rietveld analysis, was employed to monitor simultaneously the transformation and stress evolution during vacuum hot (550°C) pressing of such composites with a special compressive load frame and die set. The results revealed that the transformation onset can preceed the applied loading in the powder compact at high temperatures, but that the transformation kinetics were accelerated markedly by the applied compression. This illustrates the opportunities to validate and build materials processing models with in situ neutron diffraction. Supported by USDOE-BES.

9:10 AM
Neutron Diffraction Investigation of NiTiFe Shape-Memory Alloys During Mechanical Loading at Cryogenic Temperatures: *S. B. Shmalo*[1]; C. R. Rathod[1]; T. R. Woodruff[1]; V. Livescu[2]; B. Clausen[2]; M. A.M. Bourke[2]; W. U. Notardonato[3]; Raj Vaidyanathan[1]; [1]University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]NASA, Kennedy Space Ctr., FL 32899 USA

NiTiFe shape-memory alloys exhibit a stress and/or temperature induced phase transformation between cubic, rhombohedral and monoclinic phases. The low hysteresis associated with the rhombohedral or R phase transformation coupled with superior fatigue properties, makes them candidates for actutator applications at low temperatures. This work reports on neutron diffraction measurements of NiTiFe shape-memory alloys during mechanical loading at cryogenic temperatures, with the objective of probing deformation in the R phase. For this purpose, a low temperature loading capability for in situ neutron diffraction measurements was implemented on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory. The in situ diffraction measurements, during loading at 216 K, observed twinning in the R phase prior to a reversible martensitic transformation to the monoclinic phase at higher stresses. Comparisons with room temperature measurements from NiTiFe were also made. This work was supported by grants from SRI, NASA (NAG3-2751) and NSF (CAREER DMR-0239512) to UCF.

9:30 AM
Lattice Strain, Phase, and Texture Evolution During Strain-Induced Martensitic Transformation at a Cryogenic Temperature: *Kaixiang Tao*[1]; James J. Wall[1]; Donald W. Brown[2]; Sven C. Vogel[2]; Mark A.M. Bourke[2]; Hahn Choo[3]; [1]University of Tennessee, Matls. Sci. & Engrg., 318 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

The strain-induced martensitic phase transformation during quasi-static uniaxial compression testing of 304L stainless steel at 300K, 268K, and 203K was investigated with in-situ time-of-flight neutron diffraction using SMARTS at Los Alamos Neutron Science Center (LANSCE). The in-situ neutron diffraction study provides bulk measurements of the evolution of phase fractions, texture, and lattice strains during the austenite to martensite transformation. To achieve a better understanding of the transformation textures and phase fraction evolution, pre-compressed samples with different strain levels were investigated with HIPPO at LANSCE, using its multidetector capabilities. The texture analysis shows that the martensite is highly textured, due to the selective phase transformation. The unique orientation relationships between the austenite and martensite result in the texture development in the parent austenite as well.

9:50 AM
Retained Austenite Stability Investigation in TRIP Steel Using Neutron Diffraction: *Jozef Zrnik*[1]; Petr Lukas[2]; Ondrej Muransky[2]; Petr Sittner[3]; Zbysek Novy[1]; [1]COMTES FHT Ltd., Pilsen 320 13 Czech Republic; [1]COMTES FHT Ltd., Pilsen 320 13 Czech Republic; [2]Institute of Nuclear Physics, Rez near Prague 250 68 Czech Republic; [3]Institute of Physics, Prague 182 21 Czech Republic

The neutron diffraction experiment was focused on transformation behavior of Si-Mn TRIP steel upon thermomechanical treatment. In order to control the TRIP effect in low alloyed TRIP steel it is necessary to understand the stability of retained austenite. The analysis was carried out with the aim to learn whether the neutron diffraction technique can successfully monitor the conditioned austenite transformation process. The relevant information on the course of transformation is extracted from neutron diffraction spectra received. The in-situ neutron diffraction experiment conducted at room temperature was focused on the quantification of retained austenite volume fraction as well as on measurement of the internal stress rising in ferrite and austenite phases during mechanical loading of specimens with different retained austenite volume fraction in complex structure. The transformation behavior of specimens with different volume fraction of retained austenite showed a similar character. The measured difference in retained austenite volume fraction seems to have no substantial effect on deformation behavior of TRIP steel.

10:10 AM Break

10:30 AM
Influence of a Deformation-Induced Phase Transformation on the Mechanical Behavior of a Co-Based Superalloy Studied by In-Situ Neutron Diffraction: *Michael Lee Benson*[1]; Tarik A. Saleh[1]; Peter K. Liaw[1]; Hahn Choo[1]; Don W. Brown[2]; Mark R. Daymond[3];

Xun-Li Wang[4]; Alexandru D. Stoica[4]; Raymond A. Buchanan[1]; Dwaine L. Klarstrom[5]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., Los Alamos, NM 87545 USA; [3]Queen's University, Dept. of Mechl. & Matls. Engrg., Kingston, ON K7L3N6 Canada; [4]Spallation Neutron Source, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [4]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA; [5]Haynes International, Inc., Kokomo, IN 46904 USA

ULTIMET® is a cobalt-based superalloy that is metastable face centered cubic (fcc) at room temperature. The stable hexagonal close packed (hcp) phase forms via a strain-induced phase transformation. In-situ monotonic and cyclic loading experiments were performed at Los Alamos Neutron Science Center (LANSCE) and ISIS, respectively, in order to study the influence of the developing hexagonal phase on the deformation behavior of the material. During monotonic loading, the developing phase was shown to grow at the expense of grains oriented with the {220} plane normal parallel to the axial direction of the specimen. During low cycle fatigue, the new phase formed on the 12th fatigue cycle and increased in volume fraction as fatigue progressed. Also, the intensity of the hcp diffraction peak fluctuated within one fatigue cycle, suggesting some reversibility of the transformation. The study completed here should provide valuable insight into the influence of the developing phase on the mechanical behavior of the material. The author acknowledges the financial support of the National Science Foundation, the Combined Research-Curriclulm Development (CRCD) Progrmas, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI), under DMR-0231320, to the University of Tennessee, Knoxville, with Ms. M. Poats, Dr. L. Clesceri, Dr. W. Jennings, and Dr. C. Huber as contract monitors, respectively. In addition, the financial support of the Tennessee Advanced Materials Laboratory (TAML), with Prof. E. W. Plummer as director, is recognized.

10:50 AM

Influencing Parameters of Martensitic Transformation During Low Cycle Fatigue for Steel AISI 321: *Mirco Grosse*[1]; Dietmar Kalkhof[2]; Markus Niffenegger[2]; Lukas Keller[3]; [1]Paul Scherrer Institut, Spallation Neutron Source Div., Villigen 5232 Switzerland; [2]Paul Scherrer Institut, Nuclear Energy & Safety, Villigen 5232 Switzerland; [3]Paul Scherrer Institut, Lab. of Neutron Scattering, Villigen 5232 Switzerland

The present investigations include the study of the influence of thermo-mechanical manufacturing conditions and of low cycle fatigue parameters, (load amplitude, load frequency, cycle number and fatigue test temperature) on content and texture of the martensite. The material investigated was the austenitic stainless steel AISI 321. This steel is commonly used as material for pipes of cooling circuits in nuclear power plant. The content of martensite after the LCF test and the martensite texture were determined by neutron diffraction using the DMC diffractometer at SINQ/PSI (Switzerland). Whereas no influence was found for the load frequency the martensite content linear increases with increasing cycle number and load amplitude. It decreases exponentially with increasing LCF test temperature. The thermo-mechanical manufacturing conditions show a strong influence. The amount of martensite formed during LCF is much higher after cold rolling than after solution annealing as final manufacturing process.

11:10 AM

On-Line Low Cycle Stress Rig Neutron Diffraction Study of a Martensite Phase Transformation in Stainless Steel AISI 321 Ad Interim High Cycle Fatigued: Yu. V. Taran[1]; *M. R. Daymond*[2]; E. C. Oliver[2]; J. Schreiber[3]; [1]Joint Institute for Nuclear Research, Frank Lab. of Neutron Physics, Dubna, Moscow Region 141980 Russia; [2]Rutherford Appleton Laboratory, ISIS, Chilton, Didcot, Oxon OX11 0QX UK; [3]Fraunhofer Institute for Nondestructive Testing, EADQ, Dresden D-01326 Germany

Earlier we have described results of the tensile test of samples from a steel AISI 321 ad interim cycle fatigued at frequency of 5 Hz (HCF-samples) and 0.5 Hz (LCF-samples). In HCF-samples the elastic constants of austenite and martensite were found out to be strongly different as against LCF-samples in which they are close. More over, the ratio of axial and transverse elastic constants for martensite in the HCF-samples is almost twice that observed 0.28-0.30 in austenite and in both phases of the LCF-samples. The mechanism for this unusual behaviour is unclear, but may be linked to the shape of the martensite. One of HCF-samples was anew tested using the in situ stress rig in : 1) low cycle mode at a frequency of 0.1 Hz to increase the fatigue level, and 2) a quasistatic mode to measure the applied stress-elastic strain responses of both phases. The HCF-LCF-transformation was studied at increasing martensite fraction from 5 to 19 Wt. %. It is exerted a week influence on mechanical properties of the sample.

11:30 AM

Spatially Resolved Neutron Diffraction Measurement of Heterogeneous Stress-Induced Phase Transformation in a Superelastic NiTi Disc: *C. R. Rathod*[1]; S. B. Shmalo[1]; B. Clausen[2]; M. A.M. Bourke[2]; Raj Vaidyanathan[1]; [1]University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Previous work has reported on the use of neutron diffraction to probe predominantly homogeneous stress-induced transformations in NiTi associated with uniaxial stress states (Vaidyanathan et al, Acta Mater 1999). In the following, we report on neutron diffraction measurements from a NiTi disk specimen loaded laterally in compression and associated with a macroscopically heterogeneous stress state. Measurements were performed on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory in a spatially resolved mode. Neutron spectra confirm the presence of both monoclinic and cubic phases of NiTi, with the respective volume fractions depending on the location of the measurements (and the corresponding stress state). The neutron diffraction measurements of the strain, texture and phase volume fraction offer insight into accommodation mechanisms as the cubic and monoclinic phases co-exist in a macroscopically heterogeneous stress state. This work was supported by a grant from NSF (CAREER DMR-0239512) to UCF.

Powder Metallurgy Research and Development in the Transportation Industry: Nano-Materials, Intermetallics, Amorphous and Composites - P/M Developments

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Powder Materials Committee

Program Organizer: James W. Sears, South Dakota School of Mines & Technology, Additive Manufacturing Laboratory, Rapid City, SD 57701 USA

Thursday AM
February 17, 2005

Room: 3008
Location: Moscone West Convention Center

Session Chair: James W. Sears, South Dakota School of Mines & Technology, Additive Mfg. Lab., Rapid City, SD 57701 USA

8:30 AM

Preliminary Evaluation of Equal Channel Angular Extrusion Followed by Combustion Synthesis of TiAl: *K. Morsi*[1]; Sandeep Goyal[1]; [1]San Diego State University, Mechl. Engrg., 5500 Campanile Dr., San Diego, CA 92182 USA

Titanium aluminides are one of the most promising intermetallics today. They have previously been processed using a variety of routes to generate different microstructures. Recently, severe plastic deformation by equal channel angular extrusion (ECAE) has been found to be a viable method for producing ultrafine materials in bulk form. The work presented in this paper involves preliminary investigations of ECAE of elemental titanium and aluminum powders (one pass) followed by combustion synthesis. The effect of processing (ECAE vs. uniaxial pressing) on the reaction characteristics, product homogeneity and microstructure is presented.

8:55 AM

Plasma Synthesis of Ultrafine AlTi-TiC Composites: *Lirong Tong*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35401 USA

A novel in_situ processing technique of ultrafine AlTi-TiC composite by thermal plasma was developed to investigate synthesis of ultrafine AlTi-TiC composite powders. A basic understanding of thermodynamics of synthesis of ultrafine AlTi-TiC composite powders is essential. Thermodynamic analysis was performed to predict conditions of synthesis and recovery rate of ultrafine AlTi-TiC composite powders. The paper emphasizes on the investigation of feeding rate, input power, mole ratio and other process parameters of synthesis of ultrafine AlTi-TiC composite powders by thermal plasma. The experimental results showed that ultrafine AlTi-TiC composite powders can be synthesized and the average size of ultrafine AlTi-TiC composite powders was less than 200 nm.

9:20 AM
Powder Extrusion of Nanostructured Aluminum Alloy Created by Machining: *Balkrishna C. Rao*[1]; Srinivasan Chandrasekar[1]; Kevin P. Trumble[1]; W. Dale Compton[1]; [1]Purdue University, Ctr. for Matls. Procg. & Tribology, Coll. of Engrg., 315 N. Grant St., W. Lafayette, IN 47907-2023 USA

An exploratory study has been made of extrusion of nanostructured Aluminum 6061-T6 particulate derived from machining chips. For this purpose, chips with nanocrystalline microstructure were created by machining bulk Al6061-T6 alloy. The hardness of the chips was found to be greater than that of the same alloy produced by Equal Channel Deformation Processing. The chips were converted into particulate with an average particle size of ~75 micrometers by attrition milling. Monolithic bulk forms of the 6061-T6 were created by axisymmetric cold extrusion of the particulate through a conical die. Additionally, bulk composites were produced by cold extrusion of mixtures of the 6061-T6 particulate and commercially pure aluminum particulate. Microstructure, mechanical and physical properties of the extruded bulk forms are characterized and compared with those of conventionally processed Al6061-T6 alloy. Some of the attractive advantages of the ultrafine-grained Al6061-T6 extruded samples are highlighted with reference to the results. Implications to up-cycling of machining chips produced in discrete product machining operations are also discussed.

9:45 AM
Compressive Behavior of a Novel Aluminum Metal Matrix Composite: *Jichun Ye*[1]; Bing Q. Han[1]; Julie M. Schoenung[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., 3118 Bainer Hall, One Shields Ave., Davis, CA 95616 USA

Al 5083 and B4C were cryomilled in liquid nitrogen to form a composite of B4C particles in nanocrystalline Al. The cryomilled composite powders were blended with coarse-grained Al 5083. The blended powders were consolidated using cold isostatic pressing and extrusion. The resultant composite is a complicated materials system with three phases, 10 wt. % B4C, 50 wt. % coarse-grained Al 5083 and the balance nanocrystalline Al 5083. B4C and nanocrystalline Al contribute to the high strength, while coarse-grained Al was introduced to achieve ductility because of the dislocation activity it provides. Compression tests were conducted on this composite at various temperatures to examine its mechanical behavior. The results show that this composite exhibits an extremely high yield strength of 1150 MPa at room temperature with 2.5% elongation. The microstructure of this tri-modal composite was investigated, and the relationship between the excellent mechanical behavior and the microstructure are discussed.

10:10 AM
Effects of Non-Equilibrium Phases on Mechanical Properties of Al Composites Processed by Mechanical Alloying: *Kwang Seon Shin*[1]; *Woo Kil Jang*[1]; [1]Seoul National University, Sch. of Matls. Sci. & Engrg., San 56-1 Shinrim-dong Kwanak-gu, Seoul 151-742 Korea

The effects of non-equilibrium phases on the microstructures and mechanical properties of Al composites were investigated in the present study. The non-equilibrium phases such as quasicrystalline phase and amorphous powders were utilized as strengtheners in this study. Powders of the quasicrystalline phase were produced by casting and subsequent milling. The amorphous powders were produced by the gas atomization method. Mechanical alloying process was utilized in order to produce appropriate powders for the Al matrix composites reinforced with either quasicrystalline or amorphous phases. The composite powders were canned in the Al can and extruded at elevated temperatures. The microstructures of the extrusions were examined by OM and SEM. In order to investigate the mechanical properties of the extrusions, hardness and compression tests were carried out. It was found that the mechanical properties and thermal stability of the Al composites reinforced with quasicrystalline phase or bulk amorphous phase significantly increased compared with those of the conventional Al matrix composites.

10:35 AM
High Cycle Fatigue Studies on Devitrified Amorphous Aluminum Alloys: S. Qi[1]; *P. Wesseling*[1]; J. J. Lewandowski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA

Devitrified amorphous aluminum rods produced by extrusion of amorphous aluminum powders were used in this investigation. Deformation processing of the amorphous aluminum alloy produced a nanocomposite structure possessing very high bend strength and non-zero ductility with ductile fracture as the fracture mode. Smooth bend bar specimens were prepared from these rods to conduct high cycle fatigue tests over a wide range of stresses and test temperatures. The high cycle fatigue behavior will be compared to the behavior of other aluminum alloys.

Rare Earths: Science, Technology and Applications V: Rare Earths

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Renato G. Bautista, University of Nevada, Department of Chemical and Metallurgical Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada, Chemical and Metallurgical Engineering, Reno, NV 89557 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA

Thursday AM — Room: 3001
February 17, 2005 — Location: Moscone West Convention Center

Session Chairs: Scott W. Jorgensen, General Motors Corporation, Cheml. & Environml. Scis. Lab., Warren, MI 48090-9055 USA; Dhanesh Chandra, University of Nevada, Cheml. & Metallurgl. Engrg., Reno, NV 89557 USA

8:30 AM Keynote
The Influence of Rare Earths on the Corrosion Resistance of High Temperature Materials: *David A. Shifler*[1]; [1]Naval Surface Warfare Center, Marine Corrosion Branch, 9500 MacArthur Blvd., W. Bethesda, MD 20817-5700 USA

Selected reactive elements such as zirconium, hafnium, yttrium, and rare earths or the presence of selected oxide dispersions added to selected alloys or coatings often have shown marked improvement of the oxidation resistance, corrosion resistance, and scale adhesion. These effects may include (1) more rapid formation of a continuous oxide scale, (2) reduction in the oxidation rate, (3) improved scale adherence, (4) a change in the scale-forming reaction location from the scale-gas interface to scale-alloy interface. Reactive elements improve the oxidation resistance and high-temperature corrosion of many high temperature alloys, particularly if the resistance is dependent on oxide formation. This paper will provide an overview of the bases by which rare earths and other reactive elements influence the oxidation and high temperature corrosion resistance of materials.

8:50 AM Invited
Production of Value Added Rare Earths from Monazite by Solvent Extraction: *L. N. Maharana*[1]; V. R. Nair[1]; [1]Indian Rare Earths Limited, Rare Earths Div., Udyogamandal-683 501, Kerala State India

The Rare Earths Division of Indian Rare Earths Limited (IREL) started processing of monazite in 1952. IREL is one of the pioneers in the processing of. monazite. Over the years IREL perfected the monazite processing technology and is producing rare earth compounds, trisodium phosphate, thorium and uranium chemicals at present. In recent times IREL has focussed its attention to produce value added high purity rare earth chemicals to meet the demand for these materials in the international and indigenous markets. The R&D laboratory of IREL has developed several processes for the separation of rare earths based on solvent extraction technology. These solvent extraction processes developed were scaled up and implemented on plant scale. The solvent extraction separation processes employed at IREL include separation of cerium, neodymium and samarium by using PC-88A(mono-2-ethyl hexyl ester of mono-2-ethyl hexyl phosphonic acid) and the separation of yttrium using the quarternary ammonium compound Aliquat-336(tri octyl methyl ammonium chloride) as the extractants. The details of the solvent extraction process development and flow sheets employed by IREL will be presented in this paper.

9:10 AM
Novel Functional and High Temperature Applications of Nanocrystalline Rare Earth Phosphate Coatings and Composites: *K. G.K. Warrier*[1]; K. Rajesh[1]; R. Rohith[1]; V. R. Nair[2]; [1]Regional Research Laboratory (Council of Scientific & Industrial Research), Ceram. Tech. Div., Thiruvananthapuram 695 019 India; [2]Indian Rare Earths Limited, Udyogamandal India

The range of rare earth phosphates has been identified for their high temperature phase stability and high melting points. Non reactivity with certain other ceramic materials such as alumina, use of the rare earth phosphate as an interface in alumina matrix composites,

machinability of the alumina-rare earth composites, possible applications as catalysts and the luminescent and optical properties have been subject of investigation over the last decade. Synthesis and characterization of nano crystalline rare earth phosphate has been of recent interest in view of the wide possibilities of this range of materials for various applications. The present work relates to the synthesis of 30-50 nm size particles of lanthanum and cerium phosphates by sol-gel colloidal technique starting from respective nitrates. The gel to phosphate transformation, the high temperature stability, densification characteristics at as low temperatures as 1400°C are presented. The electrical properties of these phosphates indicate low dielectric constant and loss factors. Nano crystalline sintered rare earth phosphate also indicates high temperature deformation behaviour. Lanthanum phosphate coatings on various substrates have been developed and certain features of such coatings have also been investigated. The paper covers an overview of the range novel characteristics of the rare earth phosphates. Experimental details of synthesis in nano crystalline phosphate and certain novel findings are presented. The possibility of practical applications of this range of materials for a variety of applications is highlighted.

9:30 AM

Ductile Intermetallic Compounds! The Rare Earth RM B2 CsCl-Type Intermetallics: *Karl A. Gschneidner*[1]; Alan M. Russell[1]; Alexandra O. Pecharsky[1]; Zhehua Zhang[1]; James R. Morris[1]; Tom A. Lograsso[1]; C. H. Chester Lo[1]; Yiying Ye[1]; [1]Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

A number of the rare earth intermetallic B2 CsCl family of compounds (~100 members) exhibit unprecedented large ductilities at room temperature, up to ~25% elongation at failure. These compounds are fully ordered, stoichiometric, and in general line compounds. To date quantitative measurements have been made on RCu (R = Y, Dy and Er), YAg and (Tb<0.88>Dy<0.12>)Zn. Of the five compounds only the RZn phase is brittle. YAg is the most ductile compound examined to date with an elongation of 27% at failure with a maximum tensile strength of ~150 MPa (comparable to a typical solid solution aluminum alloy). Preliminary measurements indicate ten other RM phases are also ductile RCu (R = Gd, Ho, Dy), RAg (R = Ce, Nd and Er), YRh, YIn, ErIr and ErAu. The results of mechanical property tests and ab initio calculations, including band structures and unstable stacking fault energies, will be discussed.

9:50 AM

Microstructural Investigation of Mixed Rare Earth Iron Borides Processed Via Melt Spinning and Gas Atomization: *Nick Buelow*[1]; Iver E. Anderson[2]; William McCallum[2]; Matthew Kramer[2]; Wei Tang[2]; Kevin W. Dennis[2]; [1]Iowa State University, Matls. Sci. & Engrg., 221 Metals Dvlp., Ames, IA 50014 USA; [2]Ames Laboratory, Matls. & Engrg. Physics, Ames, IA USA

Novel mixed rare earth iron boride (MRE-Fe-B) permanent magnet (PM) alloy combines Nd, Y, and Dy with Fe, B, and other substitutions to help stabilize the temperature dependent magnetic properties remenance, coercivity, and BH_{max}. MRE-Fe-B has demonstrated stabilization of the temperature dependent magnetic properties beyond 200°C; whereas, $Nd_2Fe_{14}B$ based PMs have magnetic properties that deteriorate rapidly at ~125°C. The challenge has been to adjust alloy designs and annealing treatments to convert the developments made with melt spun ribbons into gas atomized powders with fine spherical particulate form, preferred for polymer bonding. Synthesis of an optimum fine uniform microstructure for the $MRE_2Fe_{14}B$ alloys is being accomplished through melt spinning and high pressure gas atomization. Both processes can boast cooling rates of ~10^{6o}C/s which promote the formation of similar amorphous and nanoscaled structures, but with differences in the nucleated phases and growth orientations. Support from USDOE-EE is acknowledged through contract no. W-7405-Eng-82.

10:10 AM Break

10:20 AM

Crystal Growth of RE-Si-Ge Magnetocaloric Compounds: *Deborah L. Schlagel*[1]; Thomas A. Lograsso[1]; Alexandra O. Pecharsky[1]; Juraci A. Sampaio[1]; [1]Ames Laboratory, Matls. & Engrg. Physics Prog., Ames, IA 50011 USA

Single crystals of RE5(SixGe1-x)4 have been prepared by both the Bridgman and by the tri-arc crystal pulling method. Bridgman grown crystals, grown in welded tungsten crucibles, contained large grains but were severely cracked due to alloy/crucible thermal mismatch. The Tri-arc method had the advantage of being crucible-less and the usable crystal size has been increased with this method. Overall, the bulk crystal solidified in the monoclinic phase with a slight increase in Si content and decrease in Ge content as the growth proceeded. This increase in Si:Ge ratio resulted in a slight increase in lattice parameter of the monoclinic phase and an increase in the magnetostructural transformation temperature of ΔT = 10K. AC susceptibility and differential scanning calorimetry (DSC) measurements indicated a small fraction of orthorhombic phase was present throughout the ingot which accounts for the decrease in MCE values along the length of the ingot.

10:40 AM

Amorphization and Nanocrystallization of the Intermetallic Compound $TbFe_2$ by Ball Milling: *Jiahong Zhu*[1]; Zigui Lu[1]; Chain T. Liu[2]; Joe A. Horton[3]; [1]Tennessee Technological University, Dept. of Mechl. Engrg., 115 W. 10th St., Box 5014, Cookeville, TN 38505 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831 USA

The rare earth-transition metal system based on $TbFe_2$ possesses huge magnetostriction. However, due to its large magnetocrystalline anisotropy, a high magnetic filed is required to obtain the desired magnetostrictive strains. One way to reduce the magnetostrictive anisotropy and thus the actuation field is to refine the grains to the nanometer scale. In this paper, we tried to produce the nano-sized grain structures in binary $TbFe_2$ via amorphization by high-energy ball milling followed by nanocrsytallization treatment. To separate the different stages of ball milling process, the formation of an amorphous phase starting from an intermetallic compound was investigated. Experimental conditions were described under which the intermetallic compound $TbFe_2$ became amorphous. The crystallization behavior of the amorphous phase was studied using a DSC apparatus. The optimal conditions to generate nanocrystalline materials were determined. The magnetostrictive properties of amorphous and nanocrystallized $TbFe_2$ were evaluated by the standard strain gauge method.

11:00 AM

Characterization of R5(SixGe1-x)4 Alloys, Where R is Gd, Tb, Dy and Er: *Ozan Ugurlu*[1]; Scott L. Chumbley[1]; Deborah L. Schlagel[2]; Thomas A. Lograsso[4]; Alexandra O. Pecharsky[3]; [1]Iowa State University/Ames Laboratory, Matls. Sci. & Engrg., 206 Wilhelm Hall, Ames, IA 50011 USA; [2]Ames Laboratory, 110 Metals Dvlp., Ames, IA 50011 USA; [3]Ames Laboratory, 239 Spedding Hall, Ames, IA 50011 USA; [4]Ames Laboratory, Matls. Sci. & Engrg., 111 Metals Dvlp., Ames, IA 50011 USA

Bulk microstructures of R5(SixGe1-x)4 alloys, where R is Gd, Tb, Dy and Er, have been examined using transmission (TEM) and scanning (SEM) electron microscopy. The microstructure of all alloys consisted of large grains with linear features present within the grains. The features in general are on the order of 1 micron or less in width and hundreds of microns in length, and are seen in all alloys independent of their crystal structure. Single crystals of Gd5Si2Ge2 have been used to study the crystal orientation of the linear features by using a combination of back-reflection Laue x-ray diffraction and SEM. Systematic tilting experiments of a single crystal of Gd5Si2Ge2 with controlled polishing angles showed that these linear features grow in specific directions as thin plates. Energy Dispersive Spectrometry (EDS) results revealed that the plates have a composition approximating R5(SixGe1-x)3 type phases in all the alloys systems studied, and TEM studies using electron diffraction and high resolution TEM confirmed this. These results verify an earlier study that suggested the features might possibly be a Widmanstatten structure that forms during sample preparation.

11:20 AM

Structure/Properties Relationships in the Pr2Ni1.5-xSi2.5+x Alloys Series, Where $0 \le x \le 1$: *Alexandra O. Pecharsky*[1]; Karl A. Gschneidner[1]; Kevin W. Dennis[1]; R. William McCallum[1]; [1]Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

The $Pr_2Ni_{1.5-x}Si_{2.5+x}$ alloys, where $0 \le x \le 1$, adopt the AlB2 structure type with the P6/mmm space group symmetry. These alloys exist over an extended solid solution at 33.3 at.% of Pr and a random occupation of the boron sites by Ni and Si. This crystal structure belongs to one of the simplest and most extended classes of structure types which consists of a trigonal prismatic arrangement of the large atoms. The trigonal prisms form the columns with the Pr atoms in the corners and the Ni/Si atoms located in the center of each trigonal prism. The axes of the trigonal prisms are collinear and their bases are coplanar. The specific heat and magnetic measurements of the as-prepared alloys indicate antiferromagnetic ordering at low temperatures (~5K), which depends upon the Ni/Si ratio. The samples appear to undergo a metamagnetic transition at ~10 kOe, where the critical field also depends upon the Ni/Si ratio.

11:40 AM Invited
Structure and Magnetocaloric Properties of Fe-Doped HoTiGe Compound: *Ben Baumgold*[1]; V. Provenzano[1]; A. J. Shapiro[1]; R. D. Shull[1]; [1]National Institute of Standards and Technology, Magnetic Matls. Grp., 100 Bureau Dr., MS-8552, Gaithersburg, MD 20899 USA

The structure and the magnetocaloric properties of the iron-doped HoTiGe compound have been studied by means of SEM, EDS, XRD, and SQUID magnetometry. Consistent with earlier studies by Tegus and his co-workers on the iron-free compound (1-3), the iron-containing compound exhibited a highly textured microstructure and a antiferromagnetic-to-paramagnetic phase transition near 90 K with the attendant magnetocaloric effect peak centered at the same temperature. However, the iron-doped alloy contained three additional minor phases not present in the HoTiGe compound, two of which were titanium and iron-rich, while the third phase was Ho-rich. The corresponding magnetization versus temperature data clearly showed the presence of two additional magnetic phase transitions, occurring at about 2 K and at 10 K. Correspondingly, the magnetic entropy change, -DS, versus temperature plots, computed from the isothermal magnetization data, showed the presence of magnetocaloric effect peaks, also centered at about 2 K and 10 K, whose respective magnitudes are factors of 6 and 2 larger than that for the 90 K peak. It is believed that the minor phases present in the iron-doped alloy give rise both to the two additional low temperature magnetic phase transitions and to the coincident DS peaks. These results have positive implications for cryogenic magnetic refrigeration applications, including detector cooling in space-based systems and hydrogen liquefaction, which will be important for a future hydrogen-based economy.

Recycling - General Sessions: Post-Consumer Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, LMD/EPD-Recycling Committee
Program Organizer: Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA

Thursday AM Room: 2011
February 17, 2005 Location: Moscone West Convention Center

Session Chair: Michael L. Free, University of Utah, Dept. of Metall. Engrg., Salt Lake City, UT 84112 USA; Markus A. Reuter, Delft University of Technology, Delft 2628 The Netherlands

8:30 AM
Differential Recycling Fees for Containers Made of Multiple Materials: *Esher Hsu*[1]; Chen-Ming Kuo[2]; [1]National Taipei University, Dept. of Stats., 67 Sect. 3, Min-Sheng E. Rd., Taipei 104 Taiwan; [2]I-Shou University, Dept. of Mechl. Engrg., 1 Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

In Taiwan, manufacturers and importers have responsibility to pay the recycling fees for the packaging containers based upon the recycling rates and the amount produced or imported. The containers made of multiple materials may cause more impact on environment and recycling cost than these of simple materials. This paper aims to explore the feasibility of differential rates for containers with multiple materials. A sampling survey is employed to estimate environmental impact, reused value, and recycling cost, which are further used to calculate the differential recycling rates; whereas the economic impact is also explored to evaluate its feasibility. Study results show that charging extra fee for the containers with multiple materials based upon the additional recycling cost of complicate materials is a good way to lead producers to develop more environmentally friendly products with simple material, but this could also cause economic impact more or less.

8:55 AM
The Calculation of the Recycling Rate of Cars: *Markus Andreas Reuter*[1]; Antoinette van Schaik[1]; [1]TU Delft, Applied Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

This paper will provide an overview of the present methods used for the calculation of recycling rates also that provided by the ISO norm. An alternative improved method will be discussed and presented based on the inclusion of statistical distribution functions. The method will be illustrated by detailed calculations to illustrate the difference between present superficial and non-rigorous methods and the method discussed in this paper. It will also suggest that the whole chain of recycling has to be controlled in order to arrive at suitable recycling rate numbers which have enough basis to satisfy car industry during the type approval of vehicles for example but also to provide the insight and numbers required by the recycling industry to evaluate their own processes.

9:20 AM
Identifying Economic and Scrap Reuse Benefits of Light Metals Sorting Technologies: Preston P. Li[1]; Sigrid Guldberg[2]; Hans Ole Riddervold[2]; *Randolph E. Kirchain*[1]; [1]Massachusetts Institute of Technology, Matls. Sys. Lab., Rm. E40-421, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]Hydro Aluminum, Drammensveien 264, N-0240, Oslo Norway

The changing pattern of aluminum scrap usage has created material reuse challenges for the industry. For instance, mixed scraps consisting of wrought and cast alloys often cannot be directly re-melted and reused due to compositional incompatibility. Various new sorting technologies promise to address these challenges. It is critical to understand how, when, and to what extent sorting should be applied in different circumstances. This paper examines the use of linear programming methods to identify economically efficient sorting strategies and their impact on scrap usage. Economic efficiency was tested for various states of scrap material supply, finished good demand, sorting technology type, and sorting performance. The model can be used to identify optimized specific sorting schemes. The overall goal is to support industry decision-making regarding the application of sorting technologies to increase scrap use and lower production costs.

9:45 AM
A Comparison of the Modelling of Comminution and Liberation in Minerals Processing and Shredding of Passenger Vehicles: *Antoinette van Schaik*[1]; Markus Andreas Reuter[1]; [1]Delft University of Technology, Mijnbouwstraat 120, Delft 2628 RX The Netherlands

The material connections and combinations in car design determine the particle size reduction and the degree of liberation during shredding, which affects the composition of the intermediate recycling streams and the efficiency of physical separation. The quality of intermediate recycling products is important for optimizing material recovery in metallurgical processing, to achieve the high recycling rates required by the European Union. Modelling of liberation is extensively applied in classical minerals processing. However, modelling of liberation for consumer goods differs fundamentally from minerals processing. This paper investigates the fundamental differences between comminution and liberation in minerals processing and shredding modern consumer goods, such as the car. From this research, it will become clear to what extent classical minerals processing approaches can be used to model the design and shredding of the car. A clear direction will be given for future modelling and optimisation work on the recycling of end-of-life vehicles.

10:10 AM Break

10:40 AM
Removal and Recovery of Solder from Printed Circuit Boards: Robert W. Gibson[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The quantity of electronic scrap is growing almost exponentially, and this needs to be recycled into the constituent metals and components rather than landfilled. This paper will concentrate on the removal of solder from both complete printed circuit boards and shredded boards. The leachant used was fluoroboric acid with Ti(IV) added as an oxidant. It was found that the solder was dissolved in the leachant and could be subsequently recoved by electrowinning. For complete printed circuit boards, the electronic components either fell off the boards or could be eaily removed, and were found to have been completely unaffected by the leachant.

11:10 AM
Feasibility Study on the Recycling of Cadmium-Telluride Photovoltaic Modules: *Wenming Wang*[1]; *Vasilis M. Fthenakis*[1]; [1]Brookhaven National Laboratory, Environml. Scis. Dept., Bldg. 830, Upton, NY 11973 USA

Cadmium and selenium are used to manufacture thin film photovoltaic modules. The environmental concerns associated with these heavy metals have prompted recycling of the PV modules. A hydrometallurgical processing route has been investigated at the Brookhaven National Laboratory. Cadmium and tellurium were stripped from PV module substrates by leaching with hydrogen and dilute sulfuric acid in a tumbling machine. Both were readily solubilized with low strength acid (1.0M H2SO4) at ambient temperature. Leaching the PV module scraps completely extracted both cadmium and tellurium. In the separation

step, cation exchange resin completely separated cadmium from tellurium-rich leach liquor of 0.5 M H2SO4. Ion exchange column studies showed that elution of the resin with high strength H2SO4 generated concentrated cadmium solution, from which cadmium was recovered by electrowinning.

11:35 AM
Recovery of Metals from Dilute Solutions by Pulsed Electrodeposition: *Michael L. Free*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Many industrial solutions contain low levels of dissolved metals that can be recovered and recycled into useful products. This study shows the effects of various process parameters on the recovery of metals from dilute solutions using pulsed electrodeposition.

Superalloys and Coatings for High Temperature Applications: Superalloys - III

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Thursday AM — Room: Nob Hill A/B
February 17, 2005 — Location: San Francisco Marriott

Session Chairs: Roger C. Reed, University of British Columbia, Vancouver, BC V6T 1Z4 Canada; Qiang (Charles) Feng, University of Michigan, Dept. of Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA

8:30 AM
Comparison of Low Coefficient of Thermal Expansion (CTE) Nickel Alloys Containing 12.5% Chromium: *David E. Alman*[1]; Paul D. Jablonski[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321 USA

Research aimed at formulating low CTE nickel-base superalloys for intermediate-temperature solid oxide fuel cells (SOFCs) is reported. Alloys based on the composition Ni-12.5wt % Cr were modified with either W or Mo to lower CTE to on the order of 12.5 x 10-6 C-1. Mn was added to impart the formation of an outer Cr-Mn spinel and thus minimize chromium vaporization in moist environments. Linear variable differential transducer (LVDT) based dilatometer measurements were used to determine the thermal expansion of the alloys in accordance with ASTM standard E-228-85. Characterisation was performed by XRD and EDS and the results compared to thermodynamic predictions. Oxidation tests (in dry and wet air) were conducted at 750 and 800°C. The results were compared to the behavior of a commercial Fe-22Cr (Crofer 22APU) and Ni-22Cr (Haynes 230) alloys. The oxidation resistance of the low CTE-alloys was intermediate between Crofer 22APU and Haynes 230.

8:55 AM
High-Cycle Corrosion-Fatigue Investigation of the Nickel-Based Alloy, Haynes 2000: *Rejanah V. Steward*[1]; Ray A. Buchanan[1]; Peter K. Liaw[1]; Douglas E. Fielden[1]; Dwaine L. Klarstrom[2]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., Kokomo, IN 46904-9013 USA

Generally, deterioration of fatigue properties is caused by preferential corrosion of the distorted metal due to rapid dissolution of atoms exposed by slip band formation, which creates a high stress concentration and subsequent crack initiation. Metals that are susceptible to corrosion are intuitively expected to be susceptible to corrosion fatigue. In this investigation, the corrosion behavior of Haynes C2000, Ni-23Cr-16Mo-1.6Cu weight % (wt. %), immersed in aerated and deaerated 3.5 wt.% sodium chloride solutions, potentially detrimental metal-chloride solutions, and simulated mechanically-perturbed environments have been studied to substantiate corrosion-fatigue failure suppositions. High-cycle fatigue tests were conducted in air at a frequency of 20 Hz. X-ray photoelectron spectroscopy characterizes the alloy as having a predominantly Cr2O3 protective film, and the repassivation kinetics appears to be immediate upon film breakage. The fracture morphology is that of a faceted cleavage nature. The corrosion fatigue results are expected to be lower than those observed in air.

9:15 AM
Effect of Carbon Additions on the Mechanical Properties of a Single Crystal Ni-Base Superalloy: *Elyssa Renee Cutler*[1]; Khalid A. Al-Jarba[1]; Gerhard E. Fuchs[1]; [1]University of Florida, Matls. Sci. & Engrg., PO Box 116400, Gainesville, FL 32611 USA

Carbon was added to a model single crystal Ni-base alloy, LMSX-1, to reduce casting defects. Mechanical testing including creep, tensile and fatigue tests were carried out. Fractography revealed distinct changes in behavior according to carbon level. Samples were tested in both a heat treated and overaged condition. Though the increases in carbon content improved castability, creep lives were drastically reduced. No steady state creep rates or incubation times were seen in any samples. Carbide compositions and morphologies were altered by exposure during creep testing. Microstructural changes caused by increasing carbon additions were detrimental to mechanical properties. A preliminary Larsen-Miller curve was developed.

9:35 AM
Electrical Monitoring of Microstructural Fluctuations in Waspaloy as a Function of Thermal Exposure: *Siva Kumar*[1]; Rosario A. Gerhardt[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Microstructural evolution in Waspaloy, an age-hardenable nickel-base superalloy, was investigated as a function of thermal exposure to 1400ºF for times up to 2502 hours. Specimens sampled at several intermediate exposure times were characterized using impedance spectroscopy, optical microscopy, SEM, XRD and stereology. Repetitive upward and downward trends were noted in the lattice parameter and the composition in all cases. Similar fluctuations were apparent in the volume fraction (Vv) of the primary g¢ precipitates with progressive thermal exposure. An inverse correlation between Vv and Y-max, an electrically derived parameter was obtained. Since the average g grain size also varied considerably during the length of the experiment, solutionizing and aging experiments are ongoing in order to determine if the fluctuations in the impedance response can be quantitatively ascribed to the changes in grain size or the precipitate population of the alloy or both.

9:55 AM Break

10:25 AM
Development of Functionally Graded Materials for Manufacturing Tools and Dies and Industrial Processing Equipment: Stanley M. Howard[1]; *Sudip Bhattacharya*[1]; James W. Sears[2]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA

The goal of this investigation was to produce improved dies for the metal casting, forging and glass forming industries by laser depositing functionally graded materials (FGM) on the working surfaces of the tools and dies. Typically, inexpensive H-13 or other grade tool steels are used for tools, dies and industrial process equipment, but they exhibit numerous failure modes including heat checking, soldering, fatigue, chipping and cracking, loss of hardness and corrosion. Longer wearing but prohibitively expensive materials are available for dies. In this work, the low cost of H-13 was combined with the high performance of expensive mold surfaces. The major features of interest in the graded materials are near-net shape, surface durability, and surface finishing. The FGM surfaces were produced using a laser powder deposition system employing a 3 KW Nd:YAG laser.

10:45 AM
Oxidation Resistance of Titanium Aluminides and Nickel Aluminides Intermetallic Compound Coatings on Inconel 738: *M. Reza Bateni*[1]; Morteza Zandrahimi[2]; Jerzy A. Szpunar[1]; [1]McGill University, Mining, Metals & Matls., 3610 Univ. St., M. H. Wong Bldg., Montreal, Quebec H3A2B2 Canada; [2]Shahidbahounar University, Engrg. Dept., Matls. Engrg. Grp., Jomhourieslami Bolv., Kerman Iran

In this research, attempts have been made to develop Al-Ti and Ni-Al intermetallic compound coatings on the surface of Inconel 738. The coatings were applied through pack cementation and simultaneous diffusion annealing processes. Scanning-electron microscopy

(SEM), energy-dispersive X-ray spectrometry (EDS), thermal gravimetric analysis (TGA) and grazing angle X-ray diffraction were used for evaluating the coating. It was demonstrated that the pack cementation technique could be used to develop TiAl3 and AlNi intermetallic compounds coating on Inconel 738 substrate. When pack cementation technique is based on a halide-activated pack with aluminum and titanium powders, the coating consists of TiAl3 intermetallic compound phase. Whereas, by pack aluminization of Inconel 738, the coating consists of AlNi intermetallic compound phase. It has found that the presence of intermetallic compound coatings on the surface, increase the oxidation resistance and the loss of elements from the protective coating, replenished by the interior layers, through interdiffusion.

11:05 AM
The Influence of Boron/Carbon Alloying on the Corrosion Behavior of NiSi19Nb3 Superalloy: Dong-Yih Lin[1]; Jen-Shen Hsu[1]; Shau-Shan Yen[1]; *Shian-Ching Jason Jang*[1]; Chen-Ming Kuo[2]; Shih-Jeh Wu[2]; [1]I-Shou University, Dept. of Matls. Sci. & Engrg., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan; [2]I-Shou University, Dept. of Mechl. Engrg., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

NiSi19Nb3 superalloy due to its excellent mechanical properties and corrosion resistance usually is used as aerospace material. Parallel to the improving effect on the mechanical properties by alloying with boron and carbon in this material, its corrosion behavior is also interested for the further research. NiSi19Nb3 superalloy was melt doped with distinguished boron/carbon contents. Test materials were at 1080ºC for 4 hours solid solution and then at 700ºC for 10 hours aging treated. Various precipitates of test materials make their corrosion behavior noticeable difference that was studied according to the potentiostate (Perkin Elmer-263A) and their weight deviation. The microstructures of test materials and the precipitates in it were investigated via OM, SEM, EPMA and TEM. Some eutectic precipitates were observed obviously. Their influence on the corrosion behavior was studied in detail accordingly.

11:25 AM
Microstructural Evolution of Ni3Al Base Alloy IC6 and NiCoCrAlY Overlay Coating During Long Term Aero-Engine Test: *Yafang Han*[1]; Jinxia Song[1]; Shusuo Li[2]; [1]Beijing Institute of Aeronautical Materials, PO Box 81, Beijing 100095 China; [2]Beijing University of Aeronautics and Astronautics, Sch. of Matls., Xueyuan Rd. 37, Beijing 100083 China

The turbine vanes made of Ni3Al base alloy IC6 with NiCoCrAlY overlay coating were undergone engine test for 387h. The change of surface morphology and microstructure of both the substrate alloy and coating during the engine test has been studied. The results showed that there were no obvious microstructure change for the substrate alloy, and the NiCoCrAlY overlay coating were basically complete and still had certain oxidation and corrosion resistance, i.e., there was still a dense oxide layer mainly composed of Al2O3 on the surface although some black spots with various sizes appeared on the coating surface. The further analysis indicated that the contents of C, Na and Ca elements in the black spots area were higher than normal area without spots, suggesting that the formation of these black spots may resulted from the local corrosion and oxidation due to the agglomeration of the oil burning remants.

11:45 AM
Unusual Dislocation Density Oscillations After Local Melting and Solidification of Ni-Based Superalloys: *Oleg Barabash*[1]; S. S. Babu[1]; J. M. Vitek[1]; S. A. David[1]; G. E. Ice[1]; R. I. Barabash[1]; [1]Oak Ridge National Laboratory, Metals & Ceram., One Bethel Valley Rd., MS 6118, Oak Ridge, TN 37831-6118 USA

We studied how the dislocation structure of a Ni-based single crystal superalloy changes after melting and solidification. In our study, we combined polychromatic microbeam synchrotron diffraction measurements together with OIM, scanning electron and optical microscopy. We show that the distribution of the thermal gradient is not monotonic. Periodic dislocation structure is formed during continuous movement of melt zone in thin Ni-based superalloys sheet. Moreover we observe oscillations in the dislocation structure formed under such conditions at both macro and micro scales. Depending on the temperature, the formation of dislocations is accompanied by the partial or complete dissolution of ?×' particles in the matrix. Dislocations form and multiply due to thermal gradients. Their arrangement correlates with the temperature gradient field and with the dissolution and re-precipitation of ?×' particles. The distribution of the dislocation density at the macroscale is due to symmetric temperature gradient perpen dicular to the direction of melt zone movement. Within the above macro regions of dislocations oscillations of dislocation density due to grouping at the micro scale were also observed. Typical length scale of dislocation density oscillations is related to the dendrite size and the conditions of local melt and solidification.

Surface Engineering in Materials Science - III: Coating Properties and Processing Effects

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Thursday AM Room: 2022
February 17, 2005 Location: Moscone West Convention Center

Session Chairs: Lalgudi V. Ramanathan, Cidade Universitaria, IPEN/Matls. Sci. & Tech., São Paulo, 05508-000 Brazil; Arvind Agarwal, Florida International University, Dept. of Mechl. & Matls. Engrg., Miami, FL 33174 USA

8:30 AM
How Clean is Clean? Defining Acceptable Cleanliness Levels: *Mantosh Kumar Chawla*[1]; [1]Photo Emission Tech, Inc., 3255 Grande Vista Dr., Newbury Park, CA 91320 USA

A key to surface finishing is a "properly" prepared surface. Defining and maintaining the surface preparation at "proper" levels is at best subjective. Often the failure of surface preparation processes is not discovered until problems, such as poor adhesion, occur down stream resulting in non-conformance due to poor surface cleanliness. To assure consistent quality of surface cleanliness, one must not only specify an acceptable level of surface cleanliness but also specify a method for cleanliness monitoring. This generally leads to the question, what do we mean by "clean"? How clean is "clean"? This paper will discuss various methods available for monitoring surface cleanliness, their advantages and disadvantages, criteria for selecting an appropriate technique, approaches to establishing an acceptable level of cleanliness including optimum cost approach and introduce Optically Stimulated Electron Emission (OSEE) for quantitatively measuring surface cleanliness. Some typical OSEE application results will also be discussed.

8:45 AM
Vaporization Behavior of Solid Metal Carbonyls: *Dhanesh Chandra*[1]; Wen-Ming Chien[1]; Kai H. Lau[2]; [1]University of Nevada, Metallurgl. & Matls. Engrg., MS 388, Reno, NV 89557 USA; [2]SRI International, Menlo Park, CA 94025 USA

Carbonyls are generally used for CVD applications of high purity metal and alloy film formation at low temperatures. For these applications vaporization thermodynamics behavior of carbonyls is important. In this study, we have measured vapor pressures of low and high (>400 g/mol) molecular weight (MW) carbonyls using gravimetric-torsion effusion method. We will present results on Os3(CO)12, Rh6(CO)16, Ru3(CO)12, Re2(CO)10, Ir4(CO)12, Co2(CO)8, Cr(CO)6, and W(CO)6 carbonyls. There is propensity for disproportionation of solid carbonyls during heating, and it was virtually independent of molecular weight of the species. Vaporization studies of Rh6(CO)16 showed complete decomposition to nano-structured porous metallic Rh metal; the measured MWRh6(CO)16(effusing gas) is 27.75 g/mol was close to that of carbon monoxide as compared to MWRh6(CO)16(solid) of 1065.56 g/mol for the value of solid Rh6(CO)16. However, Ru3(CO)12 and Co2(CO)8 show very complex behavior. The total vapor pressures of all the above mentioned carbonyls, partial pressures of various species, average molecular weights of the effusing gases, equilibrium constants for the vaporization reactions, their enthalpies, entropies, and Gibbs energies have been determined.

9:00 AM
Understanding the Relationship Between Surfactant Adsorption and Metal Corrosion Inhibition: *Michael L. Free*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Surfactants can be used as effective corrosion inhibitors in many industrial settings. This study examines the relationship between surfactant adsorption and corrosion inhibition as well as the factors that affect surfactant adsorption.

9:15 AM
The Thickness Uniformity of Films Deposited by Multi-Workpiece Magnetron Sputtering: Fu Chun Lin[1]; Yang Chuan Ren[1]; [1]University of Electronic Science & Technology of China, Inst. of Microelect. & Solid State Elect., Chengdu, Sichuan 610054 China

In this paper, a new radio frequency (RF) magnetron sputtering system with six workpiece and rotation and revolution was presented. A formula on the relation between the thickness of thin film and the parameters, such as rotation speed, revolution speed and the distance from target to substrate, was obtained. According to the formula, the three-dimensional curves of film thickness versus the parameters were drawn. From the curves, the optimum parameters of the system can be easily obtained for preparing uniform films or depositing films rapidly. It is found that the maximum relative deviation of film thickness distribution is less than 6% within a diameter of 4 inches when the ratio of the rotation speed to revolution speed is 5.3 and the results show good agreement with experimental ones.

9:30 AM
The Effect of Post-Annealing on the Structure of Barium Strontium Titanate Films Deposited by Radio Frequency Magnetron Sputtering: Liao Jiaxuan[1]; Yang Chuanren[1]; *Chen Hongwei*[1]; Fu Chunlin[1]; Leng Wenjian[1]; Zhao Li[1]; Gao Zhiqiang[1]; [1]University of Electronic Science and Technology of China, Sch. of Microelect. & Solid-State Elect., Chengdu 610054 China

Barium strontium titanate (BST) films were deposited on Pt/Ti/SiO2/Si substrate by radio frequency magnetron sputtering. Effects of post-annealing parameters on the structure of the as-deposited films have been investigated. It is found that a critical annealing temperature of approximately 500°C corresponds to the appearance of crystallization, resulting in an increase of roughness obtained by AFM. When temperature increases to 600°C, the crystallization is almost completed. AFM shows that each crystal grain grows uniformly and is 20-30nm in size, causing relatively smooth and dense morphologies. When temperature further increases, the grains become larger, thus the roughness increases. Also, annealing time has similar effect on the structure. Moreover, rapid thermal annealing in oxygen ambient can obtain much smoother and denser crystallization structure. The effects of other annealing parameters on the structure are also presented.

9:45 AM
Modeling the Effectiveness of Surfactants in Sub-Micron Particle Removal from Solid Substrates: *Michael L. Free*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Surfactants can enhance the removal of sub-micron particles from critical substrates such as silicon wafers and optical lenses by orders of magnitude. This study shows how the adsorption of surfactant can be used to predict cleaning performance as well as techniques to predict surfactant adsorption as a function of ionic strength and surfactant hydrocarbon chain length.

10:00 AM Break

10:15 AM
Microstructured Electrode Arrays for Thin Film Deposition: *Nina Lucas*[1]; Philipp Sichler[1]; Christian Schrader[2]; Lutz Baars-Hibbe[2]; Stephanus Büttgenbach[1]; Karl-Heinz Gericke[2]; [1]TU Braunschweig, Inst. für Mikrotechnik, Alte Salzdahlumer Straße 203, Braunschweig 38124 Germany; [2]TU Braunschweig, Institut für Physikalische und Theoretische Chemie, Hans-Sommer-Straße 10, Braunschweig 38106 Germany

Non-thermal plasma processing techniques have been established for a wide range of applications. Microstructured Electrode (MSE) arrays are a new plasma source for generation of non-thermal plasmas. They allow to generate large-area uniform glow discharges over a wide pressure range up to atmospheric pressure. The electrode dimensions in the µm-range are realized by photolithography and galvanic techniques. They are small enough to generate sufficiently high electric field strengths to ignite gas discharges applying only moderate radio frequency voltages (RF, 13.56 MHz, 80 V to 390 V in Ne, He, Ar and N2). One area of application for non-thermal plasma processing is thin film deposition. With the MSE arrays as plasma source some applications in the field of thin film deposition (e.g. SiO2 layers on various substrates, DLC layers) were developed and successfully tested. Another atmospheric pressure application is the sterilization of food packaging materials.

10:30 AM
An Investigation of the Adhesion and Material Transfer Phenomena Between Al, Cu, Mg, Ti Metals and CrN, DLC, TiB_2 Coatings: *Erkan Konca*[1]; Yang T. Cheng[2]; Anita M. Weiner[2]; Ahmet T. Alpas[1]; [1]University of Windsor, Mechl., Auto. & Matls. Engrg. Dept., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]General Motors R&D Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090 USA

This project has been initiated to study the relative contributions of the intrinsic and extrinsic factors that influence the adhesion and material transfer between some metals and coatings of interest to automotive industry. Rounded pins of Al, Cu, Mg and Ti were run against DLC, TiB_2 and CrN coated and uncoated M2 tool steel discs under dry sliding conditions. Tests were done under different sliding speeds (0.1-1 m/s), applied loads (1-10 N) and environments (Argon and air with various %RH). Sliding distances were kept short to focus on the initial stages of sliding. Sliding speed significantly influenced the degree of material transfer. The chemical affinity of the metal pins for oxidation, properties their oxides and interactions with the coatings had profound effects on the adhesion behavior. Here, the results of the tests performed under various conditions are explained in terms of the physical and chemical properties of the materials tested.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session VII

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Thursday AM — Room: 3003
February 17, 2005 — Location: Moscone West Convention Center

Session Chairs: Alan J. Ardell, University of California, MSE, Los Angeles, CA 90095 USA; David N. Seidman, Northwestern University, MSE, Evanston, IL 60208 USA

8:30 AM Opening Remarks

8:35 AM Invited
Atomic-Scale Dynamics of Transformation Interfaces: *James M. Howe*[1]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., 116 Engr.'s Way, Charlottesville, VA 22904-4745 USA

Frame-by-frame analyses of in-situ heating HRTEM experiments were preformed to determine the dynamics of a diffuse order-disorder interface and a sharp massive-transformation interface. The diffuse interface between ordered AuCu I and disordered alpha phases in Au-41at.%Cu alloy nanoparticles was studied at 305°C. Both the thickness and average position of the interface were found to vary with time. The disordered alpha side of the interface fluctuates more rapidly and over greater distances than the ordered AuCu I side. Frame-by-frame analyses of a moving high-index incoherent massive-transformation interface in Ti-46.5at.%Al alloy at 575°C show that the interface displays dynamic fluctuations in its trace that can be described in terms of a wave-like function with a characteristic amplitude and fundamental wavelength. The interface moves forward by the spreading of critical-size fluctuations along its length. This research was supported by NSF under Grant DMR-9908855.

9:00 AM Invited
Atomistic Modeling of Crystal-to-Amorphous Transition and Calculation of the Glass-Forming Ranges for Some Miscible/

Immiscible Binary Metal Systems: *Baixin Liu*[1]; [1]Tsinghua University, Dept. of MS&E, Beijing 100084 China

A brief review is presented to show that ion beam mixing and solid-state reaction of metal-metal multilayers are capable of producing amorphous alloys (metallic glasses) not only in miscible but also in immiscible systems within much broader composition ranges than those revealed by liquid melt quenching. In atomistic modeling, n-body potentials are derived for some miscible systems by routine methods and for some immiscible systems, where exists no any equilibrium alloy, by fitting their cross potentials to some physical properties acquired by first principles calculations. Based on the derived potential of a system, molecular dynamics simulations using solid solution models clarify that the physical origin of crystal-to-amorphous transition is crystalline lattice collapsing while solute atoms exceeding the two critical solid solubilities, between which the composition range is thus the intrinsic glass-forming range of the system. The simulation results are in excellent agreement with experimental observations.

9:25 AM

Semi-Empirical Atomistic Simulations for Energy and Structure Evolution on Surfaces or Grain Boundaries: *Byeong-Joo Lee*[1]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Engrg., Pohang 790-784 Korea

Surface energy or grain boundary energy, especially its orientation anisotropy, gives great effects on microstructural evolution and eventually on the materials properties of structural materials. Knowing the grain boundary energy and its anisotropy is essential for obtaining more realistic results through microscale simulation techniques. The surface or grain boundary structural transition is another important factor that should be considered in computational approaches. In the present study, a new atomistic computation method of grain boundary energy for arbitrary misorientations will be presented. It will also be presented that the structural transition on metallic surfaces and effect of alloying on the transition can be investigated by atomistic simulations based on semi-empirical interatomic potentials (MEAM).

9:40 AM

Microstructural Evolution Via Planar Interface Migration in Nanocrystalline Ni: *Glenn Hibbard*[1]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., 184 College St., Toronto, Ontario M5S 3E4 Canada

Microstructural evolution in nanocrystalline Ni occurs through multiple grain growth sequences. A distinctly unusual stage occurs after an initial sequence of abnormal grain growth followed by normal grain growth, in which a planar discontinuous growth interface migrates into a matrix of submicron-scale grains. This planar interface is one face of a highly symmetrically (typically cubic) growing grain. This growth morphology has been characterized by conventional transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM) with energy dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM) with orientation imaging microscopy (OIM). Several interesting features are noted including, grain embedding for certain matrix grains having a low-sigma relationship with the discontinuously growing grain, and the presence of a wetting, sulfur-rich second phase at the planar growth interface.

9:55 AM

Non-Congruent Phase Transitions of S3 Grain Boundaries in Gold: *Tamara Radetic*[1]; Ulrich Dahmen[1]; [1]Lawrence Berkeley National Laboratory, NCEM, 1 Cyclotron Rd., MS 150, Berkeley, CA 94720 USA

Au mazed bicrystal thin films were grown on germanium substrates in order to study the stability of different inclinations of S3 <111> tilt grain boundaries in gold. In as- deposited thin films, single phase smoothly curved grain boundaries were observed, but the majority of boundaries exhibit two phase structure consisting of {112} facets. In-situ heating experiments confirmed expectations that the fraction of faceted boundaries increases during annealing. At temperatures above 900°C, prior studies have shown that roughening/defaceting transition takes place, transforming two-phase faceted grain boundaries into continuously curved single phase boundaries. However, we discovered that in the presence of Ge, roughening transition occurs at temperatures as low as 225°C. This paper reports the effect of Ge on the structure of grain boundaries, discusses the thermodynamics and kinetic limitations of the observed roughening transition. In addition the implications of defaceting at low temperatures on the properties of grain boundaries such as mobility and coarsening rates are also addressed.

10:10 AM Break

10:35 AM Invited

Prediction of Phase Transformations in Titanium Alloys - Different Modelling Approaches: *Elisabeth Marie Aeby-Gautier*[1]; B. Appolaire[1]; L. Héricher[1]; J. Da Costa Teixeira[1]; B. Antoine[1]; [1]Ecole des Mines, LSG2M CNRS UMR 7484, Parc de Saurupt, Nancy 54042 France

The study and modelling of successive and competitive phase transformations of hexagonal a phase from BCC high temperature b phase is considered for metastable multi component titanium alloys. To describe the transformation kinetics in a representative volume element, an analytical approach is considered for which nucleation and growth of grain boundary a phase is formed and in a second step colonies of a platelets nucleated from the a layer and grow inside the grain. A distribution of 1000 grains is considered whose grain boundary energy varies, leading to a distribution in nucleation rate. The growth of the a phase is described considering a local equilibrium at the interface for both layers and platelets. A given criteria allows nucleation of a platelets. This description allows to take into account the influence of a previous plastic deformation. In addition two models (a model with sharp interface and one with diffuse interface (phase field model) were developed to analyse, valid, test the limits of the analytical models and even improve it. Results obtained using these three approaches will be given and compared with experimental ones.

11:00 AM

Aspects of the Microstructural Evolution in Titanium Alloys: Sujoy Kar[1]; *Rajarshi Banerjee*[1]; Eunha Lee[1]; Gopal Babu Viswanathan[1]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The solid-state beta to beta + alpha transformation in titanium alloys leads to rather interesting microstructures with features spanning across a range of length scales. Consequently, in order to understand the property-microstructure relationships in these alloys a detailed understanding of microstructural evolution is imperative. A series of controlled heat-treatments have been performed on alpha + beta Ti alloys with the objective of arresting the microstructure at successive stages of development. The crystallography of the microstructure has been studied in detail by employing orientation imaging microscopy (in a high resolution SEM), site-specific TEM sample preparation using focused ion beam, and subsequent TEM characterization. The influence of variant selection on the evolution of microstructure and the resulting development of the colony (clustering of the same variant) and basketweave (clustering of multiple variants) microstructures in these alloys will be specifically addressed in this presentation.

11:15 AM

Modelling the Local and Effective Behavior of Viscoplastic Polycrystals Using Fast Fourier Transforms: *Ricardo A. Lebensohn*[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

We present some new applications of a novel and very efficient approach[1] that makes use of the Fast Fourier Transform (FFT) algorithm to obtain the intracrystalline fields and the effective response of anisotropic viscoplastic (nonlinear) polycrystals, based on the solution of a unit-cell problem for a representative volume element with periodic boundary conditions. This approach provides an exact solution of the equilibrium equation and has better numerical performance than a FE analysis of the same problem. The FFT model is used to predict local states and morphologic and crystallographic texture evolution of fcc polycrystals, as well as to obtain the effective behavior and the average field fluctuations in statistically random fcc and hcp polycrystals, for comparison with various estimates of the self-consistent type. [1]Michel, J., Moulinec, H. & Suquet P., 2000, Comput. Modelling Engng. Sc. 1, 79.

11:30 AM

The Evolution Constitution and Microstructure During Hot-Working of Titanium Aluminide Alloys: *Michael Oehring*[1]; Lorenz Uwe[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsch., Geesthacht D-21502 Germany

Titanium aluminide alloys containing relatively large Nb additions and subjected to precipitation hardening exhibit attractive thermophysical properties, which extend the service range of conventional TiAl alloys. In order to use these materials at their full potential, structural and chemical consolidation is required. To this end, the feasibility of wrought processing of high Nb containing alloys was systematically investigated. The major areas of the study involve: (i) high-temperature deformation mechanisms, (ii) primary ingot breakdown, (iii) secondary processing, (iv) texture evolution, and (v) me-

chanical properties. The experimental investigation base on standard metallography, chemical microanalysis, mechanical testing, and high-resoltuin electrom microscopy.

11:45 AM
Initiation of Adiabatic Shear Localization in a Pre-Shocked 304 Stainless Steel: *Qing Xue*[1]; Benjamin L. Henrie[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., G755, Los Alamos, NM 87545 USA

Initiation of adiabatic shear bands in a pre-shocked stainless steel was studied to characterize the influence of prestrained texture on the onset of shear bands. Forced shear tests on hat shaped specimens was conducted using a compressive Hopkinson bar to generate shear localization. The initiation of adiabatic shear localization was found to be very sensitive to the prestrained texture, especially to distribution of defects such as deformation twins. The variation of microstructure before and during the formation of shear bands was examined and compared. Twin orientation due to the prestraining was seen to exert a significant effect on triggering the nucleation of a shear band. Multiple microbands were observed to align perpendicular to the shear direction prior to the formation of shear localization. Transmission electron microscopy was applied to identify the microstructure inside and in front of shear bands.

Notes

GENERAL POSTER SESSION

Sponsored by: TMS
Program Organizer: TMS, Warrendale, PA 15086 USA

Located in the Exhibit Hall of the Moscone West Convention Center

Monday, February 14, 2005 at 12:00 Noon to Wednesday, February 16, 2005 at 3:00 PM

Authors will be present during the following hours:
Monday, 5:00-6:00 PM
Tuesday and Wednesday, 12:00 Noon-2:00 PM

Surface Science Studies of the Effect of Ni2+ on Zinc Phosphate Coatings on Aluminum: *A. S. Akhtar*[1]; D. Susac[1]; K. C. Wong[1]; P. C. Wong[1]; K. A.R. Mitchell[1]; [1]University of British Columbia, Dept. of Chmst., Vancouver Canada

The present work is part of a broader investigation of the effects of additives in zinc phosphate (ZPO) coating solutions that are designed for specific applications to Al and its alloys. ZPO conversion coatings improve the corrosion resistance of the Al substrate and increase the adhesion of paint. Coatings formed on 2024-T3 aluminum alloy, after dipping in ZPO coating baths containing Ni2+, have been studied by x-ray photoelectron spectroscopy (XPS), SEM, EDX, scanning Auger microscopy (SAM), and electrochemical measurements. Significant variations are observed in coating morphology, and in stability against corrosion, as the amount of Ni2+ in the coating solution increases through the 0 to 2000 ppm range. There are local variations in the amount of Ni2+ in the coatings, due in part to the existence of second-phase particles in the 2024-Al sample. These findings, along with chemical information about coatings formed when the pH of the coating solution is varied, give insight into the mechanistic role of Ni2+ in ZPO coating formation.

Determination of Residual Stresses on Thick Films Using Theoretical and Experimental Techniques: *Andrea Maria Hodge*[1]; Octavio Cervantes[1]; Gilbert F. Gallegos[1]; Ronald J. Foreman[1]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., PO Box 808, L-352, Livermore, CA 94550 USA

A complete characterization of residual stresses on thick films ranging from 8 to 30% of the substrate thickness was performed using three distinct characterization techniques: white light profilometry, stylus profilometry, and x-ray diffraction. The films were deposited using the magnetron sputtering technique with and without bias. The profilometry measurements were used in three different residual stress analysis equations: Stoney, Röll and Benabdi/Roche equations. Stoney's and Röll's equations are primarily used for coatings with thickness significantly less than the substrate thickness. In contrast, the Benabdi/Roche equation, which is based on Timoshenko's bilayer theory, evaluates coating thickness approximating the substrate thickness. It was observed that as the film thickness increases, Stoney's equation underestimates the residual stresses as much as 50% when compared to the Benabdi/Roche equation. The residual stress values obtained from the profilometry results using Benabdi/Roche equation were verified by the x-ray diffraction technique and have shown to be in agreement.

Educational Tools for Undergraduate Kinetics: *Afina Lupulescu*[1]; Martin Eden Glicksman[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., 110, 8th St., Troy, NY 12180 USA

The authors designed integrated sets of teaching modules for undergraduate and graduate courses in materials science and engineering. We created or found small interactive computer programs, Java applets, or movies that integrate with a set of lecture modules. Two approaches are used for developing additional module content: 1) New Java applets are conceptualized by us and then built by computer science undergraduate students, an approach partially supported by the NSF (DMR-03038132). 2) Sources on the Internet are searched for programs that integrate with our lecture modules. Students using the applets are first taught the relevant scientific concepts. They provide parameters requested in the applets, obtain results, and then compare the results with theory. The authors find that applets integrated with lecture modules serve as yet another effective means to transmit and develop knowledge, to improve understanding, and to help ascertain the comprehension and skill level of the student.

Effects of Changes in Microstructure and Strain Rate on Flow Behavior of Al 6061: Ali Shamimi[1]; Xin Tang[2]; John J. Lewandowski[1]; Vikas Prakash[2]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA; [2]Case Western Reserve University, Dept. of Mechl. & Aeros. Engrg., Glennan Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA

Aluminum-based sandwich panels with textile cores possess high stiffness and strength at low weight. Recent works have documented the energy absorbing characteristics of these materials at low strain rates. However, very little information exists on the energy absorption of these structures at high strain rates. In order to address this, the behavior of the individual constituents over a range of strain rates is first needed.In this investigation the static and dynamic deformation behavior of Aluminum 6061 in two different conditions, T6 and over aged (OA), is reported. Static and intermediate rate experiments were performed by using a MTS open-die forging machine with high load capacity (e.g. up to 220 Kip) and deformation rates up to 120"/Sec. Higher strain rate experiments were performed by using a Split Hopkinson Pressure Bar (SHPB).The results reveal changes in the behavior of the material over a range of deformation rates. The poster will summarize the effect of changes in microstructure and strain rate on flow behavior of Aluminum 6061. The implications for design of energy absorbing structures will also be presented.

Equilibrium Solid-Liquid Interfacial Properties in Lennard-Jones Alloys: *Chandler Amiss Becker*[1]; Mark D. Asta[1]; Jeffrey J. Hoyt[2]; Stephen M. Foiles[2]; [1]Northwestern University, Dept. of Matls. Sci., 2220 Campus Dr., Evanston, IL 60208 USA; [2]Sandia National Laboratories, Computatl. Matls. & Molecular Scis., PO Box 5800, MS-1411, Albuquerque, NM 87185-1411 USA

The composition dependence of the magnitude and associated crystalline anisotropy in the crystal-melt interfacial free energy is of primary importance in determining alloy solidification rates and morphologies. To date the most detailed information concerning crystal-melt interfacial properties has been derived from atomic-scale computer simulations. However, previous work has focused primarily on elemental systems, and far less is known about alloys. We are using Molecular Dynamics fluctuation techniques to determine the interfacial free energy of crystal-melt interfaces in alloys, and this is combined with knowledge of how the equilibrium interfacial solute adsorption depends on size and energy mismatch for a more complete understanding of the interfacial thermodynamics of Lennard-Jones alloy systems.

Deformation and Recrystallization Textures and Low Temperature Hardening Responses in A6111: *Cynthia Sin Ting Chang*[1]; Brain John Duggan[1]; [1]University of Hong Kong, Dept. of Mechl. Engrg., Pokfulam Rd., Hong Kong China

In this paper, the 6111 aluminum alloy received as hot band was divided into 2 batches. The first batch of material was directly rolled to 80% reduction. The second was first solution heat treated at 600°C for 1 hour and cold rolled to 80%. After cold rolling, the 2 batches of material were annealed at 450°C, 500°C and 540°C for 10 minutes in an air furnace. Tensile specimens of both materials were pulled to 5% elongation in a tensile test machine and aged at 180°C in an oil bath to simulate the paint bake cycle. For batch 1, the recrystallization texture consisted of strong {011}<100>, and {011}<211>, components with a small volume fraction of cube at all temperatures investigated. However, for batch 2, the recrystallization texture of the material which had been annealed at 450°C exhibited a retained rolling texture with cube and rotated cube components, while the texture for the materials which were annealed at 500°C and 540°C showed a fairly random texture, with the maximum intensity in the ODFs of around 3

x random. Cupping tests were also performed in material which had been annealed at 540°C, with and without solution heat treatment before cold rolling, and it was found that the earing percentage from the batch 2 materials was only 0.3% while for batch 1 material, 1%. After strain aging at 180°C, the materials which had been annealed at 540°C produced the highest peak aging hardness of 100VHN. The redistribution of atoms from precipitates into solution during solution heat treatment can affect either plastic in stability, which would modify nucleation site formation, or subgrain and grain boundary mobility, which is know to modify greater texture formation. Work is continuing on the reason for the desirablt random texture production. The strain aging behavior is due to the precipitates of Mg5Si6 in the dislocation substructures.

Plasma Surface Modification of TiO2 Photocatalysts for Improvement of Catalytic Efficiency: *Chung-Kyung Jung*[1]; Yong-Hwa Song[1]; In-Seob Bae[1]; Jin-Hyo Boo[1]; [1]Sungkyunkwan University, Dept. of Chmst., Suwon, Kyunggi-do 440-746 S. Korea

We have deposited titanium dioxide (TiO2) thin films on glass using titanium (IV) iso-propoxide (Ti[OCH(CH3) 2] 4, 97%) by sol-gel processing. In order to elevate photocatalytic activity of the as-grown TiO2 films, argon and oxygen plasmas ignited by radio-frequency (RF) and microwave (MW) under both vacuum and atmosphere conditions were also used in the range of 50-200 W within 0.5 hr at room temperature. Photocatalytic activity was evaluated by the measurements of the contact angle, UV/vis. irradiation, and toluene removal test. In this work, the effect of the plasma with photocatalyst (TiO2) on the improvement of hydrophilic properties has mainly been investigated. A superhydrophilic property and surface morphology change appeared in the light irradiation with O2 plasma treatment. Based on this work, we confirmed that the plasma treatment method was very reliable method for the synthesis of TiO2 thin films with high catalytic performance.

Dissimilar Friction Stir Welding of a Magnesium-Base Metal Matrix Composite and a Monolithic Magnesium Alloy: Chang-Yong Lee[1]; Won-Bae Lee[1]; Yun-Mo Yeon[2]; Seung-Boo Jung[1]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Chunchun-dong, Changan-gu, Suwon, Kyounggi-do 440-746 Korea; [2]Suwon Science College, Dept. of Automated Sys., Botong-li, Jungnam-myeon, Hwasung, Kyounggi-do 445-742 Korea

Joining MMCs and monolithic alloys by fusion welding is challenging as a result of segregation and reaction of the reinforcement phase in the fusion zone. However, Friction stir welding (FSW) enables us to get good welds because the overall process goes through under the melting temperature of welded material. FSW of a Mg-base metal matrix composite and a monolithic Mg alloy was conducted in this study. AZ91 Mg alloy MMCs containing 10wt% SiC Particles and AZ91 Mg alloys were well joined by FSW at some limited welding speeds and tool rotation speed of 1250rpm. The location of material against advancing direction of tool did not affect welding results. The weld zone was characterized by the homogeneously distributed SiC particles, equaxed grain structure and the dissolution of â phase. Vickers hardness and specific wear test was carried out for evaluation of mechanical properties.

The Solder Joint Reliability of Lead-Free During Thermal Cycle Test: Chang Youl Lee[1]; Jae Wook Lee[1]; Ik Won Choi[1]; Choul Su Kim[1]; Dong Hwan Kim[1]; Hun Ju Han[1]; [1]Samsung Electronic, Computer, 416, Maetan-3Dong, Yeongtong-Gu, Suwon, Gyeonggi-Do 443-742 Korea

The study of solder joint reliability of high-density packages using lead-free solder has conducted. The components studied include many SMT (surface mount technology) package types and Im-Ag has been used as PCB (printed circuit board) finishes. Lead free solder joint has been continuously monitored during Thermal Cycling Test after assemble to verify that solder joints are intact and components are oriented correctly to complete daisy chain strings.

Control of Noise and Specimen Temperature During 1 KHz Fatigue Experiment: Hongbo Tian[2]; *Douglas E. Fielden*[1]; Melanie J. Kirkham[1]; Peter K. Liaw[1]; [1]University of Tennessee, 106 Dougherty Engrg. Bldg., Knoxville, TN USA; [2]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID 83415 USA

Both sound-proofing and sound-conditioning methods were employed in the construction of a sound-insulation room to house a 1,000 Hz servohydraulic fatigue-testing machine to protect researchers from excessive noises caused by the running machine. The “Box inside a box” construction and acoustical foams were used to improve the sound insulation of the room. Special attention was paid to a door, a window, and cable holes. In addition, the specimen self-heating effect during a high-frequency experiment was found to influence the fatigue life of materials. A dedicated cooling method, using nitrogen gas, was employed to control the specimen temperature during fatigue testing.

Interfacial Reactions and Shear Strength in Flip Chip Solder Bump Using Stencil Printing Method: Dae-Gon Kim[1]; Hyung-Sun Jang[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Cheoncheon-dong, Jangan-gu, Suwon 440-746 S. Korea

Flip chip interconnection is one of the most promising electronic packaging technologies which facilitates die size package, high I/Os, and good electrical performance. In this work, we investigated the growth kinetics of the intermetallic compound layer formed between Sn-Ag-Bi-In solder bump and UBM by solid-state isothermal aging at temperatures between 343 and 423 K for periods ranging from 0 to 100 days. A quantitative analysis of the intermetallic compound layer thickness as a function of aging temperature and time was performed. The growth rate constants of the intermetallic compound, which are known to vary as a function of the aging time and aging temperature, are fitted using a simple parabolic equation. In addition, the activation energy for the growth of the intermetallic compound was obtained by means of Arrhenius-equation. Shear tests were performed to investigate the mechanical strength of the joints between the solder and the UBM.

Tensile Behavior of an Ultrafine-Grained Cryomilled Al 5083 Alloy: *David Edward McDougall*[1]; Manish Chauhan[1]; Indranil Roy[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

In the present study, the relationships between the structure and properties of a cryomilled Al 5083 alloy have been investigated. The tensile tests were performed on a cryomilled Al 5083 alloy with an average grain size of 300 nm in the temperature range of 473 to 673 K and strain rates of 10-4 to 10-2 s-1. The microstructures associated with the deformation processes were characterized using transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Testing revealed high strength, relatively high ductility, and low strain hardening. Tensile deformation was fairly uniform without major necking and the fracture surface was found to have a uniform distribution of dimples. The small grain size, solid solution strengthening, and dislocation strengthening contribute to the properties of the cryomilled Al 5083 alloy.

Plastic Strain and Grain Orientation Effects in the Surface Roughening of Aluminum Alloys: *E. J. Moore*[1]; M. R. Stoudt[2]; R. C. Reno[1]; [1]University of Maryland, 1000 Hilltop Cir., Baltimore, MD 21250 USA; [2]National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg, MD 20899 USA

Deformation-induced surface roughness presents a significant challenge to the use of aluminum alloys in many exterior automotive applications. Both grain size and grain orientation are known to have strong influences on the character of the surface roughness. A better understanding of the interaction between these two dominant influences is needed for the development of better predictive models of deformation behavior. Samples of a high-purity model Al-Mg alloy were heat treated to produce four distinct mean grain diameters ranging from less than 1 mm to 3 mm. The samples were incrementally strained in uniaxial tension to 12%. Electron backscatter diffraction (EBSD) was used to characterize the crystallographic orientation prior to and after each plastic strain increment. The roughening character was evaluated with scanning laser confocal microscopy (SLCM). Preliminary results from this study indicate that although the overall surface roughness increases with macroscopic strain level, the within-grain roughening behavior varies significantly with the orientation.

Strengthening Barrier Capability of TiN Thin Film Against Copper Diffusion by Aluminum Doping: Giin Shan Chen[1]; Chieh Chen Fu[1]; Li Chung Yang[2]; [1]Feng Chia University, Dept. of Matls. Sci. & Engrg., 100 Wenhwa Rd., Seatwen, Taichung 407 Taiwan; [2]National Huwei University of Science and Technology, Dept. of Matls. Sci. & Engrg., 64 Wenhua Rd., Huwei, Yunlin 632 Taiwan

This work employs grazing-incidence X-ray diffractometry, transmission electron microscopy, Rutherford backscattering spectrometry, and electrical measurements to examine the effectiveness and failure behavior of sputter-deposited TiN and Al-doped TiN thin films as diffusion barriers of Cu for silicon wafers (or silicon dioxide dielectric layers). Results based on these analyses consistently confirm that the performance of TiN (40 nm thick) can be greatly improved by doping ~10 atom % of Al. The as-deposited TiN barriers possess an NaCl-type and voided-columnar (~5 nm) microstructure, which, upon annealing (greated than 500°C), are transformed into equiaxed, coarse (~15 nm) grains, providing short diffusion routes (grain boundaries) for Cu to readily penetrate TiN. Conversely, the Al-doped TiN barriers

still maintain a dual-phase (NaCl-TiAlN and cubic-Ti_3AlN) and fine (~4.5 nm),"fibrous" columnar microstructure after annealing,hence being highly effective in retarding Cu penetration. The role of Ti_3AlN precipitates in enhancing the barriers is currently under investigation.

Gas-Sensing Properties of Tungsten Oxide Thin Films Loaded with Metal Nanoclusters Using Electroless Plating: Giin Shan Chen[1]; Wen Lu Liao[1]; Chung Kwei Lin[1]; [1]Feng Chia University, Dept. of Matls. Sci. & Engrg., 100 Wenhwa Rd., Seatwen, Taichung 407 Taiwan

This work examined the effects of film properties (such as microstructure and stoichiometry), stabilization treatments, and metal/oxide loadings on the gas-sensing behavior of WO_3 thin films deposited by reactive sputtering over a broad range of O_2-to-Ar pressure ratios. Preliminary results showed that gradually increasing the O_2 pressure ratio sequentially induced the formation of poor-crystallized, property-unstable oxygen-incorporated beta-W (probably W_3O) and two polymorphic forms of WO_3. The films can be crystallized and become highly stable after an optimal thermal treatment. Moreover, a novel electrochemical integrated process is employed to deposit ultra-fine (~3 nm) catalytic nanoclusters, such as Ni, Cu, and Co, on (or within) WO_3 thin films using electroless plating, in conjunction with a thermal oxidation to transform the metals into oxides. Transmission electron microscopy, x-ray absorption spectroscopy, and gas (NO_x) adsorption measurements are also used to characterize the gas-sensing properties of the metal and oxide loaded WO_3 films.

Characterizing Sputter-Deposited W-N and W-Ti-N Barriers for Copper Metallization: Giin Shan Chen[1]; Wei Cheng Hung[1]; Chen Siang Hsu[2]; Jau Shiung Fang[2]; [1]Feng Chia University, Dept. of Matls. Sci. & Engrg., 100 Wenhwa Rd., Seatwen, Taichung 407 Taiwan; [2]National Huwei University of Science and Technology, Dept. of Matls. Sci. & Engrg., 64 Wenhua Rd., Yunlin, Huwei 632 Taiwan

This work examines the influence of alloying titanium and (or) nitrogen elements on the film properties (electrical resistivity, microstructure, etc) and capability of sputter-deposited tungsten thin films as diffusion/drift barriers for copper metallization. Thin films of tungsten generally exist as a high-resistivity form (beta-W), typically greater than 120 micron ohm-cm. However, the resistivity can be significantly reduced to ~35 micron ohm-cm merely by alloying adequate amounts of titanium (~10 to 30 atom %) to the tungsten matrix, while without deteriorating the barriers. Preliminary results show that co-sputtering of W and Ti under an optimal argon-to-nitrogen partial pressure ratio results in the formation of W-Ti-N thin films with high conductivity, acting as reliable barriers for copper metallization in integrated circuits. Transmission electron microscopy, x-ray absorption spectroscopy, and various barrier evaluation methods will be used to characterize the interfacial-reacting behavior and barrier properties of the stacked film systems.

Hydrogenation Properties of Ti-xNb-10Cr Alloys: *Kyung Shin*[1]; Young-Geun Lee[1]; Sung-Lim Ryu[1]; Soon-Chul Ur[1]; Jung-Il Lee[1]; Il-Ho Kim[1]; Soon-Yong Kweon[1]; Tae-Whan Hong[1]; [1]Chungju National University, Dept. of Matls. Sci. & Engrg., 123 Gumdan-Ri, Iryu-Myon, Chungju, Chungbuk 380-702 Korea

Ti-Cr alloys consist of BCC solid solution mixing with C36, C14 and C15 Laves phases at high temperature. Among others, BCC solid solution phase was reported to have a high hydrogen storage capacity. However, wide range of hysteresis at hydrogenation/dehydrogenation and degradation of hydrogen capacity on hydriding/dehydriding cycles as well as activation capacity have to be improved for its application. In order to address these problems, Nb addition to this system was considered in this study. To obtain target materials, Ti-5Nb-10Cr and Ti-10Nb-10Cr specimens were prepared using planetary ball mill. Milling process carried out under an argon atmosphere. Specimens synthesized was characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and thermo-gravimetric analysis/differential scanning calorimetry(TG/DSC). In order to examine hydrogenation behavior, a pressure-composition-isotherm (PCI) tests were carried out at 293, 343, 393, 443, 493 and 543K.

Optimization of Continuous Casting Process for Copper Bonding Wire: *Hoon Cho*[1]; Hyung-Ho Jo[1]; Young-Jig Kim[2]; [1]Korea Intitute of Industrial Technology, Advd. Matl. R/D Crt., 994-32 Dongchun-dong, Yeonsu-gu, Incheon 406-130 Korea; [2]Sungkyunkwan University, Sch. of Metall. & Matl. Engrg., 300 Chunchun-Dong, Jangan-Gu, Suwon, Gyounggi-Do 440-746 Korea

Recently the application of copper as a substitute of gold bonding wire has been investigated because formation of brittle intermetallic compound such as aluminide between aluminium substrate and bonding wire can be suppressed. Especially, copper being cheaper than gold and with higher conductivity and better stiffness, is a viable, cost-effective alternative. In order to manufacture copper ultra fine wire for bonding wire in integrated circuit package, continuous casting process, which can produce high purity copper rod with small cross section, has to be optimized to produce cast rod without internal defects and to control microstructure orientation which can prevent wirebreaks in wiredrawing process. The paper presented here is mainly aimed at investigation of influence of varying parameters of pulling speed, superheat and rod diameter on grain morphology of the cast rod and on its drawing characteristics to ultra fine wire.

Experimental Simulation of Two-Phase Pressure Drop in the Evaporator of a McGill Heat Pipe: *Hujun Zhao*[1]; Pietro Navarra[1]; Frank Mucciardi[1]; John Gruzleski[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

The McGill Heat Pipe, containing a spring shaped flow modifier, is capable of handling the very large heat fluxes typically encountered in some metallurgical industries. Experimental simulations were carried out to investigate the flow patterns and pressure drops in the evaporator with an air-water two-phase flow by using five different spring flow modifiers. The flow patterns were captured by a video camera, and the effect of a flow modifier can be seen clearly. The relationship between the pressure drop and the returning velocity was discussed. A new non-dimensional parameter, swirler parameter, is proposed in the analysis of the friction factor in single-phase flow. It was also shown that the homogeneous model was not suitable to describe the two-phase flow in the presence of a flow modifier. Lockhart-Martinelli method was used to analyze the pressure drop,and the constant C in Chisholm's equation was modified as a function of the swirler parameter.

Effects of Sb2O3 on the PTCR Properties of (Ba,Sr)TiO3: Jong-Bum Park[1]; Sin-Wook You[1]; Kyoung-Won Cho[1]; Ho-Won Lee[2]; Young-Min Kim[2]; Jung-Il Lee[1]; Soon-Chul Ur[1]; *Il-Ho Kim*[1]; [1]Chungju National University, Dept. of Matls. Sci. & Engrg., 123 Geomdan-ri, Iryu-myeon, Chungju, Chungbuk 380-702 Korea; [2]Corea Electronics Corporation, Dept. of Matls. Dvlp., 168-16 Yongtan-dong, Chungju, Chungbuk 380-250 Korea

BaTiO3 has been widely applied to condensers, rf filters, piezoelectric oscillators, etc., since it has ferroelectric, pyroelectric, piezoelectric and optoelectric properties. Conduction behavior of BaTiO3 can be changed by substituting Ba2+ with 3+ donors such as La3+, Y3+ and Sb3+, or by substituting Ti4+ with 5+ acceptors such as Nb5+ and Ta5+. In this study, (Ba,Sr)TiO3 with perovskite structure was prepared and effects of Sb2O3 on its PTCR properties were investigated. (Ba,Sr)TiO3 with 0.05~0.25 mol% Sb2O3 showed semiconducting PTCR behavior and anomalous grain growth was also observed when sintered above 1600K. It was considered that charge compensation by doping Sb2O3 as well as abnormal grain growth by sintering lead to resistivity reduction from insulating to semiconducting transition. This research was supported by the Program for the Training of Graduate Students in Regional Strategic Industries which was conducted by the Ministry of Commerce, Industry and Energy of the Korean Governmment.

Microstructure and Mechanical Properties of Friction Stir Welded Magnesium Alloys: *Ichinori Shigematsu*[1]; Yong-Jai Kwon[1]; Naobumi Saito[1]; [1]National Institute of Advanced Industrial Science and Technology, 2266-98, Anagahora, Shimoshidami, Moriyama-ku, Nagoya 463-8560 Japan

The investigation of the microstructure of FSWed cast AZ91 magnesium alloy and the examination of their mechanical properties were carried out. As a result, the cast AZ91 that is difficult to weld by TIG or MIG can be jointed very successfully by using the FSW technique. Furthermore, it is clarified that the mechanical properties of the stir zone were improved as compared to those of the base material because of the grain refinement and the homogeneously dispersed fine precipitates. The maximum value of the tensile strength and elongation were 289MPa and 11%, respectively. 0.2% yield strength, however, was a constant value of approximately 140MPa irrespective of the tool rotation speed, which was the same value as the base material. The stir zone consisted of the equiaxed grains and some elongated grains along the metal flow. The average grain size was from 4.5 to 7 microns.

Component Corrosion: Material, Design and Operating Issues: *James T. Staley*[1]; [1]Bodycote Materials Testing, Inc., Chicago Lab., 7530 Frontage Rd., Skokie, IL 60077 USA

When investigating component corrosion, it is critical to access what role the material, design and/or operating conditions may have played in corroding the part. When this is determined, a reasonable recommendation can be made on how to avoid future corrosion that may lead to component failure. This poster highlights a variety of

component corrosion modes and a number of different roles played by the material, design and/or operating conditions. In addition, techniques used to discover these roles are displayed as well as resulting recommendations on how to avoid corrosion of various structural materials in several key industries.

The Effect of Additive V_2O_5 on Sinter Mechanism and Properties of Inert Anode of $NiFe_2O_4$ Spinel: *J. H. Xi*[1]; Y. H. Liu[1]; G. C. Yao[1]; [1]Northeastern University, Sch. of Matl. & Metall., Liaoning, Shenyang 110004 China

In order to improve the properties of inert anode of $NiFe_2O_4$ spinel, some additive V_2O_5 was added to raw materials - powders of NiO and Fe_2O_3. The powders of NiO, Fe_2O_3 and slight amount of V_2O_5 were mixed, then mould and sinter at 1200° for 6h. The sinter mechanism of powders of NiO and Fe_2O_3 with some additive V_2O_5 was researched. The effect of V_2O_5 on density, electric conductivity and corrosion resistance of inert anode of $NiFe_2O_4$ spinel was studied at the same time. The results show that the sinter mechanism of powders of NiO and Fe_2O_3 with some additive V_2O_5 is liquid-phase sintering; Additive V_2O_5 can increase the density of the samples; especially it improves the corrosion resistance of the samples remarkably. When the amount of V_2O_5 is 1.5%, the sample's corrosion rate is 1/80 of that of sample without V_2O_5. But the electric conductivity of the samples with V_2O_5 is lower than that of the sample without V_2O_5.

TEM Observation of Interfacial Layers Formed Between Pb-Free Sn-Ag Solder and ENIG Substrate: Jeong-Won Yoon[1]; Sang-Won Kim[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Chunchun-dong, Changan-gu, Suwon, Kyounggi-do 440-746 Korea

The microstructure of solder joints has been studied mostly using scanning electron microscopy (SEM). But, a high spatial resolution is required for qualitative analysis because the reaction layers or products of solder joints after reflow are quite thin or small. Therefore, transmission electron microscopy (TEM) becomes one of the most promising analytical methods for this technology. The eutectic Sn-Ag solder is one of the most promising Pb-free candidates to replace the Sn-Pb solders. In addition to solder, printed circuit boards (PCBs) and component surface finishes also have to be Pb-free. Electroless Ni has been widely used as a diffusion barrier layer on the Cu bond pad for flip-chip and ball-grid-array (BGA) solder bumps. In this study, we evaluated the interfacial reaction between eutectic Sn-Ag solder and ENIG (Electroless Nickel/Immersion Gold) plated Cu substrate by using SEM and TEM. TEM samples were prepared by ultramicrotome.

As-Cast Surface Modification of Casting Alloys Using Combustion Synthesized Intermetallic Compounds: *Kyeonghwan Choe*[1]; Guesurb Cho[1]; Kyongwhoan Lee[1]; Akira Ikenaga[2]; [1]Korea Institute of Industrial Technology, Advd. Matl. Procg. Team, 994-32 Dongchun-dong, Yeonsu-Gu, Incheon 406-130 Korea; [2]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Grad. Sch. of Engrg., 1-1 Gakuen-Cho, Sakai, Osaka 599-8531 Japan

Intermetallic compounds of nickel aluminides are considered as candidate materials for high temperature structural and coating applications owing to high specific strength at elevated temperatures and good oxidation resistance. Combustion synthesis is one of the powder metallurgical techniques for forming an intermetallic compound using the negative heat of formation. In this study, we apllied combustion synthesis to the surface modification of casting alloys. In this method, the heat of the melt acted as the ignitior of combustion synthesis, and casting of metallic substrate, synthesis of nickel aluminide and dissimilar joining of two materials were achieved simultaneously. We examined the effect of composition of powder mixture and casting alloys on formation of intermetallic compounds and interaction between metallic substrate and intermetallic compounds.

Preparation of Nano-Sized Ni 8 wt% Doped and NiTiO3 Mixture Titanium Dioxide Photocatalyst by Mechanical Alloying and Heat Treatment: *Dong Hyun Kim*[1]; Ha-Sung Park[1]; Sun-Jae Kim[2]; Kyung-Sub Lee[1]; [1]Hanyag University, Div. of Matl. Sci. & Engrg., 17, Haengdang-dong, Seoungdong-ku, Seoul 133-791 Korea; [2]Sejong University, Dept. of Nano Sci. & Tech./SAINT, Seoul Korea

In order to effectively utilize visible light in photocatalytic reaction, mixture of nano-sizd Ni 8 wt% doped TiO2 and NiTiO3 powders were synthesized by mechanical alloying and heat treatment. After heat treatment at 1000°C for 4h, MAed powders formed a mixture of Ni-doped rutile and NiTiO3 phase. The UV-DRS and PL investigation showed that the mixture powder had the higher wavelength range 550-630 nm (2.2-1.9 eV) than that of Ni doped powder (500-550 nm), indicating that the NiTiO3 mixture phase shifted the absorption onset into visible light region and enhanced the photoefficiency. The average grain size of mixture powder was in the rage 2-4 nm. EDS demonstrated that the NiTiO3 were dissolved in the Ni doped TiO2.

High Temperature Oxidation of SiOC/MoSi2/SiC Composites: *Ko Jae Hwang*[1]; Deug J. Kim[1]; Dong Bok Lee[1]; [1]Sungkyunkwan University, Sch. of Metallurgl. & Matls. Engrg., 300, Chun-Chun-dong, Suwon, Kyungi-do 440746 S. Korea

From the preceramic polymer of polymethylsiloxane (PMS) and powders of MoSi2, SiC and Si, new ceramic composites that consisted primarily of an amorphous SiOC matrix containing dispersed particles of MoSi2 and SiC were synthesized. The prepared composites displayed superior oxidation resistance at both high and low temperatures by forming the SiO2 on the surface. The thin, amorphous SiO2 layer that formed initially gradually transformed to crystalline during oxidation between 1000 and 1300°C. An outer highly porous and an inner superficial SiO2 layer that formed from the initial stage of oxidation between 400 and 500°C protected the composites from pesting.

Microstructural Evolution of TiAl Alloys Containing W and B: *Lan Huang*[1]; Chain T. Liu[2]; Peter K. Liaw[3]; Bai Y. Huang[4]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 323 Dougherty Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Knoxville, TN USA; [3]University of Tennessee, Dept. of Matls. Sci. & Engrg., 427-B Dougherty Bldg., Knoxville, TN 37996-2200 USA; [4]Central South University, Changsha, Hunan China

TiAl alloys have been considered as promising candidates for structural-material applications at around 800C. In this work, TiAl alloys, containing W and B, have been developed. Using the scanning-electron microscopy (SEM), electron-microprobe, and transmission-electron microscopy (TEM), the effect of W on the microstructural evolution of TiAl alloys, including the colony size and lamellar spacing, were analyzed. It is interesting that fine microstructures (with the colony size smaller than 50 um) can be conveniently obtained just after HIPping the as-cast alloys at 1,250C and 150 MPa for 4 hrs. It was found that tungsten prefers to react with boron to form borides, and disperses mainly in grain boundaries, and occasionally inside grains. With the increase of the tungsten content, the microstructure can be further refined. It is also noteworthy that no beta phase forms at a tungsten content up to 0.7 weight percent (wt%). Heat treatments at temperatures ranging from 900C to 1,310C were conducted. The addition of tungsten can restrain the grain coarsening and stabilize the microstructure up to 1,280C by hindering the migration of grain boundaries at high temperatures. We would like to acknowledge the financial support of the Oak Ridge National Laboratory (ORNL), the Chinese Natural Science Foundation, and the United States National Science Foundation (NSF), the Combined Research-Curriculum Development (CRCD) Program, with Ms. Mary Poats as the contract monitor, under the contract number of DGE 0203415.

Grain Growth Behavior of Nanocrystalline Ni in Low Temperature Range: *Manish Chauhan*[1]; Yuwei Xun[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

Grain growth behavior of nanocrystalline Ni, produced by an electrodeposition technique, with an average grain size of 15 and 25 nm was studied in the homologous temperature range of 0.20 to 0.40 Tm. No appreciable grain growth was observed in the temperature range of 0.20 to 0.30 Tm, even after long annealing times (as long as 4 hours). However, in the temperature range of 0.3 to 0.4 Tm, the rate of grain growth was high up to 2 hours of annealing time, then decreases with longer times. The grain growth behavior of 25 nm Ni sample was compared with 15 nm Ni sample. It was found that grain growth in the sample with starting grain size of 15 nm was very unstable and rapid grain growth was observed as compared to 25 nm sample at 693 K.

Fabrication and Frequency Response of Complex Ultrasonic Transducer for Multi-Layers Evaluation: *Man-Jong Lee*[1]; Young Soo Yoon[2]; Joo Sun Kim[3]; [1]Agency for Defense Development, PO Box 35, Yousung, Daejon 305-600 Korea; [2]Konkuk University, Dept. of Advd. Fusion Tech., 1 Hwayang-dong, Gwangjin-gu, Seoul 143-701 Korea; [3]Korea Institute of Science and Technology, Nano-Matls. Rsch. Ctr., PO Box 131, Cheongryang, Seoul 130-650 Korea

The main purpose of present study is to develop a complex ultrasonic transducer (UT) comprising a dual-element ultrasonic transducer (DEUT) and an additional active element for the characterization of multi-layered facilities used in the corrosive fluid flow system. The target transducer in this study was designed to measure the remaining thickness of parent materials as well as the coated layers regardless of the type of coated materials (paints, epoxy, and etc). As active dual piezoelectric layers (active element 1) responsible for the measurement of the thicker part, lead metaniobate (PbNb2O6) was selected. To fabricate high quality lead metaniobate thick films, tape casting

was selected, which is a convenient and highly useful method for the large-scale production. Also, the other single active layer (active element 2) measuring the coated layer of the test piece, spin coated Pb(Zr0.52Ti0.48)O3 (PZT) thin film using sol-gel process was selected. PZT is an excellent candidate not only because it has excellent properties such as high dielectric constant (es/e0=830) and sensibility (kT=0.49), but because the fabrication of PZT thin films using sol-gel technique is quite matured to the level of direct device applications. After adjusting materials parameters of both phases, transducers comprising two types of active layers were assembled and tested for the evaluation of UT performance in the characterization of coated facilities. By the impulse response characterization and direct thickness measurement, the complex UT assembled in this study showed excellent properties capable of direct application in related fields.

Electroactive Electrodes, Main Production Methods and Metallurgical Importance: *Ozgenur Kahvecioglu*[1]; Servet Timur[1]; [1]Istanbul Technical University, Metallurgl. & Matls. Sci. & Engrg., Chmst. & Metall. Faculty, Maslak, Istanbul 34469 Turkey

In the Electrochemical Processes, a wide range of harsh operating conditions and corrosive chemical environments are encountered. This often makes materials specification expensive and time-consuming, requiring high-efficiency materials for the process. Electroactive electrodes are widely used in industry to protect structures against corrosion, and in applications such as the electrowinning of metals, chlor-alkali production and fuel cell technology. This type of electrodes, such as DSA®, solid oxide electrodes and metal dioxide electrodes, also show lower overpotentials and besides they are dimensionally stable, tough, ductile and available in different shapes and sizes.In this work the importance of electroactive electrodes in metallurgical applications is tried to be explained and also the main production methods of various types of such material are going to be emphasized.

The Design of a Web Site to Deliver an On-Line, Introductory Course in Materials Science: Janet Alvarado[1]; Patrick Ray[1]; Brian Marks[1]; Mike Fleck[1]; Ralph H. Locklin[2]; Eric J. Spielvogel[3]; Andy J. Wiesner[2]; *Paul R. Howell*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 231 Steidle Bldg., Univ. Park, PA 16802 USA; [2]Pennsylvania State University, Schreyer Inst. for Excellence in Teaching, Rider Bldg. II, Univ. Park, PA 16802 USA; [3]Pennsylvania State University, Coll. of Earth & Minl. Scis., E-Education Inst., Univ. Park, PA 16802 USA

An introductory course in Materials Science and Engineering, Materials in Today's World, has been completely restructured from a conventional lecture-base course, into an E-Education Course. The new course may be taught completely on-line, or in a hybrid on-line/in-class environment. The course may be accessed through the Penn State delivery system, ANGEL, or using a generally available, specially created website. This web site, together with the course textbook, Earth, Air, Fire and Water: Elements of Materials Science, and the quizzing tool Perception, represents a complete course offering. In the current presentation, we shall describe the development of the web site, present our overall philosophy for the course design, and outline our assessment protocols. We shall also present examples of videos, and flash movies/animations, which are designed to replace the lectures of a conventional lecture course. Finally, we shall present preliminary student assessments from the previous two semesters.

The Writing, and Use of "Camera-Ready Review Articles" as a Means of Instruction and Assessment in an Introductory Materials Science and Engineering Course: *Paul R. Howell*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 231 Steidle Bldg., Univ. Park, PA 16802 USA

The author has developed an essay-based approach for teaching and evaluating Materials Science and Engineering. The thematic essays are called Camera-Ready Review Articles, because they are formatted like conference proceedings. Each essay deals with a single material, and each essay is constructed around the four elements of Materials and Science and Engineering: structure, properties, processing and performance. However, each essay also contains relevant historical information on the discovery and importance of that material. For example, in the essay on tungsten, the metal's role in the development of the incandescent light bulb, and the early electronic devices, is explored. Where appropriate, biographical information on persons who are associated with that material is also included in the essay. The author has developed a series of essays, which will be made available, but the presentation will concentrate on one particular material, Tungsten.

Neutron Diffraction Study of an Fe-Au Alloy: Ho-Gyu Kim[1]; Ian Swainson[2]; *Paul R. Howell*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 231 Steidle Bldg., Univ. Park, PA 16802 USA; [2]National Research Council of Canada, Neutron Prog. for Matls. Rsch., Bldg. 459, Sta. 18, Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada

A series of neutron diffraction studies, using a powder diffractometer at Chalk River National Laboratories, in Canada, have been conducted on a powder-metallurgy, Fe-5 wt %Au alloy. Real-time data on the interdiffusion kinetics of Au in Fe, and the precipitation behavior of Au in austenite during sintering have been obtained. In addition, the precipitation behavior of Au, at both the austenite/ferrite interface, and within the ferrite, during subsequent cooling, has been monitored. The large differences in atomic radius between Au and Fe produce large peak shifts during the diffusion of Au into both austenite and ferrite. Structural and chemical data, including lattice parameters, volume fractions, and the compositions of phases developed during sintering and subsequent cooling, have been obtained using structure refinement techniques.

Novel Deformation Processing of Amorphous Metal MEMS and Larger Structures: *Paul Wesseling*[1]; Ali Shamimi Nouri[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Matls. Sci. & Engrg., 10900 Euclid Ave., White Bldg., Rm. 401, Cleveland, OH 44106-7204 USA

Currently nearly all MEMS devices are silicon-based and made using batch fabrication techniques similar to those used for integrated circuits. The ability to use a deformation process to manufacture MEMS could greatly reduce production time and cost, increase the range of sizes and shapes possible, and improve the lifetime of the devices. The benefits of this deformation process can also be realized on larger scale systems not related to MEMS. Low and intermediate temperature deformation processing of MEMS can be accomplished using amorphous metals in place of silicon. The high strength (near theoretical values) and damage tolerance of such amorphous metals, combined with beneficial magnetic, electrical, and corrosion resistance properties, provides an attractive set of characteristics for MEMS devices, far superior to silicon-based systems. Initial results of deformation processes using Vitreloy I will be presented.

Understanding of the Formation Reasons of Residual Stress in Steam Generator Tubes: *SungSoo Kim*[1]; Joung Soo Kim[1]; [1]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. Dvlp., PO Box 105, Yousung-Ku, Taejon 305-353 Korea

In order to investigate the reason why the residual stress is developed in the steam generator tube systematically, the tubes were cooled both at inside and at outside tube after heat treatment and the residual stress formed during heat treatment were measured by strain gage using sectioning method. The inside-cooled tube was widened after spilt, and the residual stress at inside and outside tube appeared to be compressive and tensile, respectively. The component of the residual stress in the outside-cooled tube appeared reversely, and the magnitude of the residual stress was ~150MPa. This means that the residual stress of the steam generator tubes is originated to be the quenching process after heat treatment. The reason for this behavior seems to be due to the fact that the cooling effect at inside is more larger than that at outside because the tubes are surrounded each other as bundles during quenching.

Improving FRP Reinforced Concrete Beam With Shape Memory Superelastic Nitinol Wires: *Solmaz Torabi*[1]; [1]University of Texas, Matls. Sci. & Engrg. Dept., 325 Woolf Hall, Box 19031, Arlington, TX 76019 USA

Fiber reinforced polymer (FRP) composites are very interesting for civil engineering applications due to their high strength-to-weight and also their stiffness-to-weight ratios, corrosion resistance, light weight and potentially high durability. These characteristics make them a perfect alternative for reinforcing concrete beam instead of using steel reinforcement which has corrosion problem. However, the ductility of FRP is lower than steel (approximately two third). The present paper suggests using SMA superelastic wires inside FRP in order to improve the ductility. Superelasticity is one of the important mechanical properties of shape memory alloys (SMA) and their ability to recover 8% of their plastic deformation during unloading makes them superior to steels which usually have 0.2% recovery. This proposal suggests three different ways for applying SMAs' superelastic Nitinol to prevent FRP failure in tension and pull-out tests. The first way is using straight superelastic Nitinol wires inside FRP beams, the primary results showed improvement in tensile strength and ductility in comparison to usual FRP. The second way would be to install SMA wires in the shape of a spring around the FRP beam to close the cracks oriented in the direction perpendicular to the direction of tensile loading. And finally, the third method proposed in this research is testing different shapes of FRP other than beam in order to increase bonding strength. Results of this research and other studies have shown that it is not

feasible to use FRP in the same shape as the steel beams, used to reinforce concrete. As a result, a sheet of FRP is applied in a concrete with a helical shape and it is reinforced with SMA superelastic wires to improve its ductility. Moreover, SMA in FRP will induce some amount of compressive prestress in concrete and this can be counted as another benefit of using SMAs in FRP reinforced concrete beams.

Effects of MnO2 on the Piezoelectric Properties of PMN-PZT: *Sin-Wook You*[1]; Jong-Bum Park[1]; Kyoung-Won Cho[1]; Jae-Chang Kim[1]; Dong-Yeon Hwang[1]; Young-Min Kim[2]; Soon-Chul Ur[1]; Il-Ho Kim[1]; [1]Chungju National University, Matls. Sci. & Engrg., 123 Geomdan-ri, Iryu-myeon, Chungju, Chungbuk 380-702 Korea; [2]Corea Electronics Corporation, Matls. Dvlp., 168-16 Yongtan-dong, Chungju, Chungbuk 380-702 Korea

Perovskite Pb(Mg1/3Nb2/3)O3-Pb(Zr,Ti)O3 (PMN-PZT) was prepared and MnO2 doing effects on its piezoelectric properties were investigated. Grain size decreased with increasing the MnO2 content, and the pyrochlore phase was not identified in the sintered PMN-PZT with 0.0--1.0 wt% MnO2. Sintered density was changed from 7.63 to 7.74 g/cc with MnO2 content. Piezoelectric voltage and charge constants were reduced and mechanical quality factor increased with increasing the MnO2 content. This was related to increase in internal strain (stress) and suppression against domain rotation due to MnO2 addition. Electromechanical coupling factor slightly decreased with increasing the MnO2 content. Relative dielectric constant showed maximum value for 0.2 wt% MnO2-doped PMN-PZT, which was related to the domain wall pinning mechanism. This research was supported by the Program for the Training of Graduate Students in Regional Strategic Industries which was conducted by the Ministry of Commerce, Industry and Energy of the Korean Government.

In Situ Resistivity Measurements at the Initial Stage of FAST: George Aldica[2]; Vladimir Y. Kodash[1]; *Joanna R. Groza*[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]National Institute for Materials Physics, Bucharest-Magurele Romania

Newly developed sintering techniques employing application of electrical field (known as Field assisted sintering techniques or FAST) are currently intensively used to consolidate ceramic, metal and composite powders. These techniques combine a pulsed electrical current application with external pressure to activate powder densification. The pulsed current promotes electrical discharges at powder particle surfaces, thus activating them for subsequent bonding. The benefit of this activation is in the elimination of surface contaminants and a short densification time usually associated with minimal grain growth, particularly important in nanopowders. To characterize the response of a powder system to an applied pulse voltage at the initial stage of sintering, in-situ resistivity measurements were carried out. Voltage-current profiles were analyzed in Ni powder samples during the initial stage of sintering using a Spark Plasma Sintering machine. For some samples a phase shift on voltage-current patterns was observed. Possible nature of this shift is discussed. It is assumed that a powder system has a reactive component of electrical resistivity and may be considered as a sequence of connected resistors and capacitors.

The Effect of the Additive on the Properties of the $NiFe_2O_4$ Spinel: *Y. H. Liu*[1]; G. C. Yao[1]; J. H. Xi[1]; X. M. Zhang[1]; [1]Northeastern University, Sch. of Matls. & Metall., Shenyang 110004 China

In the process of preparation the ceramic material, there much of ceramic material is hard to sinter. We usually add some foreign substance, to drop the sintering temperature, promote sintered ingredients flowing and drawing up, and obtain the high quality product. The additives also have apparent effect on the microstructure and the properties of the sample sintered. Selecting by a series test, we particularly studied the influence of the MnO_2 and TiO_2 as the additives added into the Fe_2O_3, and NiO composition. The results show that these additives could upgrade the properties of the spinel sample and accelerate the rate of spinel sintering, for these oxides can dissolve in the $NiFe_2O_4$ and bring on lattice defect to promote sintering. In addition the Mn4+ and Ti4+ as quadrivalent ions once replace the Ni2+ or Fe3+, can they bring about anionic space bit, so as to can they increase the electric conductivity of the $NiFe_2O_4$ spinel sample.

The Structural and Physical Properties of TiOxNy Films with Controlled Nitrogen Partial Pressure: *Yong-Hwa Song*[1]; Chung-Kyung Jung[1]; In-Seob Bae[1]; Jin-Hyo Boo[1]; [1]Sungkyunkwan University, Dept. of Chmst. & Inst. of Basic Sci., Suwon, Kyounggi-do 440-746 Korea

We have deposited titanium oxynitride thin films on Si(100) substrates at 500°C using RF PECVD system. Titanium iso-propoxide was used as precursor with different nitrogen flow rate to control oxygen and nitrogen contents in the films. Changes of chemical states of constituent elements in the deposited films were examined by X-ray photoelectron spectroscopy analysis. With increasing nitrogen flow rate the total amounts of nitrogen and titanium were increased while that of oxygen was decreased. The XPS result also showed a binding energy shift toward high energy side with increasing nitrogen content. The film growth orientation and nanostructural characteristics were also analyzed by scanning electron microscopy as well as transmission electron microscopy. Deposition at higher nitrogen flow rate results in finer clusters with a nano-scale grain size and a slower growth rate. Nanoindentation experiments showed strong dependency on the composition and nanostructure in the hardness range of 10 and 16 GPa.

Effects of ZnO on the Piezoelectric Properties of PMS-PZT: *Young-Jin Son*[1]; Kyoung-Won Cho[1]; Dong-Yeon Hwang[1]; Jae-Chang Kim[1]; Young-Min Kim[2]; Il-Ho Kim[1]; Soon-Chul Ur[1]; [1]Chungju National University, Matls. Sci. & Engrg., 123 Geomdan-ri, Iryu-myeon, Chungju, Chungbuk 380-702 Korea; [2]Corea Electronics Corporation, Matls. Dept., 168-16 Yongtan-dong, Chungju, Chungbuk 380-250 Korea

Perovskite Pb(Mn1/3Sb2/3)O2-Pb(Zr,Ti)O3 (PMS-PZT) was prepared and ZnO doing effects on its piezoelectric properties were investigated. Pyrochlore phase was not identified in the PMS-PZT with 0-5 mol% ZnO sintered at 1373K for 2 hours, and maximum sintered density of 7.92 g/cc was obtained. Piezoelectric charge constant and voltage constant increased to 359x10^-12 C/N and 22.5x10^-13 Vm/N, respectively, with increasing ZnO addition. Mechanical quality factor reduced considerably with increasing ZnO content. When the ZnO content is 3 mol%, electromechanical coupling factor and relative dielectric constant showed maximum values of 56% and 1727, respectively. This should be evaluated by complicated variations of sintered density, tetragonality of lattice, grain size, and A-site vacancy generated by ZnO addition and Zn2+ substitution. This research was supported by the Program for the Training of Graduate Students in Regional Strategic Industries which was conducted by the Ministry of Commerce, Industry and Energy of the Korean Government.

Evaluation of Ultra-Thin Co-Based Barriers for Copper Metallization Grown by Self-Aligned Electroless Deposition: Giin Shan Chen[1]; Ying Sen Tang[1]; Sung Te Chen[2]; [1]Feng Chia University, Matls. Sci. & Engrg., 100 Wenhwa Rd., Seatwen, Taichung 407 Taiwan; [2]Tung Fang Institute of Technology, Electl. Engrg., 110, Tung Fang Rd., Hu-Nei Shang, Kaohsiung Co. 829 Taiwan

An electrochemically integrated seeding and plating deposition process is developed to deposit Co-based barriers for copper metallization on silicon-based dielectric films. The effects of the quantities of alloyed elements, particularly boron, phosphorus and tungsten, on the (1) developments of microstructure, phase transformation, and electrical properties due to annealing and (2) capability improvement of the barriers (Co-B, Co-W-B, etc.) are investigated. The barriers are highly resistive (greated than 1000 micro ohm-cm) in the as-deposited state, but become good conductors (~50 micro ohm-cm) following optimum (300-500°C) annealing. The aims of this work are to (1) grow ultrathin (~20 nm) and high-perforance electroless barriers and (2) to demonstrate the feasibility of the electrochemical-integrated process to fabricate the Cu gated, barrier capped MIS capacitor in a self-aligned manner. Finally, the physical properties and failure behavior of the barriers are also evaluated.

Microstructural Evolution of 5xxx and 7xxx Aluminum Wrought Alloys in the Partially Remelted Semisolid State: *Young-Ok Yoon*[1]; Young-Jig Kim[1]; Hoon Cho[2]; Shae K. Kim[2]; Hyung-Ho Jo[2]; [1]Sungkyunkwan University, Advd. Matls. Engrg., 300 Chunchun-Dong, Jangan-Gu, Suwon, Gyounggi-Do 440-746 S. Korea; [2]Korea Institute of Industrial Technology, Advd. Matls. Ctr., 994-32 dongchun-Dong, Yeonsu-Gu, Incheon 406-130 S. Korea

The microstructural evolution of 5xxx and 7xxx aluminum wrought alloys for the application of thixoextrusion has been investigated as a function of isothermal holding temperature and time in the partially remelted semisolid state. These alloys generally show low extrusion speed, low extrudability and rather high extrusion pressure index when extruded conventionally. It is expected that thixoextrusion could obtain a fine filling into the die with reduced extrusion pressure. The results show that the fraction of liquid and the average size of solid increased with increasing isothermal holding temperature and time. The interesting point of this study is that the transition to globular solid could be achieved without any semisolid billet pretreatment processes such as SIMA(Strain Induced Melt Activated) or RPR(Recrystallization & Partial Remelting).

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