TMS2006

135th Annual Meeting & Exhibition

■ *Linking science and technology for global solutions*

Meeting Information

March 12-16, 2006
Henry B. Gonzalez Convention Center
San Antonio, Texas, USA

Technical Program Follows Meeting Information

You are invited...

Help chart the technological course for future TMS annual meetings, as well as the materials science and engineering profession, by participating in a TMS technical committee. While attending TMS 2006 Annual Meeting & Exhibition, you are invited to attend the technical committee meeting that best serves your area of interest.

Technical Committee Schedule

Sunday, March 12

Pyrometallurgy Committee	10 to 11 a.m.	M	Conference Room 15
Recycling Committee	11 a.m. to noon	M	Conference Room 2
Waste Treatment & Minimization Committee	noon to 1 p.m.	M	Conference Room 16
Thin Films & Interfaces Committee	2 to 3 p.m.	M	Salon M
Process Fundamentals Committee	3 to 4 p.m.	M	Conference Room 15
Aluminum Committee	3 to 5 p.m.	M	Salon E
Copper, Nickel, Cobalt Committee	4 to 5 p.m.	M	Conference Room 5
Lead, Zinc Committee	4 to 5 p.m.	M	Conference Room 8
Nanomechanical Material Behavior Committee	4 to 6 p.m.	M	Conference Room 7
Magnesium Committee	4:30 to 6 p.m.	M	Conference Room 1
Aqueous Processing Committee	5 to 6 p.m.	M	Salon M
Precious Metals Committee	6 to 7 p.m.	M	Conference Room 4
Mechanical Behavior of Materials Committee	6:30 to 8 p.m.	M	Conference Room 16
Materials Characterization Committee	7 to 8 p.m.	M	Conference Room 2
Computational Materials Science & Engineering Committee	7:30 to 8:30 p.m.	M	Conference Room 10
Alloy Phases Committee	7:30 to 9 p.m.	M	Conference Room 8
Phase Transformations Committee	7:30 to 9:30 p.m.	M	Conference Room 5

Monday, March 13

Chemistry & Physics of Materials Committee	7:30 to 8:30 a.m.	M	Conference Room 8
Solidification Committee	12:30 to 1:30 p.m.	M	Conference Room 8
Process Modeling Analysis & Control Committee	12:30 to 2 p.m.	M	Conference Room 6
Advanced Characterization, Testing & Simulation Committee	5:30 to 6:30 p.m.	M	Conference Room 6
Surface Engineering Committee	5:30 to 6:30 p.m.	M	Conference Room 2
Nuclear Materials Committee	5:30 to 7 p.m.	M	Conference Room 10
Composite Materials Committee	5:45 to 7:15 p.m.	M	Conference Room 7
Biomaterials Committee	6 to 7 p.m.	M	Conference Room 11

Tuesday, March 14

Electronic Packaging & Interconnection Materials Committee	7 to 8 a.m.	M	Conference Room 10
Nanomaterials Committee	7:30 to 8:30 a.m.	M	Conference Room 7
Powder Materials Committee	12:15 to 1:45 p.m.	M	Conference Room 10
Reactive Metals Committee	5 to 6 p.m.	M	Conference Room 10
Refractory Metals & Materials Committee	5:30 to 6:35 p.m.	M	Conference Room 12
Shaping & Forming Committee	5:30 to 6:30 p.m.	M	Conference Room 4
Titanium Committee	6 to 7 p.m.	M	Conference Room 16
High Temperature Alloys Committee	6:30 to 8 p.m.	M	Conference Rooms 7

Institute of Metals/Mehl Lecture

"The Promise and Perils of Extreme Grain Refinement to Produce Superior Structural Materials"


Julia R. Weertman

Monday, March 13, 12:30 to 1:30 p.m., Henry B. Gonzalez Convention Center

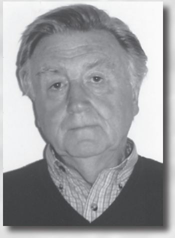
by Julia R. Weertman, *Department of Materials Science and Engineering, Northwestern University*

About the Topic
The ability to produce metals with very small grain sizes has led to materials with both the positive aspect of high strength and a number of negative attributes, especially brittle behavior. Attendees of this lecture are updated on the recent developments in the study of the mechanical properties of nanocrystalline metals and alloys, including attempts to make them into useful materials. Julia Weertman's research is sponsored by the Department of Energy Grant DE-FG02-02ER.

About the Speaker
Julia R. Weertman is the Walter P. Murphy Professor Emerita in Service at Northwestern University. At Northwestern for nearly 20 years, she holds three patents and has authored more than 150 technical publications. Weertman has received many professional honors, and is a member of the NRC National Materials Advisory Board.

Hume-Rothery Award Lecture

"Entropies of Formation and Mixing in Alloys"


W. Alan Oates

Monday, March 13
Henry B. Gonzalez Convention Center
Room 202A, Level 2

by W. Alan Oates, *University of Salford, UK*

About the Topic
Attendees learn about:

- Examples in which entropy plays a major role in determining the relative stability of phases in a system at high temperatures
- Methods used for estimating the magnitude of the contributions to formation/mixing entropies
- Models of value in the calculation of formation/mixing entropies for real multi-component alloys and of value in the calculation of phase diagrams for multi-component, multi-phase systems
- The value of the cluster/site approximation for describing the configurational contributions in multi-component, multi-phase systems, and recent developments in its application
- Methods suitable for the estimation of the magnitude of other contributions to formation/mixing entropies

About the Speaker
W. Alan Oates is the Honorary Visiting Professor at the Institute for Materials at the University of Salford in the United Kingdom. A Fellow at the Institute of Metals, Mining and Materials in London since 1978, he earned his doctorate from The University of Newcastle in Australia. Oates' interests recently are in developing a higher order approximation which is suitable for multi-component alloys and in the thermodynamic modeling of intermetallic compounds.

Extraction & Processing Division Luncheon Lecture

"China's Growing Importance in the Metals Field With an Emphasis on Alloying Additions for the Aluminum Industry"


Albert Hayoun

Tuesday, March 14, Noon to 1:45 p.m.
Henry B. Gonzalez Convention Center
Ballroom C3, Level 3
Luncheon tickets may be purchased at the meeting registration desk.

by Albert Hayoun
President of Standard Resources Corporation

About the Topic
This presentation relates the history leading to China's ascendance to the important position it now holds in the metals market and traces its recent history as a supplier and consumer of alloying additions, such as silicon metal and magnesium metal, in the aluminum industry.

About the Speaker
Albert Hayoun is president of Standard Resources Corporation, a marketing firm specializing in metals, minerals and alloys. He assisted in establishing the company in 1994 and has helped develop relations with a number of mining and metallurgical companies in China. Hayoun has also established agencies, companies and subsidiaries in several countries including China, Mexico and Venezuela. His work over the past 33 years has involved importing and exporting castings, forgings, pig iron, ferro alloys, metals, minerals and alloys. He began his career in the metallurgical field in 1973 after receiving a bachelor's degree from Brooklyn College.

Honor Your Colleagues by Attending These Special Events

Extraction & Processing Division Distinguished Lecture

"Extractive Metallurgy Principles Applied to the Synthesis of Value-Added Materials, Waste Minimization and Recycling"

Patrick R. Taylor

Tuesday, March 14, 1:45 to 2:30 p.m.
Henry B. Gonzalez Convention Center
Ballroom C2, Level 3
Luncheon tickets may be purchased at the meeting registration desk.

by Patrick R. Taylor, *Colorado School of Mines*

About the Topic
Attendees benefit from Patrick Taylor's overview of extractive metallurgy techniques and principles, which are essential tools when addressing technology for the synthesis of value-added materials, waste minimization and recycling. Various laboratory scale experiments are described, illustrating applications of pyrometallurgy, hydrometallurgy and electrometallurgy to these resource recovery opportunities.

- Thermal plasma synthesis of ultra-fine ceramic powders from minerals
- Reactive thermal plasma spraying of specialty coatings
- Closed-top cyclone treatment of radioactive wastes
- High-temperature oxide electro-reduction and metal recovery from residues through leaching

About the Speaker
Patrick R. Taylor is the George S. Ansell Chair Distinguished Professor of Chemical Metallurgy, and director of the Kroll Institute for Extractive Metallurgy, at the Colorado School of Mines. His research expertise includes mineral processing, extractive metallurgy, chemical processing of materials, thermal plasma processing, recycle and waste minimization. He holds seven patents and has published more than 135 papers. Taylor is a registered Professional Engineer with a doctorate in metallurgical engineering. He has been an active member of TMS for more than 30 years and is currently a member of the Process Fundamentals Committee.

Light Metals Division Luncheon Lecture

"Design Drives Consumption: The Revolution in Metal Packaging Design"

Edward B. Martin

Wednesday, March 15
Noon to 2 p.m.
Henry B. Gonzalez Convention Center
Ballroom C3, Level 3
Luncheon tickets may be purchased at the meeting registration desk.

by Edward B. Martin, *CCL Container*

About the Topic
Attendees learn about:

- New innovative designs driving consumer products companies to utilize better metal packaging
- How and why rigid aluminum bottle packaging has displaced glass and plastic packaging in segments of the beverage industry and other markets
- A market that has traditionally utilized metal packaging but now is using new designs and shapes to drive industry growth

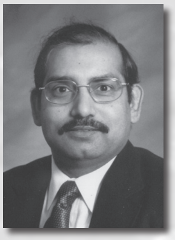
The common thread weaving through this presentation is that the world is evolving, and the end-use consumer will pay more for relevant value.

About the Speaker
Edward B. Martin is the vice president of sales and marketing for CCL Container. He has helped to diversify the company's business mix over the past seven years while continuing to grow its core aluminum aerosol container business. Martin has worked in the packaging industry for more than 20 years with experience in manufacturing management and planning in addition to sales and marketing. Prior to joining CCL Container, he was the national accounts manager for Tenneco Packaging Paperboard Pkg. Division.

Martin serves on the board of directors of both the Consumer Specialty Products Association (CSPA) and the National Aerosol Association (NAA). He is also a member of the Rochester Institute of Technology's (RIT) School of Packaging Science Industry Advisory Board. Martin holds a master's degree in international business from the University of Connecticut.

135th TMS & AIME Dinner and Awards Presentation
With Installation of 2006 TMS President

Tuesday, March 14, San Antonio Marriott Rivercenter Hotel • 6 p.m. Cash Bar; 7 p.m. Dinner
Tickets are $65 and may be purchased at the meeting registration desk.



Brajendra Mishra
2006 TMS President

Tresa M. Pollock
2005 TMS President

This annual, time-honored event includes recognition of the 2006 society and technical division award recipients followed by an address from 2005 TMS President Tresa M. Pollock. She also introduces the new president, Brajendra Mishra.

Brajendra Mishra is professor and associate director at the Colorado School of Mines Kroll Institute for Extractive Metallurgy. A TMS member for 18 years, Mishra is an accomplished educator and researcher in the areas of extraction and processing of materials, thin films processing, and corrosion engineering. Mishra received his doctorate from the University of Minnesota, Minneapolis in 1986.

He has held leadership positions on TMS committees in both the Light Metals Division and the Extraction & Processing Division (EPD). In addition, he has served as treasurer and chair of EPD and as a member of the Publications Coordinating Committee. Mishra played a key role in enhancing the TMS international membership through dialogues with the executives of the Indian Institute of Metals. He joined TMS as a student in 1982 and has remained an active member.

AIME AWARDS

AIME HONORARY MEMBER • **Merton C. Flemings,** *Massachusetts Institute of Technology*

AIME MINERAL ECONOMICS AWARD • **Chris Twigge-Molecey,** *Hatch*

AIME ROBERT EARLL MCCONNELL AWARD • **Don J. Glenister,** *Alcoa Inc.*

SOCIETY AWARDS

TMS FELLOW CLASS OF 2006
Diran Apelian, *Worcester Polytechnic Institute*
Clyde L. Briant, *Brown University*
Doris Kuhlmann-Wilsdorf, *University of Virginia*

APPLICATION TO PRACTICE
Vinod Kumar Sikka, *Oak Ridge National Laboratory*

JOHN BARDEEN AWARD
Isamu Akasaki, *Meijo University*

BRUCE CHALMERS AWARD
Diran Apelian, *Worcester Polytechnic Institute*

EDUCATOR AWARD
John P. Hager, *Colorado School of Mines*

ROBERT LANSING HARDY AWARD
Mark C. Hersam, *Northwestern University*

WILLIAM HUME-ROTHERY AWARD
William Alan Oates, *University of Salford*

INSTITUTE OF METALS/
ROBERT FRANKLIN MEHL AWARD
Julia R. Weertman, *Northwestern University*

LEADERSHIP AWARD
Toni Grobstein Marechaux
Strategic Analysis Inc.

CHAMPION H. MATHEWSON AWARD
K.S. Ravi Chandran, *University of Utah*

TMS FOUNDATION SHRI RAM ARORA AWARD
Krishau Biswas, *Indian Institute of Science*

TECHNICAL DIVISION AWARDS

EXTRACTION & PROCESSING DISTINGUISHED LECTURER
Patrick R. Taylor, *Colorado School of Mines*

EXTRACTION & PROCESSING SCIENCE AWARD
Gamini Senanayake, *Murdoch University*

EXTRACTION & PROCESSING TECHNOLOGY AWARD
Michelle G. Lee, *MSE Technology Applications Inc.*
Jay McCloskey, *MSE Technology Applications Inc.*
Jennifer Saran, *Kennecott Copper*
Larry G. Twidwell, *University of Montana*

LIGHT METALS DISTINGUISHED SERVICE AWARD
John Hryn, *Argonne National Laboratory*
Howard J. Kaplan, *US Magnesium*

LIGHT METALS TECHNOLOGY AWARD
Jan Van Linden, *Recycling Technology Services*

LIGHT METALS AWARD
Gary P. Tarcy, *Alcoa Inc.*
Knut Torklep, *Elkem Aluminium*

STRUCTURAL MATERIALS
DISTINGUISHED SERVICE AWARD
Russell H. Jones, *Pacific Northwest National Laboratory*

STRUCTURAL MATERIALS DISTINGUISHED SCIENTIST/
ENGINEER AWARD
Edgar A. Starke Jr., *University of Virginia*

Materials Library Lecture & Exhibit

Tuesday, March 14, 12:30 to 1:30 p.m.
Exhibit Hall, Booth #501

by Mark A. Miodownik
King's College London

"We are becoming more and more theoretical; we are losing touch with the more sensual side of what we do," so says Mark Miodownik, the curator of the Materials Library at King's College London, but the university lecturer is trying to change that.

Join us as we bring materials science to life through Dr. Miodownik's materials library exhibit and his presentation about the development and uses of materials libraries. More than 300 new materials, including aerogel, magnetic liquid and artificial skin, will be within reach at this interactive, tactile, aesthetic display!

About the Speaker
Mark A. Miodownik is a materials scientist and NESTA Fellow at King's College London, as well as the curator of the Materials Library there. He works in the department of Mechanical Engineering in the school of Physical Sciences and Engineering. In 2005 Miodownik organized and chaired a seminar series at the Tate Modern Museum in London on the influence of new materials on the arts. He is engaged in several collaborative art/science projects including a NESTA project to build a new materials library for artists and designers, and organizes "EngineeringArt," a network dedicated to the art and science of materials. Miodownik has published 37 research papers and writes a regular column on the arts and science of materials for the journal *Materials Today*. He earned his doctorate in turbine jet engine alloys from Oxford University in 1996.

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Attendees registered in the Nonmember Author and Nonmember categories receive a one-year, complimentary associate membership to TMS for 2006!

As an associate member, you will have access to the technical information and professional network you need to advance your work:

- Free print and electronic subscription to *JOM*, a monthly technical journal covering varied subjects important to the minerals, metals and materials world
- Access to TMS E-Library, with online databases, engineering reference books and analytical tools, powered by Knovel
- Free subscription to *TMS Letters*, the online technical journal providing updates to cutting-edge research in a concise format.
- Discounts on additional TMS publications, including archival technical journals and proceedings
- Reduced registration fees for TMS meetings
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Your membership card and new member packet will be on their way to your mailbox following the annual meeting, but you may begin taking advantage of your membership once your annual meeting registration fee is received and processed.

For additional information about activating your membership, contact TMS Member Services at (800) 759-4TMS or (724) 776-9000, ext. 259.

TMS

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The Minerals, Metals & Materials Society (TMS) is the professional organization encompassing the entire range of materials in science and engineering, from minerals processing and primary metals production to basic research and the advanced applications of materials. The Society's broad technical focus covers light metals; electronic, magnetic and photonic materials; extraction and processing; structural materials; and materials processing and manufacturing.

Our Members

Included among TMS professional and student members are metallurgical and materials engineers, scientists, researchers, educators and administrators who work in industry, government and academia. They hail from more than 70 countries on six continents.

Our Mission

The mission of TMS is to promote the global science and engineering professions concerned with minerals, metals and materials. The Society works to accomplish its mission by providing technical learning and networking opportunities through interdisciplinary and specialty meetings; short courses; publications, including five journals and proceedings; and its Web site.

To learn more, visit www.tms.org.

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TMS2006

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Technical Program

March 12-16, 2006
Henry B. Gonzalez Convention Center
San Antonio, Texas, USA

TUESDAY

2006 Nanomaterials: Materials and Processing for Functional Applications: Nanoscale Electronics

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: W. Jud Ready, GTRI-EOEML; Seung Hyuk Kang, Agere Systems

Tuesday AM Room: 214C
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Seung Hyuk Kang, Agere Systems; W. Jud Ready, GTRI-EOEML

8:30 AM Introductory Comments

8:35 AM Invited
Performance Modeling for Metallic Single Wall Carbon Nanotube Interconnects for Gigascale Integration (GSI): *Azad Naeemi*[1]; James D. Meindl[1]; [1]Georgia Institute of Technology
In this presentation, after reviewing the circuit models for single wall carbon nanotubes (SWCN), their potential application as interconnects in gigascale chips is evaluated. The results offer important guidance regarding the nature of carbon nanotube technology development needed for improving interconnect performance. For short local interconnects whose resistances are dominated by their drivers, monolayer nanotube interconnects offer latencies smaller than those of their copper counterparts due to significantly smaller lateral capacitances. Significant decreases in power dissipation, crosstalk, and dynamic delay variation are other advantages of replacing copper wires with monolayer nanotube interconnects. For longer lengths, however, bundles of densely packed single wall nanotubes are needed to outperform copper interconnects in terms of resistance and to adequately improve the wave propagation speed. The impact of electron-phonon scattering on the performance of carbon nanotube interconnects is demonstrated to be negligible. This is in sharp contrast with the transistor application of carbon nanotubes.

9:00 AM Invited
Controlled Positioning of Nanoparticles in a Wafer-Level: *Seong Jin Koh*[1]; [1]University of Texas, Arlington
Precise positioning of nanoscale objects (such as nanoparticles, carbon nanotubes, nanowires, DNAs, proteins, etc) onto exact substrate locations is one of the key requirements for the realization of nanoscale functional devices. Combining wet chemistry and CMOS fabrication technology, we have developed a technique which enables wafer-level positioning of nanoparticles with nanoscale precision. In this approach, self-assembled monolayers (SAMs) of organic molecules are formed on selected areas of a lithographically patterned silicon wafer. Gold nanoparticles in the range of 20-250 nm selectively attach to the areas functionalized with SAMs, enabling one-dimensional alignment of nanoparticles along the predefined patterns. This technique can be applied to the alignment of other nanoscale objects in general. The application of this technique to the fabrication of single electron devices and carbon nanotube devices will be discussed. Supported by ONR (N00014-05-1-0030) and NSF CAREER (ECS-0449958).

9:25 AM Break

9:35 AM
Fabrication and Characterization of Novel InGaN/GaN Multiple Quantum Disk Nanocolumn Light Emitting Diodes: *Akihiko Kikuchi*[1]; Makoto Tada[1]; Katsumi Kishino[1]; [1]Sophia University
Novel device structure feasible for current injection into nanocolumns (nanorods) is presented. GaN nanocolumns; self-organized columnar nanocrystals grown normal to substrate with diameter of 20~200nm, have superior optical quality due to dislocation-free nature. But only a few reports on LED application have been reported, because formation of electrical contact was difficult. In this study we give a novel technique to solve this problem. Following to the growth of n-GaN and InGaN/GaN multiple quantum disk nanocolumns on n-Si(111), p-GaN nanocolumns were grown with gradually increasing their diameter and finally connected to neighboring nanocolumns. Consequently, the nanocolumn device had a continuous surface without chasms. This novel structure enables to make electrodes on the top of nanocolumn devices. The room temperature bright electro-luminescence of the InGaN/GaN nanocolumn LEDs from violet to red was successfully demonstrated. This technique is attractive to realize practical nano-crystal devices and applicable to various nanocolumn materials such as ZnO.

9:55 AM
Structural Characterization of InAs/GaAs Quantum Dot Molecules: Coupling and Strain Effects: *Anup Pancholi*[1]; Valeria G. Stoleru[1]; [1]University of Delaware
The growth of quantum dot molecules, defined by a pair of electronically coupled quantum dots, represents a major step in tailoring the electronic properties of nanostructures. Besides great flexibility, the vertical coupling between the dots allows one to shift optical transitions to a specific spectral range, such as the Terahertz regime. Drastic modifications of the basic features of single layer dot assemblies occur as soon as the thickness of the GaAs barrier layer is reduced to a few nanometers, so as to induce strong coupling between vertically adjacent dots. The strain distribution in the InAs/GaAs quantum dot molecule is calculated by taking into account the material intermixing, as well as the shape and the size of the dots, as obtained from cross-sectional high-resolution transmission electron microscopy. The influence of the coupling strength and the strain effects on the properties of quantum dot molecule are further analyzed.

10:15 AM
Characterization of Conductive Inks Deposited with Maskless Mesoscale Material Deposition (M3D): *Jacob M. Colvin*[1]; Michael Carter[1]; Jan A. Puszynski[1]; James W. Sears[1]; [1]South Dakota School of Mines and Technology
Direct Write Technologies are being utilized in antennas, engineered structures, sensors, and tissue engineering. One form of Direct Write Technology is Maskless Mesoscale Material Deposition (M3D). The M3D process is a Direct Write Technology that uses aerosol formation, transport and deposition. Conductive inks for the M3D process utilize metal nanoparticles in suspension for deposition. Several different conductive inks were deposited with the M3D process and the deposits characterized for electrical resistivity and microstructure. Physical properties of the deposited inks are also measured and reported. This paper will report on the results obtained after sintering conductive nano-particle inks. Sintering was performed with a 2W frequency doubled Nd:YAG CW laser and a conventional muffle furnace. Sample thickness, microstructural details and sintering characteristics are also examined for the depositions of various conductive ink.

10:35 AM Break

10:45 AM
Modeling of Dielectric Nanocomposites: *Thomas K. H. Starke*[1]; Clair Hincliffe[1]; Colin Johnston[1]; Peter Dobson[1]; Patrick S. Grant[1]; [1]Oxford University
Polymer-ceramic composites are gaining in popularity as candidate materials for lightweight dielectric films in capacitor applications. A large number of theoretical studies have sought to express the relationship between the volume fraction of the ceramic materials and the resulting effective properties of the composite film. Due to their strong van der Waals and electrostatic forces, the behavior of nanosized ceramic particles in these films may be markedly different from larger micron-sized counterparts. With a decrease in diameter particle clustering increases significantly giving rise to morphologies that cannot easily be described by the widely used classical effective medium theories. We propose a model for the dielectric constant of nanocomposites and compare resulting predic-

tions with experimental results from the literature, in-house measurements as well as standard EMT formulations. The physical principles in practical nanoscale systems will be discussed in orderto guide further experimental approaches in the field.

11:05 AM
Nanomaterials in Novel Circuit Production Methods: *Alan Rae*[1]; [1]Nanodynamics Inc

As nanomaterials become more available the number of novel deposition techniques is increasing to exploit their unique processing characteristics such as low temperature reactivity and self-assembly. This paper outlines developments in three application areas for nanomaterials; materials in inkjet circuit development; thermal transfer of novel silver-palladium internal electrode material for ceramic capacitors; and atomic cluster deposition to prepare lithographically defined nanowires for sensor and other applications.

11:25 AM
Characterization of Diffusion Barrier in Nanometer Range Cu Interconnects: *Dongmei Meng*[1]; Choong-Un Kim[1]; Nancy Michael[1]; [1]University of Texas, Arlington

With the miniaturization of Cu interconnects integrated with low-k dielectrics, the thickness of the diffusion barrier must be just a few nanometers to maintain the advantage of the low resistance of Cu, yet it has to be good enough to prevent Cu diffusion from the Cu trenches. Extensive studies are being conducted to enable the nano-scale barrier, however, the progress has been slow due to the lack of an effective characterization method. In this paper, a new methodology based on conventional two-electrode electrochemical cell is developed to evaluate the quality of the barrier, in which two interconnects are used as electrodes and electrolyte is infiltrated into the low-k forming the electrochemical cell. The method is found to be extremely sensitive to defects in the barrier, with the potential to be extended to evaluation of porosity and pore distribution in the low-k materials by measuring diffusivity of the electrolyte.

11:45 AM Break

11:55 AM
Nonlinear Optical Dynamics of Glass-Embedded Silver Nanoparticles: *Sergiy Lysenko*[1]; Jose A. Jimenez[1]; Guangjun Zhang[1]; Huimin Liu[1]; [1]University of Puerto Rico

The nonequilibrium carrier dynamics in Ag nanoparticles (NP) was explored by femtosecond optical pump-probe spectroscopy. Metal-dielectric nanocomposite materials with different sizes of Ag NP were prepared by thermal treatment of aluminophosphate glasses. Laser-induced nonlinear optical response of NP was observed in transient reflection and degenerate-four-wave-mixing measurements. The third-order susceptibility is observed in a nonlinear holographic experiment. The ultrafast relaxation dynamics of electron-phonon coupling on the 10^{-13}-10^{-11} sec temporal-scale shows size- and pump power-dependent properties. Dependencies of electron-phonon coupling rate and coherent acoustic oscillations on NP size will be discussed in terms of a two-temperature model and stress generation by optical absorption. Kinetics study suggests that the nonequlibrium optical excitation of the NP ensemble leads to coherent acoustic oscillations of NP with different eigenfrequencies in the nanocomposite. Complete relaxation of nanocomposite materials occurs on a ~10^{-9}sec time scale and can be assigned to phonon system thermalization.

3-Dimensional Materials Science: X-Ray Methods

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Jeff P. Simmons, U.S. Air Force; Michael D. Uchic, Air Force Research Laboratory; Dorte Juul Jensen, Riso National Laboratory; David N. Seidman, Northwestern University; Anthony D. Rollett, Carnegie Mellon University

Tuesday AM Room: 205
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Dorte Juul Jensen, Riso National Laboratory; Ulrich Lienert, Argonne National Laboratory

8:30 AM Invited
3D Synchrotron Imaging Techniques: Current State and Perspectives: *Wolfgang Ludwig*[1]; [1]INSA Lyon

State of the art synchrotron imaging techniques are powerful tools for the non-destructive characterizion of 3D microstructures, applicable to a variety of engineering materials. In particular X-ray microtomography, conventionally used in absorption mode, has seen major improvements and offers now complementary contrast mechnisms exploiting either the coherence of 3rd generation synchrotron beams (phase contrast) or the extinction contrast in crystalline materials (diffraction contrast tomography). In terms of spatial resolution this technique covers the range from 10 down to 0.1 micrometers and consequently allows to address a variety of problems in materials science. The possibilities of synchrotron X-ray microtomography will be presented by discussing selected examples of recent applications, including in-situ fatigue crack propagation and 3D grain mapping experiments in Al alloys.

8:55 AM
3D Characterization of Titanium Alloys Using Phase-Contrast Tomography: *Erik M. Lauridsen*[1]; Richard W. Fonda[2]; Wolfgang Ludwig[3]; George Spanos[2]; [1]Center for Fundamental Research: Metalstructures in Four Dimensions; [2]U.S. Naval Research Laboratory; [3]European Synchrotron Radiation Facility

Knowledge of the 3-dimensional spatial arrangement and morphology in multi-phase materials are essential for a full understanding of the processes taking place during thermal-mechanical processing. As a result 3D characterization techniques plays an increasingly important role in material science. This presentation will focus on some recent results utilising phase-contrast tomography to reveal the detailed 3D microstructure of alpha and beta phase in two different titanium alloys with a spatial resolution of ~1 micron. The differences in microstructure between the two alloys are investigated both qualitatively by 3D renderings of the morphologies and quantitatively using relevant 3D microstructural descriptors.

9:15 AM
3D Grainmaps from X-Ray Diffraction Data with the Algebraic Reconstruction Technique: *Erik Knudsen*[1]; Henning F. Poulsen[1]; [1]Risø National Laboratory

In this paper we explore methods for generating 3-dimensional maps of the crystallographic structure of monophase materials from x-ray diffraction data. In particular, we focus on applying the algebraic reconstruction technique (ART), to data obtained by 3-dimensional x-ray diffraction microscopy (3DXRD). Although originally designed for applications in medical imaging, ART is a general algorithm for producing solution estimates to underdetermined systems of equations. The non-destructive experimental procedure of 3DXRD, provides a unique ability to study grain morphology in-situ. It also poses a challenge for data-analysis tools to process the vast amounts of data associated with high-resolution 3d maps on minute-scale time resolution. We present result indicating that the ART-formalism is a practical, efficient and reliable procedure for producing high-resolution grain maps. Furthermore, we show that the framework also holds exciting prospects for furthering the analysis beyond grain boundary mapping.

9:35 AM
4D Measurements of Grain Growth: *Soeren Schmidt*[1]; Dorte Juul Jensen[1]; [1]Center for Fundamental Research: Metal Structures in Four Dimensions

Data showing the simultaneous evolution of hundreds of grains during grain growth in AlMn is presented. Before annealing and following each annealing step a cylindrical volume with a diameter of 700 microns and height of 350 microns was fully characterized, i.e. the morphology as well as the crystallographic orientation was determined for each grain in the volume. The measurements were collected non-destructively utilizing the Three Dimensional X-ray Diffration microscope (3DXRD). After 5 annealing steps few tens of grains were left. The talk will also present the reconstruction algorithm applied to this data and the potential of the method is discussed.

9:55 AM
3 Dimensional Characterization of Inhomogeneous Plastic Deformation during Friction Stir Processing via Polychromatic Microdiffraction: *Rozaliya I. Barabash*[1]; Gene E. Ice[1]; Wenjun Liu[1]; Oleg M. Barabash[1]; Z. Feng[1]; S. A. David[1]; [1]Oak Ridge National Laboratory

Plastic deformation and structural changes of the Ti surface after Friction Stir Processing (FSP) were analyzed by means of SEM, EBSD and advanced 3D polychromatic X-ray micro diffraction at the synchrotron. Spatially resolved 3D Laue diffraction allowed understanding the changes in dislocation arrangement with depth in different regions of the FSP Ti. Formation of two specific zones was established: friction stir zone (FSZ), with the size of 300 microns, and thermal mechanical affected zone (TMAZ) with the size of 800 microns. It was shown that FSP generates a large number of dislocations. Maximal dislocation density is located within the TMAZ. Dislocation density gradually decreases and reaches the value typical for base metal. Within the TMAZ dislocations are distributed inhomogeneously. Inhomogeneity of plastic deformation and dislocations arrangement is found in 3D both within the individual grains and between separate grains.

10:15 AM Break

10:35 AM Invited
3-Dimensional Characterization of Polycrystalline Materials Using High Energy Synchrotron Radiation at the APS: *Ulrich Lienert*[1]; Jon Almer[1]; Dean Haeffner[1]; Bo Jakobsen[2]; Wolfgang Pantleon[2]; Henning Friis Poulsen[2]; Daniel Hennessy[3]; Changshi Xiao[3]; Robert Suter[3]; [1]Argonne National Laboratory; [2]Risø National Laboratory; [3]Carnegie Mellon University

The use of high-energy synchrotron x-rays provides a unique combination of bulk penetration power with high spatial and temporal resolution. Their potential to characterize polycrystalline materials under thermomechanical processing has recently been demonstrated. The Advanced Photon Source (APS) at the Argonne National Laboratory is one of the three existing high energy, 'third-generation' synchrotron facilities worldwide where the described experiments can be performed. A dedicated experimental station is currently under development at the APS 1-ID beamline. The beamline status and preliminary work will be presented. Case studies include 3-dimensional strain/texture mapping, the mapping of the orientation and boundary topology of individual grains, and the evolution of dislocation structure during plastic deformation by measuring diffraction peaks from grains and subgrains with high reciprocal space resolution.

11:00 AM
Characterization of Dislocation Structures during Deformation of Polycrystals by 3DXRD: *Rozaliya I. Barabash*[1]; H. F. Poulsen[2]; U. Lienert[3]; W. Pantleon[2]; [1]Oak Ridge National Laboratory; [2]Riso National Laboratory; [3]Argonne National Laboratory

As supplement to a 3DXRD setup at beam line 1-ID at APS, the possibility for measuring individual diffraction peaks with high resolution has been established. Several reflections of an individual grain in the bulk of a polycrystalline specimen have been recorded during in-situ tensile loading. The diffraction peaks have been analyzed and related to the deformation microstructure. From the spread perpendicular to the scattering vector, the orientation distribution function of the grain is inferred and information on the non-redundant dislocations is obtained. Additionally, asymmetries in the radial profile (along the scattering vector) are interpreted in terms of internal stresses and strains developing in the dislocation boundary structure.

11:20 AM
A System for Measuring Lattice Strain Pole Figures Using Synchrotron X-Rays: *Matthew Miller*[1]; Joel Bernier[1]; Jun-Sang Park[1]; Alexander Kazimirov[1]; [1]Cornell University

This talk describes a system for mechanically loading test specimens in situ at the A2 experimental station at the Cornell High Energy Synchrotron Source (CHESS) for the determination of lattice strains and their evolution in multiphase alloys via powder diffraction. Relatively thin (0.5mm) iron/copper specimens were axially strained using an electromechanical load frame beyond the macroscopic yield strength of the material. The loading was halted at multiple points during the deformation to conduct a diffraction experiment using a 50 keV. Entire Debye rings of data were collected for multiple {hkl}s in both copper and iron using a MAR345 online image plate detector. Strain Pole Figures (SPFs) were constructed by rotating the loading frame about the specimen transverse direction (TD). The CHESS data were validated using in situ data from neutron diffraction experiments. Use of the 3 dimensional SPF data as a material model validation tool is discussed.

11:40 AM
Micro-Laue Diffraction Study of Fatigue Crack Wake Plasticity: *Vipul K. Gupta*[1]; Yun Jo Ro[1]; Judy WL Pang[2]; Richard P. Gangloff[1]; Sean R. Agnew[1]; [1]University of Virginia; [2]Oak Ridge National Laboratory

Micro-Laue diffraction was used to investigate crack wake plasticity in aluminum alloy 2024 fatigue cracked in vacuum and saturated water vapor environments. The experiments involved differential aperture depth resolved line scans and automatic indexing software was used to produce orientation image maps showing the grain morphology beneath the sample surface. Such images tomographically reveal the sub-surface crack as well. Crystal defects associated with plasticity were evidenced by streaking of the Laue diffraction peaks in the crack wake and the plastic damage was quantified in terms of peak width and the crystal misorientation within each measurement voxel. The measurements verify the theoretical expectation that the cyclic plastic zone size is a function of stress intensity range, rather than the maximum stress intensity (constant for all the measurements.) The effect of environment is more subtle, yet it appears that there is more localized plastic damage within the moist environment.

12:00 PM
Microtomography and 3-Dimensional Stresses of Compressed Low-Density Amorphous Metal Foam: *Jay C. Hanan*[1]; Jin Ma[1]; Chris Veazey[2]; Marios D. Demetrio[2]; Hongbing Lu[1]; Ersan Ustundag[3]; William L. Johnson[2]; [1]Oklahoma State University; [2]California Institute of Technology; [3]Iowa State University

Low density metallic foams of consistent controlled morphology offer unique material and mechanical properties, yet they have eluded traditional metallurgical engineering methods. New metallic foam, processed by thermo-plastic expansion, has recently been discovered and its mechanical properties characterized using microtomography combined with in-situ compression. The new foaming process enables porosities unachievable through traditional metallurgical processes. The metal foams exhibit excellent morphological characteristics as evidenced by the microtomography analysis. The high porosities, exceeding 85%, contribute to enhanced plastic yielding. Microtomography of the amorphous metal foam during in-situ compression reveals the 3-dimentional deformation. The stress in all directions was modeled using the generalized interpolation material point method applied in continuous 3-D space and compared to the deformations observed in tomography. Localized regions of failure were observed allowing the foam to absorb significant energy during deformation. The deformation mechanisms for these foams are discussed.

7th Global Innovations Symposium: Trends in Materials R&D for Sensor Manufacturing Technologies: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Global Innovations Committee
Program Organizers: Hamish L. Fraser, Ohio State University; Iver E. Anderson, Iowa State University; John E. Smugeresky, Sandia National Laboratories

Tuesday AM Room: 204A
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Iver E. Anderson, Iowa State University

8:30 AM Invited
Controlled Growth of Oriented Nanostructures and Their Applications: *M. Meyyappan*[1]; [1]NASA Ames Research Center
Carbon nanotubes (CNTs) exhibit unique electronic properties for sensors applications. However, a lack of selectivity between metallic vs. semiconducting tubes has limited progress in nanoelectronics. Nevertheless, there has been much progress in sensor applications that use mulitwalled structures. We report here a carbon nanofiber (CNF) based nanoelectrode array for biosensor and neural interfacing applications. The CNTs are grown using PECVD. The gap between CNFs is filled with SiO2. This nanoelectrode array (NEA) has been successfully used for developing a biosensor and also as a neural interface for deep brain stimulation applications. Inorganic nanowires have been emerging as an adequate candidate in areas envisioned for application of CNTs, based on the publications we have seen in the literature in the last five years. Here, the growth is reported of well-aligned, vertical nanowires of ZnO, In2O3, silicon and germanium and their applications in electronics and optoelectronics.

9:00 AM Invited
Structural Iron-Based Magnetostrictive Alloys for Sensor Applications: *Thomas A. Lograsso*[1]; A. E. Clark[2]; M. Wun-Fogle[3]; J. Restorff[3]; [1]Iowa State University; [2]Clark Associates; [3]Naval Surface Warfare Center
$Fe1_{-x}Ga_x$ alloys offer relatively high magnetostriction along with combination of high mechanical strength, good ductility, low saturation fields, high blocking stress, and low cost. Substituting Ga for Fe in the α-Fe structure, increases the tetragonal magnetostriction, λ100, over 10-fold above that of pure α-Fe. Experimentally it has been shown that single crystalline 3/2λ001 increases monotonically with the Ga concentration to approximately 400 microstrain for x<0.19, decreases thereafter for 0.19<x<0.23, and increases again to the same level at x~ 0.29. Further because of its relatively good mechanical properties, built-in stress anisotropies allow for use in both compressive and tensile regimes, opening up device design space not allowed by other, brittle smart materials. An overview of the magnetic and mechanical properties will be presented.

9:25 AM Break

9:45 AM
Al-Sb Nanoelectrode Arrays for Radiation Detection: *Krishnan S. Raja*[1]; Manoranjan Misra[1]; Susant Mohapatra[1]; Thulasidharan Gandhi[1]; [1]University of Nevada, Reno
Stoichiometric single crystal Al-Sb compound semiconductor shows excellent electronic properties that can be tailored for radiation sensing applications. Al-Sb semiconductors can be used for detecting low-medium energy radiation at room temperature. These detectors will have higher resolution and lower noise levels than those of CdZnTe. Al-Sb crystals are manufactured, in general, by Bridgman technique. This investigation reports an electrochemical synthesis of stoichiometric Al-Sb semiconductor nanowires in non-aqueous solutions at room temperature. Suitable electrochemical window for deposition of stoichiometric Al-Sb nanowires were determined by conducting cyclic voltammetry. The nanowires were characterized by determining composition, electronic band gap, and electrical resistivity.

10:10 AM
Characterization of Piezoelectric Aluminum Nitride Thin Film by RF Magnetron Sputtering: Shih-Jeh Wu[1]; Ting-Wei Shen[1]; *Chen-Ming Kuo*[1]; [1]I-Shou University
Surface Acoustic Wave Filter devices have been the main stream of filters in last generation of communication industry under 2GHz. However, for the new specification of mobile telecomm the frequency requirement can be higher than 3GHz. Film Bulk Acoustic Resonator becomes the mainstream of duplexer and filter in communication industry. Aluminum nitride thin film is the best piezoelectric material for the above applications. It has hexagonal crystal structure and is a covalent bonded material. It has high dielectric constant, acoustic speed (high Young's modulus), melting point, energy band gap, good thermal insulation, and low thermal expansion. The piezoelectricity depends on good <002> crystaline structure. In this study, we use RF magnetron sputtering to deposit AlN thin film on different substrates and the film are characterized by several means e.g., SEM, TEM, XRD, and AFM. Also simple devices are fabricated and the piezoelectric properties are also well demonstrated by network analyzer.

Advanced Materials for Energy Conversion III: A Symposium in Honor of Drs. Gary Sandrock, Louis Schlapbach, and Seijirau Suda: Complex Hydrides I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada; John J. Petrovic, Petrovic and Associates; Renato G. Bautista, University of Nevada; M. Ashraf Imam, Naval Research Laboratory

Tuesday AM Room: 214B
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Craig Jensen, University of Hawaii; Carole Read, U.S. Department of Energy

8:30 AM Plenary
Monitoring of Defect Complexes Arising in the Dehydrogenation of Sodium Aluminium Hydride by Anelastic Spectroscopy: *Craig Jensen*[1]; Rosario Cantelli[2]; Oriele Palumbo[2]; Annalisa Paolone[2]; Sesha Srinivasan[1]; [1]University of Hawaii; [2]Universita di Roma
The mechanism of the reversible, solid-state dehydrogenation of Ti-doped NaAlH4 has been a topic of considerable recent interest. We have probed this phenomenon through anelastic spectroscopy and found that dehydrogenation of the hydride can be monitored through its effects on the elastic constants. We have also obtained evidence for the formation of highly mobile, stoichiometric defects in Na3AlH6 that arise at temperatures much lower temperatures in the Ti-doped samples. These species "jump" at a rate of 1000/s at 70 K corresponding to an activation energy of 0.126 eV and which is typical of point defect relaxation. The results of these studies will be presented and discussed in terms of a model in which this mobilized population of hydrogen plays a fundamental role in the dehydrogenation process that is kinetically enhanced by Ti acting to decrease its migration energy in the crystal lattice.

8:55 AM Invited
Mechanochemical Synthesis and First-Principles Simulations of Mg-Based Hydrogen Storage Materials: *Z. X. Guo*[1]; C. X. Shang[1]; Y. Song[1]; [1]Queen Mary, University of London
A systematic investigation of the structural stability, evolution and hydrogen-storage properties of Mg-based hydrides and nanostructures was carried out, involving experimental mechanical milling and chemical alloying, and electronic structural simulations of hydrogen-metal interactions. The effects of milling on particle size, lattice parameters, microstructure, and phase composition of the powder mixtures were characterised using SEM, X-Ray diffraction analyses. Mechanical milling was shown to be an effective method of refining the particle size, particularly when MgH2 is involved. The influences of the selected chemical elements, in-

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cluding transition metals, on hydrogen desorption of various milled mixtures were clearly identified using coupled Thermogravimetry (TG) and Differential Scanning Calorimetry (DSC). The as-received MgH2 shows an onset desorption temperature of 420°C. Mechanical milling reduces the onset temperature to 330°C. Chemical alloying, via surface catalysis and/or solid-solutioning, further increases the desorption kinetics and reduces the desorption temperature down to 250°C. The degree of such effect decreases from Ni, Al, Fe, Nb, Ti, to Cu. A multi-component mixture of (MgH2+Al+Ni+Y+Ce) exhibits relatively fast desorption kinetics and the lowest desorption temperature at about 200°C. Electronic structural simulations further clarify the effects of alloying elements on the stability and bonding of modified hydride systems. The coupled experimental and theoretical approach has laid a valuable foundation for continued development of new and cost-effective hydrogen storage systems with a high capacity, a low desorption temperature and rapid kinetics.

9:20 AM Invited
Preparation of LiH0.5D0.5 Using Isotopic Exchange of Lithium Hydride: *Joseph R. Wermer*[1]; Steven N. Paglieri[1]; Blake P. Nolen[1]; John T. Gill[1]; Jane Poths[1]; [1]Los Alamos National Laboratory

Lithium hydride (LiH) powder and compacts were exchanged with flowingdeuterium (2H2) at temperatures from 300–550ºC and pressures of 600–1400 mm Hg (torr) to obtain lithium-hydride deuteride (LiH0.5D0.5). The kinetics of the exchange reaction increased with temperature so that a complete exchange in powder (equimolar composition of hydrogen and deuterium) occurred after 0.5 h at 450ºC. Powder exchanged more quickly than partial density LiH compacts, as indicated by real-time analysis on a mass spectrometer connected to the system. The samples were assayed by amalgamating the Li(H,D) with tin and analyzing the released gases with high-resolution mass spectrometry. For example, the offgas from LiH powder exchanged with deuterium at 450ºC was composed of 48.9% hydrogen and 51.1% deuterium, indicating that the exchange with deuterium was complete. Analysis of Li(H,D) taken from the core of a sintered compact exchanged with deuterium at 350ºC for 24 hours indicated that essentially complete exchange had occurred (LiH0.52D0.48). Experiments are continuing with higher density solid compacts to determine whether exchange is practical in compacts with >70% theoretical density.

9:40 AM Invited
Reversible Hydrogen Storage in Mixtures of Mg(NH2)2 and LiH Studied by X-Ray and Neutron Diffraction: Yan Gao[1]; Job Rijssenbeek[1]; *Ji-Cheng Zhao*[1]; [1]General Electric Company

Mixtures containing various ratios of LiH and Mg(NH2)2 theoretically release up to 9 wt % H2 and are therefore attractive as hydrogen storage materials. An in-situ synchrotron X-ray diffraction (XRD) study was performed on mixture of LiH and Mg(NH2)2 at ratios of 2:1, 8:3 and 4:1 to examine similarities and differences in the reaction pathways and products. Detailed analysis indicates that the products consist of mixed Li/Mg imides, whose structures have not been determined previously. For the 2:1 ratio, the product is Li2Mg(NH)2 which displays three different crystal structures as a function of temperature: an orthorhombic structure at low temperature (alpha), a primitive cubic structure at intermediate temperature (beta), and a fcc-based structure at high temperature (gamma). The crystal structures were identified based on a combination of high-resolution XRD and neutron diffraction.

10:00 AM
High Pressure Raman Spectroscopy Studies on $LiAlH_4$: *Raja S. Chellappa*[1]; Dhanesh Chandra[1]; Yang Song[2]; Steve Gramsch[2]; Russell Hemley[2]; [1]University of Nevada; [2]Carnegie Institution of Washington, DC

LiAlH4 (10.6wt% hydrogen) belongs to a class of light metal complex hydride is a potential candidate for hydrogen storage due to its high theoretical hydrogen weight content. Hydrogen desorption from LiAlH4 has been generally successful in the presence of catalysts (with some loss of hydrogen content), however reversibility and enhancing the hydriding kinetics is still an active area of research. Recent theoretical calculations predicted a pressure induced phase transformation at room temperature from ambient pressure (α-LiAlH4) monoclinic (P21/C) structure to a high pressure (β-LiAlH4) phase (structure not known, predicted tetragonal) around ~3GPa. It is also predicted that the high pressure (β-LiAlH4) phase undergoes a huge volume collapse of about ~17% at phase transformation pressure. If this high pressure dense phase can be stabilized at ambient pressures, it can be very attractive for hydrogen storage (due to gravimetric as well as volumetric efficiency). Our recent in-situ high pressure Raman spectroscopy studies on LiAlH4 suggests a α-LiAlH4 to β-LiAlH4 transition beyond ~3GPa. We were also successful in retaining the high pressure (β-LiAlH4) phase by "pressure quenching" (rapid reduction in pressure) to a lower pressure of about ~1.2GPa. Raman spectra characteristics of the quenched sample do not change even after 6 days. The stabilization of the high pressure phase at ambient pressure holds promise for developing novel hydrogen storage materials that are both volumetrically as well as gravimetrically efficient. The crystal structure determination of the "pressure quenched" high pressure phase will be performed to conclusively establish the retention of the high pressure dense phase. The changes in vibrational mode characteristics as a function of pressure will be discussed in detail.

10:20 AM Invited
Li-C-H Systems as a Novel Family of Hydrogen Storage Media: *Takayuki Ichikawa*[1]; Hiroki Miyaoka[1]; Yong Zhong[1]; Shigehito Isobe[1]; Hironobu Fujii[1]; [1]Hiroshima University

Hydrogen storage properties of ballmilled mixtures composed of the hydrogenated nano-structured carbon ($C^{nano}H_x$) and lithium hydride (LiH) were examined by thermal and optical spectroscopic analyses. Actually, LiH needs higher temperature than 600°C for thermal decomposition even under vacuum condition, while $C^{nano}H_x$, which was mechanochemically synthesized from graphite powder by ballmilling under hydrogen atmosphere, reveals hydrogen and hydrocarbons desorption in wide range from 300 to 800°C. Nevertheless, the mixtures of $C^{nano}H_x$ and LiH mainly desorb hydrogen around 350°C. These phenomena indicate that the thermally liberated hydrocarbon radicals immediately react with LiH and release hydrogen instead of hydrocarbons, because $C^{nano}H_x$ contains hydrocarbon groups at the edge of nanosized graphene, which are recognized as IR active C-H stretching mode, and LiH is well-known as an ionic crystal. Furthermore, it is of interest that the mixtures keep a nanostructural feature even after annealing at 350°C revealed excellent cycle properties as hydrogen storage media.

10:40 AM Break

10:55 AM Keynote
Materials for Hydrogen Storage: From Nanostructures to Complex Hydrides: *Puru Jena*[1]; [1]Virginia Commonwealth University

The limited supply of fossil fuels, its adverse effect on the environment, and growing worldwide demand for energy has necessitated the search for new and clean sources of energy. The possibility of using hydrogen to meet this growing energy need has rekindled interest in the study of safe, efficient, and economical storage of hydrogen. The current methods for storing hydrogen as a compressed gas or liquid are not suitable for practical applications. An alternate method for hydrogen storage involves metal hydrides. Although conventional intermetallic hydrides can store hydrogen reversibly at around room temperature, the relative weight of stored hydrogen in these materials is rather low (1 – 3 wt %) and do not meet the requirements of the transportation industry (~10 wt %). For this the host materials have to consist of light elements such as Li, Be, B, C, Na, Mg, and Al. Unfortunately, the bonding of hydrogen in these materials is rather strong (covalent or ionic) and the thermodynamics and kinetics are poor. Ways must, therefore, be found to weaken the hydrogen bond strength so that light metal complex hydrides can be used as effective hydrogen storage materials. This talk will discuss the issues and challenges in storing hydrogen in light complex hydrides and discuss the role of nanostructuring and catalysts that can improve the thermodynamics and kinetics of hydrogen. In particular, I will discuss storage of hydrogen in Boron Nitride and Carbon nanocages and demonstrate that metallization of these nanostructures is necessary to store hydrogen with large gravimetric density. I will also discuss the properties of a class of materials called alanates which have the chemical composition [Mn+(AlH4)n-, M= Li, Na, K, Mg] and can store up to 18 wt % hydrogen, although the temperature where hydrogen desorbs is rather high. It was recently discovered that doping of Ti-based catalysts in NaAlH4 can significantly lower the hydrogen desorption temperature, but why and how Ti accomplishes this task remains a mystery. Using first principles calculations, I will provide a fundamental understanding of the electronic structure and stability of sodium alanates and how it is affected due to Ti doping. The role of Ti based precursors in introducing vacancy like defects and their influence on hydrogen desorption will be highlighted. It is hoped that the understanding gained here can be useful in designing better catalysts as well as hosts for hydrogen storage.

11:15 AM Invited

Light Metal Hydrides Studied by High Pressure Techniques: S. Doppiu[1]; A. Comin[1]; S. Sartori[2]; G. Principi[2]; *Oliver Gutfleisch*[1]; [1]IFW Dresden; [2]Università di Padova

The application of high hydrogen pressure techniques, such as high-pressure ball milling (HP-BM, up to 90 bar) and high-pressure differential scanning calorimetry (HP-DSC, up to 140 bar), is advantageous to synthesize novel hydrides for hydrogen storage under non-equilibrium conditions and to characterize their complex thermodynamic and kinetic properties. In the present study, two different classes of hydrides were investigated: (a) Mg and Mg-based alloys, (b) lithium alanates and modified lithium alanates. It is shown that milling Mg at high hydrogen pressure allows to shorten the time for the complete conversion into the hydride with the formation of a very fine nanometric powder. The hydride synthesised in this way shows better desorption kinetics than nanocristalline MgH2 obtained by milling under argon atmosphere. The thermal stability of LiAlH4 under different hydrogen pressure has been studied. Work is in progress to elucidate with further experiments the observed behaviour.

11:35 AM Invited

Recent Developments of Dopants for Hydrogen Exchange in Alanates: *Maximilian P. Fichtner*[1]; [1]Forschungszentrum Karlsruhe

One of the major obstacles to the application of complex hydrides in mobile devices is the slow hydrogenation kinetics of the materials. Therefore, a number of attempts have been undertaken to develop both fast and cost-effective catalysts. This contribution will give a short review on the state of the art as outlined in literature. Furthermore, the properties of new dopants for alanates and the results of recent attempts to drastically reduce costs of Ti-based catalysts will be presented.

11:55 AM Invited

Amorphization of Laves Phase Hydride: *Dhanesh Chandra*[1]; Ricardo B. Schwarz[2]; [1]University of Nevada; [2]Los Alamos National Laboratory

Crystalline $GdFe_2$ Laves phase are formed at relatively low temperatures; for example $GdFe_2H_{4.8}$ is formed at room temperature, which is reversible, with a H/M ratio of 1.6. However, at this temperature desorption of all the hydrogen is difficult because of slow kinetics. An amorphous $GdFe_2H_x$ phase forms at intermediate temperatures and pressures. The absorption isotherm is quite unusual, as the crystal-to-amorphous transformation is accompanied by either a gain or a loss of hydrogen and is temperature depended. Absorption isotherms taken below 475K showed that there is an abrupt decrease in the hydrogen capacity of the alloy during the crystalline-to-amorphous hydride phase transformation. Whereas the absorption isotherms taken above 475K showed that there is an abrupt increase in the hydrogen capacity during amorphization. At temperatures above 525 K, hydrogen absorption causes to the disproportionation of the $GdFe_2$ crystal into a two-phase mixture of GdH_2 and bcc α-Fe. The formation of the crystalline and amorphous $GdFe_2H_x$ phases, phase stability regions, disproportionation of the hydride will be presented.

12:15 PM Keynote

Aluminum TriHydride Studied by Powder Synchrotron X-Ray Diffraction: Crystal Structure and Thermal Decomposition: *Volodymyr A. Yartys*[1]; Jan Petter Maehlen[1]; [1]Institute for Energy Technology

Aluminium hydride is considered as a prospective solid H storage material for transport applications, having high gravimetric (10 wt.% H) and volumetric density of H (2 times higher compared to LH2) and, also, a convenient range of thermal stability (below 170°C, dependant on synthesis and storage conditions). Achieving sufficiently rapid and controllable decomposition of AlH3 and proposal of efficient synthesis routes to make the system Al-AlH3 reversible are focused in ongoing research. This work was aimed on studies of crystal structure of AlH3 and phase-structural transformations in the system Al-AlH3 during decomposition of the hydride by application of in situ synchrotron X-ray powder diffraction (SR-XRD). The SR-XRD studies were performed at the Swiss-Norwegian Beam Line (SNBL) at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. A specially designed cell for performing studies in controlled atmosphere was used. The studied sample is placed inside a quartz glass capillary, which is attached to a remote controlled gas distribution system providing either high-purity hydrogen at convenient pressures from a metal hydride hydrogen storage unit developed at IFE, or vacuum. During the present study of AlH3, a high-resolution data set (at room temperature) was first obtained, followed by an in-situ measurement by heating with a constant heating rate under secondary vacuum. The high-resolution SR XRD pattern was indexed in the trigonal unit cell; a = 4.44994(5) Å, c =11.8200(2) Å, V = 202.701(4) Å3; space group Rc (No.167). The hydrogen sublattice was successfully located from the refinements of the powder XRD pattern started with Al placed in a special position 6a [0,0,0]. This yielded the Al-H bonding distance of 1.712(3) Å and an octahedral coordination of Al in the AlH6 units. Furthermore, the refinements indicated a small charge transfer from Al to H corresponding to the formation of Al+0.15 and H-0.05. This charge distribution can be observed in the Fourier transform of the observed XRD pattern. We note a good correspondence between the data of this XRD work and the results of the powder neutron diffraction study of AlD3 [2], where the Al-D bond distance of 1.715 Å in the AlD6 octahedra was reported.

Advances in Furnace Integrity: Advances in Furnace Integrity and Pyrometallurgical Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Copper and Nickel and Cobalt Committee, TMS: Lead and Zinc Committee, TMS: Pyrometallurgy Committee

Program Organizers: Robert L. Stephens, Teck Cominco Metals Ltd; Christopher J. Newman, Chris Newman Metallurgical Consulting

Tuesday AM Room: 7D
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Robert L. Stephens, Teck Cominco Metals Ltd

8:30 AM

CONSCAN, A Laser Measurement System for Furnaces Which Are Operating on High Temperatures: *Oliver Zach*[1]; [1]RHI Non Ferrous Metals Engineering GmbH

To know as much as possible about the condition of the refractory material (lining thickness, slag layer, build ups, ...) in an high temperature furnace is very important due to the safety and economic impacts. The new CONSCAN system offers the possibility to measure the inner surface of an furnace, and based on this results to calculate the thickness of the refractory lining, slag layer, build ups,.... The CONSCAN System provides this results as figures and graphics. This system is also working under hot conditions so it is not necessary to cool down the furnace. Based on these results the process control and the refractory lining concept could be optimized for maximum campaign life of the furnace.

8:55 AM

Innovative Taphole and Launder Design Using Computer Assisted Engineering Tools: *Hugo Joubert*[1]; Bernice Leong[1]; Russel Tomlinson[1]; [1]Pyromet Technologies Pty Ltd

Tapholes and launders experience high temperature fluctuations during operating cycles, resulting in thermal stresses and thermal fatigue. This is true for all components including refractory materials and water-cooled copper elements. The technology supplier needs to design tapholes and launders, and select materials, to cope with these temperature fluctuations, not only during normal operating cycles, but also under extreme operating conditions. This paper discuss the use of advanced Computer Assisted Engineering (CAE) tools to assist in the design of tapholes and launders to achieve safe operation, increase campaign life, and limit maintenance cost and downtime. As an example the design of matte and slag tapholes for a PGM slag cleaning furnace is discussed, as well as an innovative water-cooled copper launder design that is inherently safe during, and re-usable after, damage caused by superheated metal or matte. Feedback on the operational performance of both the tapholes and copper launders is presented.

9:20 AM
Recent Improvements in Evaporative Cooling Technology for Copper Tapholes and Launders: *Pietro Navarra*[1]; Frank Mucciardi[1]; Tim Van Rompaey[2]; [1]McGill University; [2]Umicore Research

A great deal of effort has been expended on how to optimize conventional water cooling circuits in furnace cooling elements, and the results have been largely satisfactory. However, there is a general agreement that the proximity of high-pressure water passages to the flow of molten furnace products is a safety concern. Nonetheless there exists little alternative to the formula: increasing furnace integrity implies the incorporation of more extensive forced-convection water cooling systems. Recent developments in evaporative cooling technology have resulted in the development of a high-capacity heat pipe capable of operating under the thermally intense environments of metallurgical furnaces. The system functions using several kg of a working fluid (water) in total, has no moving parts, and allows for a significant reduction in cooling water requirements. Full-scale pilot tests of a copper taphole and slag launder were done in 2003 and 2005, with detailed results given in this paper.

9:45 AM Break

10:05 AM
Hacer Castillos en el Aire: Development of Oil Shale Retorting (Esta Fiesta se ha Preparado a Tontas y a Locos): *Larry M. Southwick*[1]; [1]LM Southwick & Assoc

The doubling of oil prices over the last two years has generated renewed interest in producing petroleum from oil shales and tar sands. This interest extends even to mining and metals companies, where many of the technologies used are similar. While tar sands processing is an economic reality, brought about primarily by improved and larger scale mining techniques, satisfactory economics have yet to be achieved with oil shale. Thermal processing of minerals is required, involving volatilization of the product in a retort and external condensation. The process is much like zinc smelting, some being very similar to the Imperial Smelting Process shaft furnaces. These oil shale plants involve multiple trains, each of fairly small size. As with tar sands, the route to achieving economic viability in oil shale processing must be through larger processing units. The one technology that offers such potential for large size is fluid bed retorting. In this paper, a review and critique will be provided of previous development efforts. A design will then be described which eliminates many of the problems experienced with earlier fluid bed configurations.

10:30 AM
Acoustic-Pulsation-Based Intensification of Heat Transfer in Continuous Furnace: *Alexey N. Lozhko*[1]; Olena V. Sorokina[1]; [1]National Metallurgical Academy of Ukraine

The technical-and-economic furnace exploitation indices are considerably influenced by the system of heat utilization. In the situation of total output decrease works are forced to exploit furnaces at the partial loading, sometimes even at the idle running for the sake of steam production. The furnace exploitation in this regime is known to cause the increase of the heat portion, taken away together with waste flue gases. The losses can be partially compensated at the expense of heat exchange efficiency development in the recuperator or heat-exchanger. One of the well-known methods, characterized by high efficiency and low expenditures is the intensification of the heat exchange process by the means of pulsation method.

TUESDAY AM

Alumina and Bauxite: Joint Session of Alumina and Bauxite & Aluminum Reduction Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Jean Doucet, Alcan Inc; Dag Olsen, Hydro Aluminium Primary Metals; Travis J. Galloway, Century Aluminum Company

Tuesday AM — Room: 7A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Renaud Santerre, Alcan Primary Metal

8:30 AM Introductory Comments by Renaud Santerre, Jean Doucet and/or Steve Lindsay

8:40 AM Invited
Smelter/Refinery Teaming for Alumina Supply: *Peter Bailey*[1]; [1]Sherwin Alumina Company

The speaker will address procedures and mechanisms necessary to ensurethat Refineries and Smelters team to optimize the supply of alumina.

9:05 AM
FLUIDCON – A New Pneumatic Conveying System for Alumina: *Andreas Wolf*[1]; Peter Hilgraf[1]; [1]Claudius Peters Projects GmbH

Alumina is transported by mechanical and pneumatic systems. The primary disadvantage of pneumatic conveying systems is the high-energy demand verses the mechanical processes. FLUIDCON is pneumatic conveying technology combining the advantage of airslide conveying and pneumatic pipe transport. The operating characteristic of the FLUIDCON is an extremely low transport velocity and low energy requirement. The advantages of the FLUIDCON system are discussed with the aid of the results of comprehensive systematic measurements performed in the research centre of Claudius Peters Technologies GmbH and in operating plants.

9:30 AM
Fundamentals of Crust and Alumina Powder Dissolution in Smelter Electrolytes and Their Impact on Cell Feeding: *Barry J. Welch*[1]; [1]University of New South Wales and Welbank Consulting Limited

The blend of knowledge derived from fundamental studies of the physics and science of alumina dissolution help understand the roles of key properties of smelter grade alumina - especially those impacting the operating disturbances linked with alumina "solubility". More important, the information also provides guidelines for good feeding practice and hence feeder design. The knowledge also presents a warning light on issues linked with the accessible bath volume when retrofitting larger anodes and operating at higher line currents.

9:55 AM
Evolution of Microstructure and Properties of SGA with Calcination of Bayer Gibbsite: Scott Powell[1]; Tania Groutso[1]; Margaret M. Hyland[1]; *James B. Metson*[1]; [1]University of Auckland

The microstructure of smelter grade alumina is relevant to smelter performance in a number of critical areas. The connection between the degree of calcination, the residual structural hydroxide content and HF generation, has recently been explored,[1] and impacts on alumina dissolution have also been reported. A critical parameter of relevance to both of the above, and particularly to dry-scrubber performance, is the evolution of surface area and its relationship to the progression of transition alumina phases.[2] The relationship of surface area and microstructure with equilibrium heating of gibbsite has been explored in the laboratory and results compared with those from several industrial calciner technologies. For the equilibrium samples, it is possible to accurately represent the transition alumina structure with the γ, γ', θ model previously developed,[3] and to correlate the evolution of phase with changes in surface area. The relative rates of hydroxyl loss versus loss of surface area are of critical importance in understanding the balance between HF generation at the cell and HF capture in the scrubber. [1]M.M. Hyland, E. Patterson, and B.J. Welch.

Light Metals 2004 Edited A.T. Tabereaux, The Minerals, Metals & Materials Society p.361-366 (2004). [2]K.Wefers and C.Misra, Alcoa Laboratories, Technical paper No. 19 Revised (1987). [3]J. B. Metson, M. M. Hyland and T. Groutso. Alumina Phase Distribution, Structural Hydroxyl And Performance Of Smelter Grade Aluminas In The Reduction Cell. Light Metals 2005 Edited by Halvor Kvande. The Minerals, Metals & Materials Society), p. 127-131, (2005).

10:20 AM Break

10:30 AM
Optimizing the Alumina Feeding Strategy for Different Pot Technologies, Work Practices, and Pot Process Parameters: *Pablo R. Navarro*[1]; [1]Aluar Aluminio Argentino

This presentation emphasizes the need to optimize the alumina feeding strategy for each combination of pot technology, lining design, work practices, and process parameters. The evolution of Aluar's alumina feeding algorithm is presented together with the resultant improvements in alumina content control and anode effect frequency.

10:55 AM
Alumina Requirements of Soderberg Smelters: *Alton T. Tabereaux*[1]; [1]Alcoa Inc

This presentation addresses the key physical and chemical properties required for side-break and point feeder Soderberg cells. Issues regarding the use of various types of side break operations using wheels, bars, hammer and point feeders on crust breaking and dissolving alumina in Soderberg operations are discussed. Strategy and countermeasures have been developed to reduce the impact of using alumina that have problematic particle size distributions (PSD) and other properties to improve alumina management in Soderberg cells.

11:20 AM
Beryllium Mass Flow in Pot Room Bath: *Stephen Joseph Lindsay*[1]; Charles L. Dobbs; [1]Alcoa Inc

Concentration of beryllium in pot room bath has become an issue of concern in more than one country during recent years. In this paper the author discusses the mass flow mechanisms that permit beryllium to become concentrated in pot bath as well as those factors that control reductions in beryllium concentrations when the source of contamination is removed. Some rules of thumb are proposed regarding decay rates in pot room bath.

11:45 AM
Automated Crystal Optical Technique for Quantitative Determination of Phase and Particle Size Composition of Alumina: *Alexander Gennadievich Suss*[1]; Andrey Vladimirovich Panov[1]; [1]VAMI-RUSAL

For many years specialists of VAMI have been involved in developing of automated techniques and new equipment arrangement for determination of phase and particle size composition of products in alumina manufacture. Crystal optical analysis being a complicated technique requiring professional background however is not free from subjective estimation as far as the quantitative analysis is concerned. Modern advances in computer diagnostics and processing allow overcoming these problems. At present at several alumina refineries a technique for automated quantitative determination of alumina phase composition using «Image Analysis System» is tested. The following automated techniques were developed with the help of «Image Analysis System»: 1. determination of alpha-modification content in alumina of all grades irrespectively of method of production of alumina and way of its formation; 2. determination of quantitative phase composition of smelter grade alumina; 3. automated determination of particle size distribution of aluminium hydroxide containing fine particles.

Aluminum Reduction Technology: Joint Session of Alumina and Bauxite & Aluminum Reduction Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Jean Doucet, Alcan Inc; Dag Olsen, Hydro Aluminium Primary Metals; Travis J. Galloway, Century Aluminum Company

Tuesday AM — Room: 7A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Renaud Santerre, Alcan Primary Metal

See the Alumina and Bauxite Symposium on page 102 for presentations.

Amiya Mukherjee Symposium on Processing and Mechanical Response of Engineering Materials: Mechanical Behavior of Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
Program Organizers: Judy Schneider, Mississippi State University; Rajiv S. Mishra, University of Missouri; Yuntian T. Zhu, Los Alamos National Laboratory; Khaled B. Morsi, San Diego State University; Viola L. Acoff, University of Alabama; Eric M. Taleff, University of Texas; Thomas R. Bieler, Michigan State University

Tuesday AM — Room: 217C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Khaled B. Morsi, San Diego State University; Viola L. Acoff, University of Alabama

8:30 AM Invited
Microstructure and Mechanical Properties of Commercial Grade Aluminium as a Function of Welding Speed Using Friction Stir Welding: *Jyoti Mukhopadhyay*[1]; G. S. Sengar[1]; T. Sakthivel[1]; [1]JNARDDC

The weld properties remain an area of uncertainty with respect to the effect of different speeds of Friction Stir Welding (FSW). For this purpose, a FSW tool was designed in such a way that the shoulder of the tool not only provides additional heat generated by friction, but also prevents plasticised material to escape. Infact, the weld was formed by the deformation of materials at temperature below the melting point. The simultaneous rotation of welding tool and the translational motion of the work piece during the welding process produced a high quality weld joint. In the present investigation, an aluminium weld was made using FSW technique. The mechanical properties and microstructural aspects of welded areas were investigated. It is observed that a good correlation exists between the mechanical properties and welding speeds. The main objective of this investigation is to evaluate the mechanical properties and microstructures at different welding speeds.

8:50 AM
Void Nucleation at Grain Boundary Triple Points in AA6022-T43 Aluminum Alloy Sheet: *Joseph Querin*[1]; Judy Schneider[1]; Mark F. Horstemeyer[1]; [1]Mississippi State University

The design and optimization of processing parameters for forming stamped sheet metal components can be expedited by using accurate material models that take into consideration the relationship between microstructure and mechanical properties. One such genre is damage modeling, where the degradation of material properties is attributed to void nucleation, growth, and coalescence. In monotonic tensile testing of 1-mm thick sheet AA6022-T43, microscopic void nucleation was found to first occur at grain boundary triple points (GBTPs). The grain orientations at various

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GBTPs in the strained specimens were determined using electron backscattered diffraction. These orientations were used in conjunction with a crystal plasticity model implemented into a finite element analysis code to reveal that a state of hydrostatic tensile stress surrounded the GBTPs where voids nucleated and a state of hydrostatic compressive stress surrounded those GBPTs that did not nucleate voids.

9:10 AM Invited

The Effect of Silicon on the Deformation and Fracture of Molybdenum: Daniel Sturm[1]; *Joachim H. Schneibel*[2]; Holger Saage[1]; Martin Heilmaier[1]; [1]Otto-von-Guericke-University Magdeburg; [2]Oak Ridge National Laboratory

Mo-Si-B intermetallic alloys are finding interest as structural materials for ultra-high temperature applications. One of their phases, the Mo-Si solid solution, is of paramount importance for the fracture toughness. In general, Si was found to reduce the lattice parameter, slow down grain growth, reduce the room temperature fracture toughness, and increase the tensile strength at temperatures up to 1100ºC. Small Si concentrations (e.g., 0.3 at%) actually cause solid solution softening at room temperature which may be attributed to a reduced Peierls stress for screw dislocations. Finally, a model is presented which accounts for the effect of Si on the yield strength.The authors appreciate experimental support from Plansee AG, Reutte, Austria. JHS acknowledges support 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 Schiebold Guest Professorship at the University of Magdeburg.

9:30 AM

Co-Continuous Composites for High Temperaure Service: *Glenn S. Daehn*[1]; Eduardo del Rio Perez[1]; James C. Nash[1]; James C. Williams[1]; [1]Ohio State University

In principle it would be desirable to create metal-ceramic composites for high temperature service, because the ceramic can reduce density and give strength. However, strain mismatch associated with differential thermal expansion typically make these materials very weak or prone to accumulating damage in thermal cycling. Here we look at a different approach. We evaluate interpenetrating phase composites of alumina and aluminum as well as alumina and NiAl. Experiments compare the thermal cycling induced damage in these materials to traditional discontinuously-reinforced composites. These show that the co-continuous morphology is much more resistant to thermal cycling induced damage. Lastly we will detail a technique whereby large volumes of near-single crystal reticulated alumina can be created. These comosites can have exceptional damage tolerance, good high temperature creep resistance and reduced density versus refractory metals.

9:50 AM Invited

High Heat Flux Exposures of NiCrAlY-Coated GRCop-84 Substrates: *Sai V. Raj*[1]; Louis J. Ghosn[2]; Raymond C. Robinson[3]; [1]NASA Glenn Research Center; [2]Ohio Aerospace Institute; [3]QSS, Inc.

An advanced Cu-8(at.%)Cr-4%Nb alloy developed at the Glenn Research Center, and designated as GRCop-84, is currently being considered for use as liners in combustor chambers in NASA's exploration vehicles. Protective overlay coatings alloys are being developed for GRCop-84 to prevent environmental degradation. The creep, tensile and thermophysical data obtained on the monolithic NiCrAlY top and copper alloy bond coats were used to evaluate the stresses in the coating. Coated and uncoated specimens were exposed to a high heat flux hydrogen-oxygen combustion flame in NASA's Quick Access Rocket Exhaust (QARE) rig to evaluate the performance of the coatings. The measured temperatures and heat fluxes were compared with those predicted from finite element analyses (FEA), and it is shown that the experimental and the theoretical results are in excellent agreement. The expected coating performance in a rocket engine chamber is modeled.

10:10 AM

Experimental Techniques for the Characterization of Thermal Barrier Coating Bond Coats: *Robert J. Thompson*[1]; J. C. Zhao[2]; Kevin J. Hemker[1]; [1]Johns Hopkins University; [2]GE Global Research Center

Thermal barrier coatings (TBCs), commonly used in modern gas turbines and jet engines, are dynamic, multi-layered, metallic-ceramic structures consisting of a superalloy substrate, an aluminum-rich bond coat, a thermally grown oxide (TGO), and a ceramic top coat. Many (Ni,Pt)Al bond coats exhibit a strain-inducing B2 to $L1_0$ martensitic phase transformation that occurs during thermal cycling. Knowledge of this transformation and its effects on the bond coat's mechanical properties are essential to modeling the behavior of a TBC. In this study, combinatorial methods are employed to examine the effect of ternary elements on the martensitic transformation and microsample tensile testing techniques are developed to characterize the elevated temperature mechanical behavior of these alloys. The influence of bond coat composition on the martensitic transformation, high temperature strength, and overall performance of TBC systems will be discussed.

10:30 AM

Overview of the Processing and Properties of Hafnium-Based Ultra High Temperature Ceramics: *Matthew J. Gasch*[1]; Mike Gusman[1]; Donald T. Ellerby[2]; Ed Irby[2]; Sylvia M. Johnson[2]; [1]ELORET Inc; [2]NASA Ames Research Center

Hafnium diboride-based materials are being investigated at NASA Ames as part of ongoing research aimed at developing superior heat resistant materials for use in extremely high temperature applications. For example, the exceptionally high melting temperatures of hafnium diboride (>3000°C) make it a suitable candidate for the extreme conditions experienced during atmospheric re-entry. Significant work on the manufacture of dense monolithic ceramics has resulted in the development of processing techniques specific to HfB2- SiC based materials. Powder handling, die configuration, and hot pressing schedule are all aspects that greatly affect the final product. Processing methods found to optimize the uniformity of the material and produce dense billets are addressed, as are the temperature dependant mechanical and thermal properties of monolithic HfB2-SiC materials.

10:50 AM Break

11:00 AM Invited

Atomistic Modeling of Dislocations in HCP Metals: *M. I. Baskes*[1]; Srinivasan G. Srivilliputhur[1]; [1]Los Alamos National Laboratory

In the first part of our talk we will discuss our atomistic calculations, in a generic hexagonal close packed (HCP) metal, wherein we systematically vary the (c/a) ratio and the basal stacking fault energy and explore its effects on the dislocation glide properties. This will be followed by a description of our atomistic calculations of dislocation motion in HCP zirconium metal (Zr). 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. For both these studies we use a semi-empirical modified embedded atom method potential that includes angular forces, to describe the interatomic interactions.

11:20 AM

Refinement of the VPSC Model for Deformation of Zirconium: *Gwenaelle Proust*[1]; Carlos N. Tome[1]; George C. Kaschner[1]; Irene J. Beyerlein[1]; [1]Los Alamos National Laboratory

Hexagonal materials plastically deform by activating various families of slip and twin systems depending on diverse processing parameters such as temperature and strain rate. In this work, we propose to further elucidate the balance between the diverse twinning and slip deformation modes as a function of temperature for high purity zirconium. We propose here a new version of the visco-plastic self consistent (VPSC) model to simulate the deformation of this material. In this new version, twinning is accounted for using a composite-grain twin model which has been shown to better reproduce strain path changes than the previously developed Predominant Twin Reorientation (PTR) scheme. We use tensile and compressive experimental data at diverse temperature regimes varying between 76K and 450K to adjust the constitutive parameters of the VPSC model. The experiments were realized on in-plane and through-thickness samples obtained from a clock-rolled zirconium plate.

11:40 AM

The Importance of Slip and Twinning within Deformation Twins in Zirconium: *Rodney J. McCabe*[1]; Ellen K. Cerreta[1]; Amit Misra[1]; Carlos N. Tome[1]; George C. Kaschner[1]; [1]Los Alamos National Laboratory

Zirconium exhibits ductile deformation behavior at liquid nitrogen (LN) temperature largely due to the operation of deformation twins. Deformation within the first generation of deformation twins can have significant impacts on microstructures, developing textures, and stress strain responses, particularly for uniaxial compressive strains greater than around 10% and for changes in strain path. In addition, our polycrystal constitutive simulations (VPSC) predict activities inside twins that need to be confirmed experimentally. In the present study we examine the role of slip and twinning within deformation twins using OIM and TEM. The results are used to explain both stress strain responses and developing textures in zirconium samples compressed either parallel or perpendicular to an initially strong c-axis texture.

12:00 PM Invited

Exploring the Dislocation/Twin Boundary Interactions in Zirconium: *George C. Kaschner*[1]; Carlos N. Tome[1]; Ellen K. Cerreta[1]; Rodney J. McCabe[1]; Amit Misra[1]; Sven Vogel[1]; [1]Los Alamos National Laboratory

We have shown in previous work that deformation of zirconium is a competing/complementary blend of twinning and slip. At moderate strains and cold temperatures, the twinned structure is sufficiently dense in forest dislocations that TEM observation of dislocation/twin interactions is obscured. We have employed in situ neutron diffraction to study the kinetics of recovery and recrystallization to produce a substructure that retains the deformation twin structure yet with a greatly reduced dislocation density. That product is then reloaded in compression and observed using TEM to study the interactions of dislocations with the deformation twin boundaries.

12:20 PM

Exploring the Dislocation-Twin Boundary Interactions in Zirconium: A TEM Investigation: *Cynthia D. Smith*[1]; Henry A. Padilla[1]; Ian M. Robertson[1]; John Lambros[1]; Armand J. Beaudoin[1]; George Kaschner[2]; [1]University of Illinois; [2]Los Alamos National Laboratory

Deformation in zirconium, as in all HCP materials, involves a competition between twinning and slip under most deformation conditions. When deforming previously rolled material, twinning is observed in both in-plane compression samples, which show an increasing hardening rate, and through-thickness compression samples, which show a constant hardening rate. The mechanical behavior of zirconium is probed using interrupted high strain rate tests, post-mortem TEM characterization and dynamic straining in the TEM. High strain rates produce a mixed response with some grains containing a high density of dislocations and others twins. The twins are predominately of the tension type. The dynamics of the interaction of twins with pre-existing grain boundaries are being investigated by re-loading the interrupted material in the TEM. In this talk, these observations will be correlated to bulk mechanical properties.

Biological Materials Science: Biological Materials

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Andrea M. Hodge, Lawrence Livermore National Laboratory; Chwee Teck Lim, National University of Singapore; Richard Alan LeSar, Los Alamos National Laboratory; Marc Andre Meyers, University of California, San Diego

Tuesday AM	Room: 212A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Marc A. Meyers, University of California; Masaaki Sato, Tohoku University

8:30 AM Invited

The Organic Layer within Abalone Nacre: *Marc A. Meyers*[1]; Albert Y. M. Lin[1]; Julie Muyco[1]; Kenneth S. Vecchio[1]; [1]University of California, San Diego

In this study, we focus on the thin viscoelastic layers between the aragonitic tiles in abalone shell and on their role in the growth of tiles as well as on the mechanical properties. AFM shows that the thin layer is comprised of linear chains froming a random planar network. This network contains small holes that expand, upon stretching of the film. They play a pivotal role in growth and enable the maintenance of the same crystallographic relationship for tiles grown sequentially in the 'terraced cone' mode. TEM analysis is carried out to establish the size and spacing of bridges between adjacent tile layers, corresponding to the holes in organic layer. The growth of nacre in abalone was interrupted after periodic times in order to observe the formation of the organic layer. The transition between spherulitic/columnar and tile growth is identified and characterized. Research Support:NSF DMR.

9:00 AM Invited

A Step-Specific Model for Shape Control of Biomineral Phases: *Roger Qiu*[1]; Andrzej Wierzbicki[2]; Germaine Fu[3]; Chris Orme[1]; John Hoyer[4]; George Nancollas[5]; Dan Morse[3]; Jame De Yoreo[1]; [1]Lawrence Livermore National Laboratory; [2]University of South Alabama; [3]University of California, Santa Barbara; [4]University of Pennsylvania; [5]University at Buffalo

Biomineralized structure often exhibit shapes that are different from crystals grown from pure solutions. Understanding the underlying principles by which biomolecules modify the shape can shed light on biomineral processes and suggest strategies for bio-inspired materials design. We use *in situ* atomic force microscopy and molecular modeling to investigate stereochemistry and molecular scale growth modification of calcite and calcium oxalate monohydrate by small molecules, polypeptides, and proteins. We find in all systems that the resulting growth kinetics and crystal habit are modified by interaction to specific atomic steps on existing crystal faces. Moreover, changes in macroscopic crystal shape mimic the modifications seen at the atomic level. Molecular modeling calculating the modifier-crystal interaction energy supports these conclusions in that binding energies are greatest at the step edges and in accordance with the step-specificity observed in the experiments. These results argue for a model of shape modification recognizing the role of steps.

9:30 AM

On the Measurement of Cell Mechanical Properties and Cell Adhesion: *Yifang Cao*[1]; Jianbo Chen[1]; Monica E. Bravo[1]; Randy Bly[1]; Alberto M. Cuitiño[2]; Wole Soboyejo[1]; [1]Princeton University; [2]Rutgers University

This paper presents a range of experimental techniques for the measurement of the interfacial strengths between human-osteo-sarcoma (HOS) cells and titanium and poly-dimethyl-siloxane (PDMS) surfaces. The interfacial strengths are measured using shear assay experiments. These involved the laminar flow of cell culture fluid over cells that were cultured onto titanium and PDMS surfaces. In-situ images of cell deformation were also obtained during the shear assay experiments. These were analyzed using digital image correlation (DIC) techniques, which were used to extract the local strain distributions. The results were then incorporated into a finite element model for the extraction of cell modulus. Subsequently,

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the cell lift-off strengths were determined using micro-pipette aspiration strengths. The implication of the results are then dicussed for the surface design of implantable microelectronics, bioMEMS structures and orthopedic/dental structures.

9:50 AM
Lobster Cuticle as an Example of Smart Anisotropic Material: *Patricia Romano*[1]; Helge Fabritius[1]; Christoph Sachs[1]; Dierk Raabe[1]; [1]Max Planck Institute for Iron Research

Many biological materials are composed of fibrils arranged according to well defined three-dimensional patterns. These materials often show a strong anisotropy. The composite microstructure, comprising hard inorganic and soft organic materials, plays a key role in enhancing the toughness, strength, and hardness of cuticles by restricting crack growth and delocalizing deformation fields. An essential characteristic of biological materials is their hierarchical organization, at the nanometer-to-millimeter scale. Lobster cuticle is a good example of this. We studied the exoskeleton of the American lobster (*Homarus americanus*) using optical, scanning and transmission electron microscopy. The results show that the structure is more complex that the commonly assumed model. The structure of the chitin-protein planes creates the twisted plywood pattern characteristic of the arthropod cuticle. However, these planes are not simple arrays of parallel chitin-protein fibers, but interconnected fibers forming planar honeycombs rotating around continuous lenticular cavities.

10:10 AM Break

10:30 AM Invited
Molecular Interaction and Modification in Human Diseased Cells: *ChweeTeck Lim*[1]; Ang Li[1]; Kevin Shyong Wei Tan[1]; [1]National University of Singapore

A key feature of the pathophysiology of Plasmodium falciparum malaria is the increased rigidity and stickiness of infected red blood cells (RBCs) which can cause serious impairment of blood flow through narrow capillaries. Proteins released from the parasite induce alterations to the membrane resulting in these changes. These proteins manifest as knob-like structures on the membrane surface of the RBC. Here, we report an atomic force microscopy (AFM) study of malaria-infected RBCs. We first obtain high resolution AFM images of the nanosized knob structures on the surface of infected RBCs to determine the 3-D structure, size and density of the knobs. Next, we quantitatively measure the stickiness or cytoadherence of infected RBCs by identifying and measuring the molecular interaction forces of the various ligand-receptor pairs contributing to cytoadherence. AFM is used to systematically measure the single bond strength between these ligand-receptor pairs occurring between endothelial cells and the infected RBCs.

11:00 AM Invited
Mechanical Properties of Isolated Internal Organs of Vascular Cells: *Masaaki Sato*[1]; [1]Tohoku University

Mechanical properties of isolated stress fibers and nuclei were measured using a newly designed micro tensile tester and a micropipette technique. Stress fibers isolated from smooth muscle cells were elongated more than two-fold of the initial length and the resistance to elongation increased with increasing the tensile load. The estimated average value of the initial elastic modulus was 2.5 MPa, which value was much lower than that of synthesized single actin filaments, 1.8 GPa. Isolated nuclei from endothelial cells remained almost rounded shape under static conditions. However, the nuclei were supposed to be compressed state in the cells by observing the 3-D topography. When we applied shear stress, the nuclei showed elongated shape. Elastic modulus measurements with pipette aspiration test showed that nuclei hardened significantly from 0.42±0.12 kPa to 0.62±0.15 kPa with the fluid shear stress.

11:30 AM
Buckling Resistance of Slender, Nano-Scale Components in Biological Materials: Baohua Ji[1]; Huajian Gao[1]; *K. Jimmy Hsia*[2]; [1]Max Planck Institute for Metals Research; [2]University of Illinois

Biological materials such as bone, tooth and sea shell exhibit a generic nanoscale composite structure consisting of mineral crystals with very large aspect ratios embedded in a soft protein matrix. It is not clear how the slender components in biological materials solve the problem of buckling under compressive loading. In the present study, a simple analytical model is developed to show that the protein matrix in the biocomposites plays a critical role in stabilizing mineral crystals under compression. It is especially interesting in view of the three orders of magnitude difference in the elastic moduli of protein and mineral. The critical load for buckling is derived as a function of aspect ratio and volume concentration of mineral. A unique feature for biocomposite structure is that there exists a lower threshold value for the critical load below which the mineral crystals become stable against buckling no matter how slender they are.

11:50 AM
Toucan and Hornbill Beaks: A Comparative Study: *Yasuaki Seki*[1]; Bimal Kad[1]; David J. Benson[1]; Marc A. Meyers[1]; [1]University of California

The structure and mechanical behavior of a Toco toucan and two species of hornbill beaks (wreathed and wrinkled hornbills) were examined. The beaks are comprised of a sandwich composite with an exterior of multiple layers of keratin scales and a core composed of fibrous network of closed cells made of bone. The weight of hornbill beaks is significantly higher than the toucan beak and the internal foam is fused to the keratin shell. A distinctive feature of the hornbill beak is its *casque* formed from the keratin layers. The *casque* has an acoustic function. Both toucan and hornbill beaks have a hollow region in the center of the foam. Tensile and compression tests were applied to determine the mechanical properties of external shells and internal foams. Micro and nanoindentation hardness measurements were used to corroborate these values. The compressive behaviors of the foams were modeled by the Gibson-Ashby constitutive equations.

12:10 PM
Influence of Absorbed Amino Acids on Hydroxyapatite Crystallization and Properties: Olga Alexandrovna Golovanova[1]; *Elena Vladimirovna Rosseyeva*[1]; Anna Alexandrovna Ogneva[2]; Olga Victorovna Frank-Kamenetskaya[1]; [1]St. Petersburg State University; [2]Omsk State University

Ca-deficit hydroxyapatite is the main inorganic phase in human organism. The study of the interaction of hydroxyapatite with organic molecules is only in progress. We have synthesized the crystalline hydroxyapatite powders with molar Ca/P ration 1.57 –1.67 at physiological condition (37ºC, phosphate concentration 0.02 mol/l, pH24 hour = 7.5±0.1). The effect of amino acids (L – forms of glutamic acid, aspartic acid, glycine and lysine) on electrokinetic properties and particle size of synthesized material has been estimated. The determination of zeta-potential allow to shown, that the absorption of glycine and lysine result in overcharge of the hydroxyapatite (Ca/P = 1,58±0,01) surface and absorption decrease as the charge on the absorbing species become more negative. It was established, that the greatest decreasing of hydroxyapatite particle size (Ca/P = 1,67±0,01) has been occurred in the presence aspartic acid, overcharging the surface of crystals also.

Bulk Metallic Glasses: Atomic Study and Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Raymond A. Buchanan, University of Tennessee

Tuesday AM — Room: 217B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Christopher A. Schuh, Massachusetts Institute of Technology; Michael Atzmon, University of Michigan

8:30 AM Keynote
Atomistic Mechanism of Atomic Transport in Metallic Glasses: *Takeshi Egami*[1]; [1]University of Tennessee

Atomic transport and mechanical properties in metallic glasses are often discussed in terms of the free-volume model. However, in glasses the Stokes-Einstein relationship that connects diffusivity and viscosity does

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not work, and in addition to free-volume sinks have to be introduced as an additional parameter. At the same time the validity of the free-volume model has been seriously questioned by computer simulations. We propose an alternative model based upon the fluctuation and exchange of atomic bonds, defined by the nearest neighbor atoms. The topology of atomic connectivity is related to the local energy landscapes through the atomic-level stresses, and then translated into thermodynamics. We discuss how glass transition, viscosity, diffusion and mechanical deformation can be elucidated by this model.

9:00 AM Invited
Application of the Dense Cluster Packing Model: *Daniel B. Miracle*[1]; [1]U.S. Air Force

The Dense Cluster Packing (DCP) atomic structural model for metallic glasses has recently been established. This structural model is derived from the requirement to efficiently fill space in a system of unequal spheres. Solute-centered clusters are proposed from the relative atomic sizes of solute and solvent atoms, and these atomic clusters are densely packed to efficiently fill space. This structural arrangement provides a number of predictions that can be compared with experiment. The purpose of this presentation will be to explore the implications of this model with respect to topological and chemical effects known to be important in the stability of metallic glasses. Successes of this model, as well as unanswered questions, will be discussed.

9:25 AM Invited
Challenges in Metallic Glass Research: From Atomic-Level Structures to Inch-Diameter Ingots: *Evan Ma*[1]; [1]Johns Hopkins University

We discuss two issues of strong current interest in MG research. The first is a fundamental question for the science of glasses. Unlike the well-defined long-range order that characterizes crystalline metals, the details of atomic arrangements in amorphous alloys remain mysterious (although short-range order is considered ubiquitous and medium-range order likely). We use a combination of the state-of-the-art experimental and computational techniques to elucidate exactly how the atoms pack themselves in 3D, in several model MGs with different chemistry and atomic size ratios. On the application side, one of the challenges is to be able to cast BMGs of large sizes using only ordinary, inexpensive metals (e.g., free of Be or Pd), and locate compositions with extraordinary glass forming ability (e.g., critical size > 1 inch). We have come up with a search strategy that will be shown to be up to the task in 3D composition space.

9:50 AM Invited
Concentration Dependence of the Glass Forming Ability in Ternary Ca-Mg-Zn and Ca-Mg-Cu Systems: *Oleg N. Senkov*[1]; James Michael Scott[1]; Daniel B. Miracle[2]; Stephane Gorsse[3]; [1]UES Inc; [2]Air Force Research Laboratory; [3]Bordeaux Institute of Condensed Matter Chemistry

Several Ca-Mg-Zn and Ca-Mg-Cu bulk metallic glasses with the thickness of at least 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. Glass forming ability was determined as a function of the alloy composition and correlated with the driving force of crystallization of different phases at a near glass transition temperature. The compositions having a minimum driving force for crystallization showed better glass forming ability.

10:15 AM
Maximum Critical Thickness in Al-La-Ni Metallic Glasses: W. S. Sanders[1]; J. S. Warner[1]; *Daniel B. Miracle*[1]; [1]U.S. Air Force

The stability of Al-rich glasses in the Al-La-Ni system has been measured. Glasses with critical thicknesses ranging from 270 μm to 780 μm have been achieved. An in-situ two-phase amorphous region containing as much as ~10% by volume of nanocrystals was also observed with critical thicknesses ranging from 420 μm to 950 μm. Measurements of Tg, Tx and Tl, as well as other empirical measures of thermal stability based on these quantities, confirm the unusual thermal stability of these glasses. These results suggest that bulk Al-based glasses with a maximum critical thickness exceeding 1 mm may be possible in alloys based on this system.

10:35 AM Break

10:45 AM
Observation of Structural Effects after Mechanical and Thermal Treatment of BMG: *Wojciech Dmowski*[1]; Cang Fan[1]; Peter K. Liaw[1]; Takeshi Egami[1]; [1]University of Tennessee

We have examined atomic structure of several Zr-based metallic glasses that have been heat treated below the glass transition temperature or mechanically deformed by an uniaxial tension and cold rolling. The atomic structure of these materials was compared to the as cast state. Structural studies on the annealed samples were performed using time-of-flight neutron diffraction and followed by the atomic pair distribution function (PDF) analyses. X-ray diffraction studies were carried out on the mechanically deformed samples using high energy synchrotron source and area detector and the data was analyzed assuming non isotropic scattering. The annealed samples exhibited structural relaxation consistent with the elimination of short and long inter-atomic distances in the first coordination shell. The mechanically deformed glasses showed evidence of induced structural anisotropy.

11:05 AM
Local Structural Fluctuations and the Glass Transition: *Rachel Aga*[1]; Jamie Morris[1]; Takeshi Egami[2]; Valentin Levashov[2]; [1]Oak Ridge National Laboratory; [2]University of Tennessee

The local structural properties of an amorphous system are examined using molecular dynamics simulations. These local properties include pressure, bulk modulus, shear modulus, volume strain and shear stress. Starting from the low temperature glass, the system starts to show fluctuations of these structural properties upon warming toward the glass transition. Further, we find that the change in the scaling factor for the diffusion and the shear viscosity in the Stokes-Einstein relation is correlated with the change in behavior of the structural fluctuations. By including the many-body effects to the initial pair potential used, we are able to examine model systems with different Poisson's ratios.

11:25 AM
Toward Fine Structures in Metallic Glasses: P. Zetterström[1]; *Yandong Wang*[2]; P. Jóvári[3]; Y. Ren[4]; H. F. Zhang[5]; R. Delaplane[1]; H. Choo[6]; P. K. Liaw[6]; L. Zuo[2]; [1]Uppsala University; [2]Northeastern University; [3]Hungarian Academy of Sciences; [4]Argonne National Laboratory; [5]Chinese Academy of Sciences; [6]University of Tennessee

The local structure in metallic glassy materials, including whether the icosahedral short-range order exists and, if so, in what detailed arrangements, is of fundamental interest, but still remains a mystery in materials science. Here, we use the Reverse Monte Carlo (RMC) method to obtain the three-dimensional discrete distributions of constitutional atoms in melt-spun CuZr and CuZrTi metallic glasses from neutron and X-ray scattering data. It was found that the topological short- and medium- range orders are dominated in the rapid-liquid-cooling metallic glasses, and the icosahedral short-range order is less stable in the CuZr binary alloy than in the Ti-doped ternary alloy. The analysis method is generally applicable to other metallic glasses, and the obtained results provide the insightful information on packing of atoms in glassy metals, which can be used as fundamental experimental inputs for modeling mechanical behaviors by the molecular-dynamics simulations and establishing quantitative models to predict the forming abilities of bulk-metallic glasses. The present work is supported by the Swedish Research Council in the frame of the SIDA project (Grant No. 348-2004-3475), the Ministry of Education of China (with the New-Century Outstanding Scholars Fund), the National Natural Science Foundation of China (Grant No. 50471026), and the National Science Foundation International Materials Institutes (IMI) Program with Dr. Carmen Huber as the Program Director.

11:45 AM
Local Atomic Structure and Size Distribution Study on $Cu_{60}Zr_{60}Ti_{10}$ Metallic Glasses: *Cang Fan*[1]; D. M. Nicholson[2]; T. Proffen[3]; R. P. Hjelm[3]; W. Zhang[4]; C. Hammetter[3]; H. Choo[5]; P. K. Liaw[1]; Akihisa Inoue[4]; T. Egami[5]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]Los Alamos National Laboratory; [4]Tohoku University; [5]University of Tennessee and Oak Ridge National Laboratory

It has been reported that the $Cu_{60}Zr_{30}Ti_{10}$ bulk-metallic glass contains nano-particles that likely contribute to the material's good mechanical properties. There is no report on the particle size distributions and local atomic structures (short-range to medium-range order), which can help us further understand the mechanism of the nanocrystallization. To study the local atomic structures, we performed neutron diffraction on as-cast and partially crystallized samples to determine pair distribution functions (PDF). The PDF analysis indicated that the peaks of the Zr-Cu pair bond-lengths are in the range of 2.82 and 3.08 angstroms in as-cast samples. The coordination numbers of Cu-Cu and Zr-Cu are increased after partial crystallization. The atomic bonding is discussed on the basis of first principles calculations of the electronic and atomic structure of instantaneously quenched liquid samples. We also performed small angle neutron scattering (SANS) on the as-cast samples to investigate the nano-particle size distributions.

12:05 PM
Optimizing Chemistry of Bulk Metallic Glasses for Improved Thermal Stability: *George S. Dulikravich*[1]; Igor N. Egorov-Yegorov[2]; [1]Florida International University; [2]IOSO Technology Center

We propose a novel methodology for predicting concentration of each of the important alloying elements in BMGs so that the new alloys will have improved thermal stability and glass forming ability. Specifically, we are concentrating on maximizing Tliquidus and Trg = Tg/Tliquidus. The method is based on computational algorithms rather than on traditional experimentation, expert experience, and intuition. Specifically, the proposed alloy design method combines an advanced stochastic multi-objective evolutionary optimization algorithm based on self-adapting response surface methodology and another algorithm that performs accurate prediction of thermo-mechanical properties from given concentrations of alloying elements. During the iterative computational design procedure, a few judiciously chosen new BMG alloys need to be manufactured and experimentally evaluated for their properties in order to continuously verify the accuracy of the entire design methodology. This BMG alloy design optimization method thus minimizes the need for costly and time-consuming experimental evaluations of new BMG alloys.

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Carbon Technology: Greenmill/Rodding

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Morten Sorlie, Elkem Aluminium ANS; Todd W. Dixon, Conoco Phillips Venco; Travis J. Galloway, Century Aluminum Company

Tuesday AM — Room: 8A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Marilou McClung, Century Aluminum of West Virginia

8:30 AM
Anodes Plant – The Next Step: Jean-Christophe Rotger[1]; Yann El Gahoui[1]; Magali Gendre[1]; Andre Pinoncely[2]; Jean-Francois Andre[2]; Jerome Morfoise[2]; *Andre Molin*[2]; [1]Alcan - LRF; [2]Solios

Investors in greenfield primary aluminium smelters nowadays often contemplate the construction and operation of one 300kA (or more) potline as a first phase followed by a second identical potline a few years later for a targeted metal production of 600 000 T/year. Alternatively the 2 potlines can be built in one single phase. In such situation there is a strong economic incentive, both CAPEX and OPEX, to consider a high capacity green anode plant, 60 t/h or more, rather than two "standard" 35t/h paste plants. This is now feasible thanks to the two major developments achieved in the last few years: the SCAP Rhodax® process for dry mix preparation and the IMC process for paste mixing and cooling. The start-up early 2005 of these 2 processes at 35t/h at respectively ALBA line 5, Bahrain and AOSTAR, China confirmed the high standard of operating and anode quality performance achieved. The equipment are already available for the design of one single line of 70 t/h capacity achieving the same performance standards.

8:55 AM
Dynamic Process Optimization(DPO) in Greenmill: *Raja Javed Akhtar*[1]; Saleh Ahmad Rabba[1]; Markus W. Meier[2]; [1]DUBAL; [2]R&D Carbon

Dubai Aluminium Company(DUBAL)is one of the largest single site aluminium smelters in the world producing 761000MT/year. It has its own carbon manufacturing plant with a production capacity of 370,000 anodes/year. As a management drive to optimise the current assets an exercise of Dynamic Process Optmisation was carried out in the Greenmill. R&D Carbon was chosen as the technical partner and worked along with DUBAL operational personnel during the three phases of the DPO. The process parameters were optimised systematically to reduce pitch content, increase paste throughput and increase anode density. A mobile pilot press was utilised to prepare samples for evaluation during the optimisation process. The optimised parameter settings were implemented on both the two anode production lines in the Greenmill following a successful 15 cell trial in the Potrooms. This paper describes the DPO methodology, highlights the results achieved in the Greenmill together with the subsequent improvement in anode quality and performance.

9:20 AM
Improve Carbon Plant Operations through Better Use of Data: *Keith Sinclair*[1]; Barry Sadler[2]; [1]Sinclair Associates, Inc.; [2]Net Carbon Consulting Pty Ltd.

Most Carbon Plants collect an enormous amount of data for control and improvement of plant operations, reporting and to characterize and assess anode quality for the customer. However, in the experience of the Authors, the way that data is used can be improved considerably - full value is rarely extracted from the information and several "data traps" are common: *Control decisions and actions are based only on comparisons to arbitrary specifications; *Averages are used inappropriately, e.g. with a lack of attention to variability in the data; *Data is over-aggregated; *Data is over-extrapolated; *Insufficient attention is given to the impact of sampling methods on the usefulness of data. This paper will demonstrate these data traps, describe their implications and suggest ways of avoiding these pitfalls using examples based on information from the sampling and testing of anode cores.

9:45 AM
Experience Report - Aostar Aluminium Co Ltd, China: Anode Paste Preparation by Means of a Continuously Operated Intensive Mixing Cascade (IMC): *Berthold Hohl*[1]; Youlai Wang[2]; [1]Maschinenfabrik Gustav Eirich GmbH and Company KG; [2]Aostar Aluminium Company Ltd

In 2003, Aostar Aluminium started the construction of a 250 000 t/y greenfield smelter near Meishan in Central China. The smelter uses GAMI 320 kA electrolysis cells. The affiliated anode plant was put into operation in January 2005. It is based on the newly developed Intensive Mixing Cascade (IMC) technology for anode paste preparation, combined with a vacuum vibrocompactor for anode forming. Design figures and current results of the plant operation are given. Typical features like self-adaptation to low raw material qualities and fluctuations of properties are described and discussed. Based on the experiences gathered at Aostar, the IMC process does not only significantly reduce both the investment (Capex) and operating costs (Opex) of an anode paste plant but also allows for the production of more than 60 t/h of anode paste in one single line. This perfectly fulfils the future requirements of the primary aluminium smelting industry.

10:10 AM Break

10:25 AM
A Study on the Quality Assessment of ETP Copper Anode Bars Used in Aluminum Reduction Process to Improve Their Life Cycle Time: *Y. V. Ramana*[1]; Rajnish Kumar[1]; [1]Hindalco Industries Ltd

Hindalco consumes about 1600 MTPA of ETP copper for production of 350,000 MTPA of aluminium metal. To reduce the consumption patterns several possible control measures were studied. The present paper deals with the study on quality parameters. Copper bars, in the aluminium reduction process are continuously exposed to different physico-chemical environments like flue gases emanating from pots, load bearing, exposure to high temperature, hot and cold working. To meet the demanding requirement of the complex environment, a comprehensive testing of physical, chemical, mechanical and metallographic properties has been introduced and narrowed down the impurities to trace levels. These measures have resulted in improving the life cycle time of the copper bar to a significant extent. The results have established significant relationships between different physico-chemical and mechanical properties, the patterns of different impurities, especially oxygen and their role in controlling the life cycle time.

Cast Shop Technology: Melt Treatment, Quality and Product Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Rene Kieft, Corus Group; Gerd Ulrich Gruen, Hydro Aluminium AS; Travis J. Galloway, Century Aluminum Company

Tuesday AM — Room: 7C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Benoit Commet, Alcan Centre de Recherches de Voreppe

8:30 AM
Jetcleaner: The Lower Cost Alternative for Degassing Metal: *Ghislain Henry Le Roy*[1]; Jean-Marie Bertrand Chateau[1]; [1]Novelis

"Jetcleaner" is an aluminum in-trough refining system recently developed by Novelis PAE. This paper reviews industrial performances, in particular using data gathered on extrusion billets casting at the BON-L casthouse in Pickering, Ontario and expert appraisement by Research Centre engineers: 1) efficient hydrogen removal, linked with some inclusion removal, as measured by Alscan and PodFa; 2) low investment and operating costs, thanks to a simple design, especially in the case of frequent alloy changes; 3) user friendliness features: *the Jetcleaner fits easily with reduced space requirement and fast installation; *little maintenance is required; *there is little metal retention between two casts; *environment constraints are respected. Starting from a concept patented in 1998, then developed in close collaboration with customers, the Jetcleaner has been well received by operators and cost-conscious casthouse managers in North America and Europe.

8:55 AM
Experimental and Numerical Study of Ceramic Foam Filtration: *Emilie Laé*[1]; Hervé Duval[2]; Carlos Rivière[2]; Pierre Le Brun[1]; Jean-Bernard Guillot[2]; [1]Alcan Centre de Recherches de Voreppe; [2]Laboratoire de Génie des Procédés et Matériaux, École Centrale Paris

Ceramic foam filtration is widely used to remove non metallic inclusions from liquid aluminium. Its performances have been largely studied in the literature and some discrepancies remain amongst the published results. Consequently, a research program was deployed to evaluate the performances of a range of CFF used under various conditions and to understand the inclusions capture mechanisms. On the first hand, laboratory trials were run on a filtration pilot under controlled thermal and hydrodynamic conditions. The filtration efficiency of the CFF was assessed by LiMCA measurements. Microscopic observations of the filter impregnated with solidified metal were carried out to know the repartition of the inclusions within the filter and their capture sites.On the other hand, the initial filtration efficiency as well as the inclusion capture sites were calculated by a two-dimension lattice-Boltzmann filtration model on different CFF, for various inclusion sizes. Results of this work are presented and discussed.

9:20 AM
A Novel Metal Treatment Process in Crucibles for Impurity Removal in Secondary Aluminum: *Gaston Riverin*[1]; Jean-François Bilodeau[1]; Claude Dupuis[1]; [1]Alcan Inc

Treatment of aluminium in crucible is well established in the smelter industry as the most efficient technology to remove alkali element such as lithium. The utilization of crucibles is common in Remelt and Recycling plants to transport metal recovered from dross recycling and scrap melting operations. In these cases, the metal is often contaminated with alkali elements, undesired contaninents. This paper presents the development work of an improved crucible treatment better adapted for metal recovered from remelt and recycling operations. This method, which involves utilization of flux to improve metal cleanliness and alkali elements removal, is specially designed to promote efficient dispersion of the reactant in the melt to achieve optimal performance. Laboratory work is explained covering new impeller design and dispersion methods to meet specific requirements for alkali, oxides and inclusions removal. Results from an industrial demonstration are detailed, including metal cleanliness measured by PoDFA and alkali removal efficiency.

9:45 AM Break

10:00 AM
Kinetics of an AlF_3 Aluminium Filter: *Harald Gorner*[1]; Thorvald Abel Engh[1]; Martin Syvertsen[2]; [1]Norwegian University of Science and Technology; [2]SINTEF

Kinetic models are proposed for the removal of Na and Ca by an active AlF_3 filter for aluminium melts. Experiments have been carried out using a setup and filter material that have shown its efficiency previously. Pure Al melts containing different levels of Na or Ca have been filtered to reveal the kinetics of the AlF_3 filter material. The surface area of the filter and the temperature of filtration have been varied in addition to the concentrations of Na and Ca. Changing only one parameter at a time in a series of three experiments while keeping the other parameters constant gave a good reproducibility. The removal of Ca and Na in the AlF_3 filter follows zero order kinetics with a transition to first order kinetics for low levels of Na and Ca. At low levels the removal rate becomes proportional to the concentration of Na and Ca.

10:25 AM
Gas Fluxing of Aluminum; Comparison of Computational Fluid Dynamics Models and Experiments: Autumn Fjeld[1]; *James W. Evans*[1]; Corleen Chesonis[2]; [1]University of California; [2]Alcoa Inc

Chlorine fluxing is an essential purification step in aluminum processing in which impurities such as Ca, Na, Li, and Mg are removed by bubbling a mixture of chlorine and argon gas through molten aluminum. The efficiency of impurity removal and control of toxic chlorine and chloride emissions are dependent upon the extent of gas dispersion or mixing, residence time of the bubbles, and surface area of the bubbles. Clearly, these gas phase parameters cannot be directly observed in molten aluminum, thus there are details of the fluxing process which are not fully understood. To gain further insight, two computational models have been developed to simulate the complex two-phase fluid dynamics of a rotary gas injection system. The results of these two modeling approaches are presented and analyzed and compared to experimental bubble measurements gathered using a capacitance probe, which detects bubbles in molten aluminum.

10:50 AM Break

11:05 AM
The Role of B Element on Refining Purity Aluminum: *Henghua Zhang*[1]; [1]Shanghai University

The effect of B element on the macrostructure and property of purity aluminum has been studied with Optical Microscopy and MTS. It is shown that although the single B element has little or none refining effect on the purity aluminum, it will obviously increase the refining effect and prolong the fading time when it was simultaneously added into the molten aluminum with Ti element. It is found from the analysis of SEM and thermodynamics that the refining mechanism of salts containing Ti and B elements on the purity aluminum are mainly contributed to the heterogeneous nuclei of TiAl3 particles reacted and dispersed in the melting. While the reacted (Al, Ti) B2 particle plays little or none role in refining purity aluminum. However, the fine (Al, Ti) B2 particle will reduce the size of TiAl3 since the TiAl3 nucleates and grows along the (Al, Ti) B2 particle.

11:30 AM
A Method for Measurement and Control of Boride Agglomeration in Grain Refinement of Aluminum: *Rein Vainik*[1]; John Courtenay[2]; Holm Boettcher[3]; Lennart Backerud[1]; [1]Opticast Aluminium AB; [2]MQP Limited; [3]AMAG

The method for grain size control during casting of billets and slabs, as developed by Opticast Aluminium AB, is now applied at several cast houses. Apart from savings and increased quality of the cast product, several effects have been encountered, which have a large impact on the grain

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refinement efficiency. Grain refining performance between rods from different manufacturers varies considerably. The structure of the master alloy is important, and determines whether the particles present in the master alloy will have enough time to disintegrate/dissolve. The rod addition point in relation to melt flow conditions are other important factors. A new method has been developed that shows the degree of agglomeration of borides and that can be used routinely in the cast house to evaluate this effect. This is used to optimise the casting process to render a fine grained structure in the castings at the lowest possible addition rate.

Characterization of Minerals, Metals and Materials: Structural Engineering Materials I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee

Program Organizers: Jiann-Yang James Hwang, Michigan Technological University; Arun M. Gokhale, Georgia Institute of Technology; Tzong T. Chen, Natural Resources Canada

Tuesday AM — Room: 206A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Toru H. Okabe, University of Tokyo; Ann M. Hagni, CTL Group

8:30 AM

Adiabatic Shear Bands Associated with Plug Formation and Penetration in Ti-6Al-4V Targets: Formation, Structure, and Performance: *Fabiola Martinez*[1]; Erika V. Esquivel[1]; Maribel I. Lopez[1]; Sarah M. Gaytan[1]; David A. Lopez[1]; Amanda Ramirez[1]; Diana A. Ramirez[1]; Priscilla A. Guerrero[1]; Larry E. Murr[1]; B. E. Schuster[2]; [1]University of Texas; [2]U.S. Army Research Laboratory

A series of electron beam, cold hearth, single melt Ti-6Al-4V plates (nominally 2.5 cm thick) impacted by 2 cm diameter, 4340 steel projectiles (with 54 g nominal mass) at velocities ranging from 0.633 to 1.027 km/s were sectioned along the impact axis and examined by optical metallography, SEM, and TEM to characterize the formation, growth, and microstructure of adiabatic shear bands which facilitate plug formation and projectile perforation by dynamic recrystallization (DRX) within the shear bands; allowing for solid-state flow. The localized DRX of the projectile and projectile-target interaction phenomena were also examined using elemental X-ray mapping of the targets and projectiles in the SEM. Near ballistic limit velocities, the shear bands propagate towards the rear of the target where they coalesce and grow to provide a cylindrical (DRX) flow regime for the plug volume (Supported by ARL Prime Contract DATM05-02-C-0046, Amendment 16).

8:55 AM Invited

Some Aspects of Free-Machining Stainless Steel with Low Carbon: Julian Ciupitu[1]; *Dana Daisa*[1]; [1]METAV

This paper-work shows some aspects regarding the obtaining, structure and properties of a free-machining Cr-Ni stainless steel using as referrence AISI 304 (annealed condition). Better machinability is the result of adding Sulphur up to 0,10% and restriction of Carbon content to a maximum of 0,03%. Sulphur was added into the melt as Fe-S master alloy, in a vacuum induction furnace. Secondary structures were examined with SEM microscope and RX diffraction. The electrochemical properties were investigated by cyclic voltametry in sulphuric acid 1N, at 22°C and a polarization rate of 100mV/sec. The tensile properties were determined. The presence and the distribution of sulphides (mainly MnS), almost no carbides in a γ- matrix, have a double effect on machining processes: chip-breaking and lubrication at high cutting speeds. The obtained free-machining stainless steel presents a good combination of technological (machinability) and functional (corrosion resistance) properties without major lowering of tensile strength.

9:20 AM

Laser Generation of Acoustic Waves in Different Laser Power Regions from Rene 77 Superalloy: Shih-Jeh Wu[1]; *Chen-Ming Kuo*[1]; Chun-Hung Lin[1]; Shian-Ching Jason Jang[1]; Dong-Yih Lin[1]; [1]I-Shou University

It is well known that the amplitude of laser generated ultrasonic signals can be significantly enhanced by increasing the power density in the laser pulse such that material ablation occurs. This leads to a direct increase in the signal-to-noise ratio of laser-based systems for the remote generation and detection of ultrasound. In this research an Nd-Yag pulsed laser (532 and 1064 nm wave length) will be used to generate ultrasonic waves from Rene 77 superalloy for the idea power region (either thermal-elastic or lower ablative) to be used to remote characterization of mechanical material properties in terms of the signal-to noise ratio and frequency response. The ultrasound signal is picked up by a Fabry-Perot interferometer and ultrasonic transducer. There were also some modeling works for the laser-acoustics under different power regions. The experimental results will also be compared to these analytical models.

9:45 AM

Ultrasonic Detection of Hydride Blisters in Zr-2.5Nb Pressure Tube: *Yong-Moo Cheong*[1]; Young-Suk Kim[1]; [1]Korea Atomic Energy Research Institute

Since Zr-2.5Nb pressure tubes have a high risk for the formation of blisters during their operation in pressurized heavy water reactors, there has been a strong incentive to develop a method for the non-destructive detection of blisters grown on the tube surfaces. However, because there is little mismatch in acoustic impedance between the hydride blisters and zirconium matrix, it is not easy to distinguish the boundary between the blister and zirconium matrix with conventional ultrasonic methods. This study has focused on the development of the ultrasonic method to detect the hydride blisters formed on Zr-2.5Nb pressure tubes. Hydride blisters were grown on the outer surface of the Zr-2.5Nb pressure tube using a cold finger attached to a steady state thermal diffusion equipment. An ultrasonic velocity ratio method as well as conventional ultrasonic parameters with immersion technique was developed to detect small hydride blisters on the Zr-2.5Nb pressure tubes.

10:10 AM Break

10:20 AM

Iron Oxide Reduction with Conventional and Microwave Heating under CO and H2 Atmospheres: *Jiann-Yang James Hwang*[1]; Xiaodi Huang[1]; Shaolong Qu[1]; Yongqing Wang[1]; Gerard T. Caneba[1]; [1]Michigan Technological University

Microwave heating has been proved an effective method to rapidly heat and reduce iron oxide with carbon. However, potential use of microwave heating for iron oxide reduction in the steel industry may include the use of coal, CO and/or H2 as the reducing agent. Other heating method may be required to combine with the microwave heating to provide the total energy need. A special microwave device has been designed and built to facilitate the investigation. With this device, iron oxides can be heated by an electric resistance heating, microwave heating, or their combinations. The incoming CO and/or H2 also can be preheated to simulate a potential utilization of recovered heat. The iron oxide weight and temperature can be monitored during an entire reduction period. A series of reduction experiments have been conducted. The results will be presented and discussed.

10:45 AM

Development of Ore-Coal Composite Pellets for Sponge Ironmaking: *B. B. Agrawal*[1]; G. I.S. Chauhan[1]; [1]RDCIS, SAIL

Iron ore fines are one concern for Indian economy and ecology. A possible avenue is to use these fines for sponge ironmaking. Work has been carried out to pelletize these fines with coal and a suitable binder, and cure them in ambient atmosphere to retain in-situ reductant. Pellets were characterized for physical-strength and metallization. Critical factors were granulometry of pellet-mix and percentage of coal and binder. In laboratory tests, pellets got metallized faster compared to lump-ore. Large-scale trials were conducted in a 12-meter long rotary- kiln plant. For 90% metallization, required residence time was only 60 minutes against 240 minutes for lump-ore. Coal requirement also decreased. Investigations established the fast reduction nature of pellets. This is due to reduction-reactions occurring simultaneously at many sites within composite pellets, along with diffusion of reducing gases from-surface-to-core. The developed process is a value addition in the conventional pelletmaking, and is environment-friendly.

11:10 AM
Microstructural and Textural Characterization of Brazilian Iron Ores for Quality Control in Ironmaking–A Geometallurgical Approach: Claudio Batista Viera[1]; *Varadarajan Seshadri*[1]; Carlos Alberto Rosiere[1]; [1]Universidade Federal de Minas Gerais

In this paper, the structures and textures of some of the iron ores from Qualdrilatero Ferrifero of the Minas Gerais state of Brazil are presented. The correlation between structure and characteristics important for suitability as charge in the blast furnace, such as decrepitation, degaradation, and reducibility based on microstructure as well as the importance of magnetic susceptibility tests in the evaluation is emphasized. It is demonstrated that granoblastic, mainly martitic ores occurring in geological low strain areas from this region display low decrepitation and degradation during reduction. On the other hand, lepidoblastic ores with strong orientation in the structure, anisotropic ores and those consisting of substantial specularite display higher indices of decrepitation and degradation during reduction. These structural and geological characteristics are also important in agglomeration processes such as sintering and pelletizing.

11:35 AM
Characterization of Softening Behavior of Pellets, Sinters and Lump Ores in Ferrous Smelting Processes: *Florian Kongoli*[1]; I. McBow[1]; S. Llubani[1]; Robert D. Budd[1]; [1]FLOGEN Technologies Inc

One of the most important properties of pellets, sinters and lump ores in ferrous smelting processes is their softening behavior along with temperature increase. A good pellet or sinter need to have an optimal softening behavior in order to respond the strict requirements for a process without problems and high productivity. This behavior becomes even more important when various new minor components come with the primary materials and influence it in various ways that could potentially cause several problems. In this work it is discussed the characterization of the softening behavior of some pellets and sinters through a unique physical modeling that reflect the physical properties of the matter and predict their softening behavior as a function of various furnace parameters and conditions.

Computational Thermodynamics and Phase Transformations: Atomic Modeling of Solid-Liquid Structures

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Dane Morgan, University of Wisconsin; Corbett Battaile, Sandia National Laboratories

Tuesday AM	Room: 210A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Christopher Mundy, Lawrence Livermore National Laboratory; Anton Van der Ven, University of Michigan

8:30 AM Invited
Using Molecular Dynamics Simulation to Study Nucleation Kinetics: *David T. Wu*[1]; [1]Yale University

Nucleation is important for phase transformations and microstructural evolution, but a predictive theory for its rate requires detailed understanding of the interfacial free energy (γ), which is difficult to measure for liquid-solid interfaces but can be estimated, e.g., by analyzing molecular dynamics (MD) simulations at equilibrium or fitting nucleation theory to experimental results. Both approaches have pitfalls, however: in the former, γ for a planar interface may not be applicable to critical nuclei with large curvature, while in the latter, different assumptions about γ's dependence on temperature or curvature may influence the fit. An alternative, brute-force approach is to simulate nucleation kinetics using MD, but this also has limitations due to possible interaction of nucleation with growth or coarsening; thus its success depends on how the computational results are interpreted. In this talk, I will discuss recent advances in designing and interpreting MD runs to study nucleation kinetics.

9:00 AM Invited
The Properties of Steps at Faceted Crystal-Melt Interfaces from Molecular-Dynamics Simulations: Dorel Buta[1]; *Mark D. Asta*[1]; Jeffrey J. Hoyt[2]; [1]Northwestern University; [2]Sandia National Laboratories

The morphologies and growth rates of crystals grown from the melt are highly sensitive to the anisotropies of solid-liquid interfacial free energies and mobilities. In systems with faceted crystal-melt interfaces these anisotropies are governed by the intrinsic properties of steps, and their interactions. Molecular-dynamics simulations have been employed to study the properties of steps at 111 faceted solid-liquid interfaces for the Stillinger-Weber model of Si. Step mobilities are derived from non-equilibrium isothermal crystallization simulations and the resulting mobilities are found to be in good agreement with measured values for large step separations. The step mobility is found to decrease with decreasing step separations, an effect attributed to a reduction in step width resulting from step interactions. Step fluctuations are measured in equilibrium simulations and analyzed in an effort to determine the magnitude of the step stiffness and interaction parameters.

9:30 AM
Crystal-Melt Interfacial Properties in Lennard-Jones Alloys: *Chandler A. Becker*[1]; Mark D. Asta[1]; Jeffrey J. Hoyt[2]; David L. Olmsted[2]; Stephen M. Foiles[2]; [1]Northwestern University; [2]Sandia National Laboratories

A primary factor in determining alloy solidification rates and morphologies is the magnitude of the crystal-melt interfacial free energy and its associated crystalline anisotropy. Much of the detailed information concerning these interfacial properties in metals comes from atomic-scale computer simulations, focused primarily on elemental systems. Factors controlling compositional dependencies of crystal-melt interface properties in alloys are far less developed. We are using Molecular Dynamics and Monte-Carlo simulation techniques to extract the stiffness and structure of crystal-melt interfaces in model Lennard-Jones alloys at several compositions. Interface stiffnesses at several interface orientations are extracted from equilibrium simulations of interface fluctuations, from which the anisotropic interfacial free energy is computed. The simulations are analyzed using the Gibbs-adsorption theorem to better understand the compositional dependence of interfacial free energies in these model alloy systems.

9:50 AM
Liquid Metal Embrittlement of Grain Boundaries in Metallic Alloys: A Systematic Simulation Investigation: *Ho-Seok Nam*[1]; Mikhail I. Mendelev II[2]; David J. Srolovitz[1]; [1]Princeton University; [2]Ames Laboratory

There are many examples in which a liquid metal in contact with a polycrystalline solid develops a deep liquid groove at the intersections of the grain boundaries and the solid-liquid interface. In some cases such as Al-Ga and Cu-Bi, the liquid film penetrates deep into the solid along the grain boundary and leads to brittle fracture under the influence of even modest stresses. We have performed a series of molecular dynamics simulations using a set of binary embedded atom method potentials that were tuned to reproduce phase diagrams characteristic of systems exhibiting liquid metal embrittlement. We report on a series of molecular dynamics simulations of how liquid films propagate into grain boundaries at the atomic-level scale as a function of thermodynamic properties of the liquid species such as solubility, diffusivity, and melting point. These results are compared with general trends gleaned from a series of experimental studies in the literature.

10:10 AM Break

10:30 AM Invited
Simulating Fluid Phase Equilibria of Water from First Principles: *Christopher Jay Mundy*[1]; J. Ilja Siepmann[2]; Matthew McGrath[2]; I.-F. Will Kuo[1]; [1]Lawrence Livermore National Laboratory; [2]University of Minnesota

Effcient Monte Carlo algorithms and a mixed-basis set electronic structure program were used to compute from first principles the vapor{liquid coexistence curve of water. A water representation based on the Becke-Lee-Yang-Parr exchange and correlation functionals yields a saturated liquid density of 900 kg/m3 at 323 K, and normal boiling and critical temperatures of 350 and 550 K, respectively. An analysis of the structural and electronic properties of the saturated liquid phase shows an increase of the asymmetry of the local hydrogen- bonded structure despite the persistence of a four-fold coordination,and decreases of the molecular dipole moment and of the spread of the lowest unoccupied molecular orbital with increasing temperature.

11:00 AM Invited
The Effect of Stress on Melting and Freezing in Nanopores: *Jeffrey J. Hoyt*[1]; [1]Sandia National Laboratories

Liquids contained in open porous materials generally exhibit a melting point depressed from the bulk equilibrium value. By contrast, for fluids entrapped in closed pores, such as those formed during ion implantation, a melting point elevation is usually observed. In this work the thermodynamics of stressed solids is used to explain the melting point elevation in enclosed, spherical nanopores. The model includes the effects of liquid pressure, the elastic strain energy in both the pore solid and the surrounding matrix material, the volume change on solidification and the phenomenon of premelting. Results from the analysis agree favorably with experimental observations of melting point elevation in ion implanted Pb droplets in Al.

11:30 AM
Comparison of Grain Boundary and Solid-Liquid Interface Migration: *Mikhail I. Mendelev*[1]; Hao Zhang[2]; David J. Srolovitz[2]; James R. Morris[3]; [1]Ames Laboratory; [2]Princeton University; [3]Oak Ridge National Laboratory

The goal of this study was to compare the mechanism of the solid-liquid interface (SLI) and grain boundary (GB) migrations in the same pure system. In order to do this, we performed molecular dynamics simulation of the SLI and GB motion in pure Al and Ni. Elasticity was used as driving force in both cases. In order to test the reliability of our results in the case of the solid-liquid interfaces we also used overheating/overcooling as source of driving force. We found that both GB and SLI mobility are strong functions of the interface inclination. Surprisingly, the comparison of these two mobilities showed that the GBs move faster in pure system than SLIs. We will discuss possible explanations for this effect.

Deformation and Fracture from Nano to Macro: A Symposium Honoring W. W. Gerberich's 70th Birthday: Nanoscale Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: David F. Bahr, Washington State University; James Lucas, Michigan State University; Neville R. Moody, Sandia National Laboratories

Tuesday AM	Room: 214D
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Robert Keller, National Institute of Standards and Technology; C. Barry Carter, University of Minnesota

8:30 AM Invited
Modeling Size Effects in Micropillar Samples: *Peter Martin Anderson*[1]; Julia R. Greer[2]; William D. Nix[2]; [1]Ohio State University; [2]Stanford University

Compression testing of gold pillars with submicron diameters shows flow strengths as large as 50 times that of bulk counterparts. Corresponding stress-strain traces at uniform strain rate show rapid build-up to a peak stress, followed by repeated release and build-up of stress. A cellular-automaton model is proposed to determine if these results can be explained by the evolution of dislocation content inside a deforming micropillar. The model is based on discretizing pillars into cells with prescribed initial dislocation density and distribution of dislocation source lengths. The dislocation density in each cell evolves due to source operation, dislocation breeding, exchange of dislocation content, and load shedding. Smaller pillars are inherently different due a distribution of source lengths that is truncated by the pillar size and also the inability to contain bursts of dislocation activity. The model predictions and assumptions are assessed in light of recent experimental stress-strain data.

8:50 AM
Flow and Fracture of Isolated Nanostructures: *William Mook*[1]; Julia Deneen[1]; Michael S. Lund[1]; Andrew M. Minor[2]; Steven L. Girshick[1]; C. Barry Carter[1]; William W. Gerberich[1]; [1]University of Minnesota; [2]Lawrence Berkeley National Laboratory

Characterizing the mechanical response of isolated nanostructures is vitally important to fields such as microelectromechanical systems (MEMS) where the behavior of nanoscale contacts can in large part determine system reliability and lifetime. To address this challenge directly, both scanning probe microscope (SPM) and transmission electron microscope (TEM) based nanoindenters have been used to compress structures such as spherical nanoparticles and lithographed nanodots between the substrate and a diamond tip. The tested materials, which include both metals and semiconductors, initially exhibit an elastic response followed by plastic flow during compression. Further compression induced fracture in semiconductor structures. The flow stress and, where applicable, fracture toughness of the structures as a function of shape, size and stress state will be presented and modeled.

9:05 AM
Indentation of Nanoparticles, Nanowires and Nanotubes: Antonio Rinaldi[1]; Veronica Salgueirino-Maceira[1]; Miguel Correa-Duarte[1]; Yen T. Yeh[1]; *Pedro Peralta*[1]; Karl Sieradzki[1]; Cody Friesen[1]; [1]Arizona State University

This talk reports preliminary experimental results and observations concerning selected elementary nanostructures. Nanoindentation is used to investigate the mechanical properties of composite Co-SiO2 nanoparticles between 100nm and 300nm in diameter. Metallic nanowires and nanotubes made of Cu or Ni and having 100nm average diameter and variable length between 500nm and 1000nm were also synthesized and tested. In many tests it was possible to use the nanoindenter to image the nanoelements after indenting, which provided extra information about the contact. Data analysis is crucial to filter out the effect o the substrate from the load-penetration curve. Experimental data capture "structural" properties. For example, the Co-SiO2 nanoparticles appear to have a characteristic load penetration curve, whose shape reflects the composite structure and consists of three distinct zones marked by sharp and consistent slope changes during loading. More experiments and simulations are being conducted to validate and explain the results.

9:20 AM
Deformation Behavior of Nanoporous Gold: *Erica T. Lilleodden*[1]; Cynthia A. Volkert[1]; [1]Forschungszentrum Karlsruhe

Here we present a combined nanoindentation and microcompression study of the deformation behavior of nanoporous gold. This material is made by electrochemical dissolution of AuAg sheets with a nominal grain size of 100 μm and results in a structure with pore and wall size on the order of 20 nm. The relevant mechanisms of plasticity in this fine-structured, coarse-grained material have be ascertained from the load-displacement measurements, in combination with microstructural characterization. The observed behavior will be discussed in terms of dislocation mechanisms and the availability of sources and sinks due to the high surface area and small cell wall size. Additionally, the measured elastic modulus and the relative brittle versus ductile behavior will be described in terms of the loading configuration and size of the deforming volume.

9:35 AM

Deformation Mechanisms in Incoherent Nanolayered Metallic Composites: *Srinivasan G. Srivilliputhur*[1]; R. G. Hoagland[1]; A. Misra[1]; [1]Los Alamos National Laboratory

A metallic multilayer composite of soft metals like Cu and Nb possess near theoretical strength when the layer thickness are less than 20nm. Deformation of these materials involves single dislocation crossing the incoherent Cu-Nb interface, which is responsible for the extraordinary strength these multilayers can achieve. Rolling experiments in these multilayers show that large reductions in thickness (> 100%) are possible but become negligible when the individual layer thickness is reduced to 4nm or less. We describe results of molecular dynamics simulations that explore the mechanisms of slip transfer through incoherent Cu-Nb interfaces. These results suggest a mechanism for shear instabilities in very thin-layered composites. (This research was sponsored by Office of Basic Energy Sciences, US Department of Energy).

9:50 AM

Tensile Strength and Ductility of Cu/Nb Multilayered Films: *J. Greg Swadener*[1]; Amit Misra[1]; Yun-Che Wang[1]; Xinghang Zhang[2]; [1]Los Alamos National Laboratory; [2]Texas A&M University

Multilayered thin films exhibit enhanced mechanical properties, such as ultra-high yield strength, but limited tensile ductility and fracture toughness. Tensile tests were conducted to examine the work hardening behaviour and the underlying deformation mechanisms. Free standing films of alternating Cu and Nb layers were synthesized by sputter deposition. The individual layer thickness was controlled for each film and varied from 5 to 100 nm. For decreasing layer thickness, an increase in yield strength was measured. The strength increase and work hardening was not in accordance with the Hall-Petch relation, but was consistent with confined single dislocation slip. For films with 100 nm layers, 4% tensile elongation was achieved. However, tensile ductility decreased with decreasing layer thickness. In addition, the elastic modulus of each film was determined from strain measurements using a laser extensometer. The modulus agrees with expectations for a composite of textured Cu and Nb layers.

10:05 AM Invited

Nanomechanical Characterization on Piezoelectric Nanowires: *Scott X. Mao*[1]; Minhua Zhao[1]; Chuanbing Jiang[2]; Shouxin Li[2]; [1]University of Pittsburgh; [2]Institute of Metal Research

Piezoelectric ZnO nanobelts were successfully synthesized by evaporating the ZnO powders at high temperatures without the presence of catalyst. Morphology analysis shows the nanobelts have a rectanglelike 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 the ZnO single nanobelt by using AFM indenter probe with radius < 25nm (Digital Instruments Nanoscope IIIa, tapping mode) and cubic corner tip with radius <40nm (Hysitron Triboscope, STM mode). It was shown that the indentation size effect was still obvious for the indentation depth under 50nm. Besides, the sharper the indentation tip, the higher the nanoindentation hardness. It is also demonstrated that nanomaching is possible on nanobelt using AFM tip. Piezoelectricity measurement was carried on the single piezoeletric nanobelt under electric force microscope. Scaling effect on piezoelectricity was found.

10:25 AM Break

10:45 AM

Deformation Mechanisms in Fine-Grained Niobium: *Alexis C. Lewis*[1]; David van Heerden[2]; Timothy P. Weihs[3]; [1]Naval Research Laboratory; [2]Reactive NanoTechnologies, Inc; [3]Johns Hopkins University

Creep rates in fine-grained Nb were measured at 600°C using free-standing Cu/Nb polycrystalline multilayered films. For specimens with grain sizes ranging from 0.5 µm to 5 µm, two distinct regimes were observed. At high stresses, the stress dependence, grain size dependence and activation energy for creep are consistent with Power Law creep, with an average stress exponent of 3.5. At low stresses, creep rates exhibited a linear dependence on stress, and an inverse linear dependence on grain size. A model is presented for a vacancy generation-controlled creep mechanism, whereby deformation rates are controlled by the rate of vacancy generation at or near grain boundaries. The proposed model is consistent with experimental observations of stress and grain size dependence, as well as the activation energy measured for creep.

11:00 AM

Deformation Behavior of Nanolayered Metal-Ceramic Laminated Composites: Xin Deng[1]; *Nikhilesh Chawla*[1]; M. Koopman[2]; Krishan K. Chawla[2]; J. Bai[3]; Camden R. Hubbard[3]; Jinn P. Chu[4]; [1]Arizona State University; [2]University of Alabama at Birmingham; [3]Oak Ridge National Laboratory; [4]National Taiwan Ocean University

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 scanning and transmission electron microscopy. Residual stresses were characterized by x-ray diffraction using a high-energy x-ray synchrotron source. The mechanical properties of the layered materials were characterized by nanoindentation and tensile testing. The influence of layer thickness on hardness and Young's modulus of individual layers was quantified by nanoindentation. Finite element modeling was conducted to elucidate the stress state under indentation. The important implications of the heterogeneous stress state, during indentation, to measurements of modulus and hardness will be discussed.

11:15 AM

Mechanical Behavior of Severely Deformed NbZr and Ti Using Equal Channel Angular Extrusion (ECAE): *Guney Guven Yapici*[1]; Ibrahim Karaman[1]; Ralph Dieckmann[2]; Gagan Singh[1]; Hans Maier[2]; [1]Texas A&M University; [2]University of Paderborn

Apart from being technologically important materials for the biomedical industry, NbZr (bcc) and Ti (hcp) are suitable for investigating the deformation mechanisms during severe plastic deformation. Equal channel angular extrusion (ECAE) is applied by extruding bulk billets through two perpendicular channels of equal cross section achieving simple shear in a thin layer at the crossing plane of the channels. Deformation mechanisms of the extruded samples are investigated through extensive TEM analysis exhibiting the microstructure at the nano to micro regime. Post-processing mechanical response of the extruded samples is investigated to reveal the processing-microstructure-property relationships. Series of experiments are conducted with the aim of capturing the yield locus evolution during and after ECAE processing. This enables us to demonstrate the effect of ECAE on the yield surface evolution compared to conventional deformation methods. It will also help in developing accurate hardening models to predict the mechanical properties of severely deformed materials.

11:30 AM

Advanced Nanoparticle-Reinforced Composite Materials in Air-Blast and Vibration Damping Protection: *Liya Bochkareva*[1]; [1]University of Sheffield

This paper relates to vibration damping and air-blast protection issues in engineering structures by nanotechnology-based solutions through the utilization of nanomaterials that dissipate a substantial fraction of the vibration energy that they receive. The mechanisms involved in such materials are analyzed and the relevance to damping is identified via both computational and experimental benchmarks. Computational work is concentrated on hierarchical multiscale modelling of damping/friction behavior as a function of frequency, amplitude and temperature. The novel concept of nanoparticle-based damping technology shows that a molecule-level mechanism can considerably enhance vibration damping and dynamic of aerospace components (fan blades) via enhanced energy dissipation because of large surface-to-volume aspects in nanoparticle-reinforced composite material, large damping energy sources for friction and slip-stick motion at interfaces of matrix and nanoparticle. The materials offer the potential to further reduce the mass and dimension, increase performance, and reduce vibrations in wide-ranging applications in areas of transportation.

11:45 AM

Mechanical Behavior of Mg-Alloys Subjected to Severe Plastic Deformation: Grigoreta M. Stoica[1]; *Liang Wu*[1]; Hao Hsiang Liao[1]; Andrew E. Payzant[2]; Sean R. Agnew[3]; John E. Spruiell[1]; Lijia Chen[4]; Peter K. Liaw[1]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]University of Virginia; [4]Shenyang University of Technology

The influence of severe plastic deformation [SPD] on the mechanical behavior of Mg alloys, ZK60 and AZ31, was investigated under monotonic and cyclic loading. The extruded material was severely deformed through equal-channel-angular processing [ECAP] at 260°C and 200°C, respectively, or axially compressed at room and intermediate temperatures. Some microstructural-characterization techniques, including X-ray diffraction [XRD], the optical and electronic scanning microscopy [SEM] were used to study the influence of SPD on the grain sizes refinement, the elastic-microstrains, and textures evolution. The tensile properties and fatigue lives were found to be dependent on preferential orientations of the grains. The microstrain build up in the samples deformed by compression at room temperature has a detrimental effect on mechanical behavior, relative to the ECAP-ed samples. On the other hand, the microstrain relaxation in ECAP-ed samples indicates that the recovery/recrystallization processes compete with the plastic deformation, enhancing the ductility.

Fatigue and Fracture of Traditional and Advanced Materials: A Symposium in Honor of Art McEvily's 80th Birthday: Fatigue and Fracture III

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee

Program Organizers: Leon L. Shaw, University of Connecticut; James M. Larsen, U.S. Air Force; Peter K. Liaw, University of Tennessee; Masahiro Endo, Fukuoka University

Tuesday AM — Room: 216
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: James M. Larsen, U.S. Air Force; Winston Oluwole Soboyejo, Princeton University

8:30 AM Invited

Fatigue Crack Nucleation in Metals and Alloys: Morris E. Fine[1]; *Shrikant P. Bhat*[2]; [1]Northwestern University; [2]Mittal Steel

Over the years, many theories and models have been proposed to explain how fatigue cracks nucleate over many cycles of applied load (strain). In the model originally proposed by Mura and coworkers, fatigue crack initiation is considered a nucleation process in that an energy barrier due to the formation of new surfaces must be overcome. Therefore, by minimizing the Gibbs free energy change, the critical number of cycles required to nucleate fatigue cracks can be estimated. In a previous contribution, the present authors demonstrated the validity of this model with data from commercial purity iron and a high strength low alloy steel. The present paper extends these concepts to other metals and alloys and in particular, explores the applicability to fcc metals, metal-metal oxide composites, influence of temperature and environment, and to subsurface nucleation of fatigue cracks.

8:55 AM

Mechanics Based Probability Modeling for Minimum Life Estimation of S-N Data: *D. Gary Harlow*[1]; [1]Lehigh University

Fatigue life prediction has historically been a challenging problem analytically as well as experimentally. Current analyses are predominantly statistical and do not adequately reflect long-term operating conditions. Recent observations of giga cycle fatigue have suggested that there may be multiple mechanisms for nucleation and growth of fatigue cracks. One has been associated with surface damage and another with internal inclusions. These mechanisms make accurate estimation and prediction of the stress dependent minimum life difficult. A simple crack growth based probability model is used to account for plausible, readily identifiable contributors to the observed minimum life, e.g., manufacturing and material properties. A connection between the proposed crack growth model and S-N response is shown. The effect on the minimum life from the contributions of multiple damage mechanisms is examined. The methodology is demonstrated through the analysis of two extensive sets of S-N data, 2024 aluminum alloy and SUJ2 bearing steel.

9:20 AM

Microstructure-Based Modeling of Crack Growth in Particle Reinforced Composites: *Adarsh Ayyar*[1]; Nikhilesh Chawla[1]; [1]Arizona State University

The crack growth behavior of particle reinforced composites is determined by several factors, such as volume fraction, particle size, and particle morphology. Numerical modeling, such as finite element modeling, can be used to simulate and understand crack growth behavior. In SiC particle reinforced Al matrix composites, for example, it is customary to represent the complex morphology of the SiC particles with simple shapes such as circles and ellipses. Because crack growth is significantly influenced by the morphology of the reinforcement particles, such approximations may lead to erroneous results. In this paper, the effect of SiC particle morphology on crack growth was studied. Two dimensional linear elastic fracture mechanics principles were used to propagate the crack and obtain the local stress intensity values. In addition, the effect of particle fracture on crack growth was also studied. Results from these analyses are compared with experimental observations of crack growth in these systems.

9:45 AM

A Physics-Based Creep-Fatigue Crack Growth Model for a Ni-Base Superalloy: Vikram Bhamidipati[1]; *Robert Tryon*[1]; [1]Vextec Corporation

A physics-based creep-fatigue intergranular crack growth model is proposed for a Ni-base superalloy at elevated temperature. An intergranular crack nucleation model under fatigue and a short crack growth model are described. The creep short crack model is two dimensional and captures changes in microstructural properties like grain size, defect spacing and grain boundary orientations as the crack grows. A HRR-type stress field solution is assumed to hold good in the region ahead of the crack tip. The principle damage mechanisms include cavity nucleation, growth and coalescence and grain boundary sliding.

10:10 AM Break

10:25 AM Invited

The Effect of Seismic Loading on the Fatigue Strength of Welded Joint: *Yoshiyuki Kondo*[1]; Kazuhiko Okuya[2]; [1]Kyushu University; [2]Kyushu Polytechnic College

Earthquakes sometimes give damages to steel structures. The structures which were not seriously damaged are still used after earthquake. The fatigue strength of these structures, however, might have been decreased due to the large cyclic loadings during an earthquake. In order to clarify the effect of seismic loading on the fatigue strength of welded joint, high cycle fatigue test after large straining in gross plasticity was performed. The cyclic application of large strain substantially decreased the fatigue strength. The large straining initiated a short crack at weld toe in low cycle fatigue manner, which acted as a trigger for the high cycle fatigue after seismic loading. The reduction of fatigue limit depended on the crack size. Although the short crack was formed by large straining in gross plasticity, the high cycle fatigue limit of short-cracked welded joint after large straining was determined by the ΔK_{th} considering short crack effect.

10:50 AM

Characterization of the Fatigue Crack Initiation Process in an $\alpha + \beta$ Titanium Alloy: *Christopher Szczepanski*[1]; Sushant K. Jha[2]; James M. Larsen[3]; J. Wayne Jones[1]; [1]University of Michigan; [2]Universal Technology Corporation; [3]AFRL/MLLMN

Low stress, long lifetime fatigue behavior of the alpha-beta titanium alloy, Ti-6Al-2Sn-4Zr-6Mo, has been characterized using ultrasonic fatigue. Fatigue lifetimes in the range of 10^6-10^9 for load ratios of R=0.05 were determined at 20 kHz and significant scatter, as much as three orders of magnitude, in the fatigue lifetimes was observed for this material. Fractographic analysis revealed that initiation occurred at one or more primary α grains. Across specimens, the crack initiation region varied in diameter from approximately 10 to 70 μm. No correlation between lifetime and size of the crack initiating region was observed, suggesting that crack initiation may play a significant role in very long fatigue lifetimes. Detailed microstructural analyses of crack initiation regions and observations of the microstructural dependence of crack initiation from ultrafast laser machined micronotches will be described in terms of the mechanisms of crack initiation in the very high cycle lifetime regime.

11:15 AM Invited
On the History of Fatigue Life Diagrams: From August Woehler till Today: *Hael Mughrabi*[1]; [1]University Erlangen-Nuernberg

Since August Woehler performed the first systematic fatigue tests one and a half centuries ago, the S-N curve (with a fatigue limit) has been the most widely used fatigue life diagram, extending, typically, to 10 million cycles. Interestingly enough, August Woehler never published a fatigue life diagram but only tables. Since then, different forms of fatigue life diagrams, based on strain rather than on stress, such as the Coffin-Manson plot or the total strain fatigue life diagram (which frequently exhibit a plastic strain fatigue limit), have become equally important. Through the cyclic stress-strain curve, these different types of fatigue life diagrams can be mutually transformed into one another. Currently, fatigue life diagrams extending into the ultrahigh cycle regime (more than a billion cycles) are of particular interest. These developments will be reviewed.

Fatigue and Fracture of Traditional and Advanced Materials: A Symposium in Honor of Art McEvily's 80th Birthday: Fatigue and Fracture IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Leon L. Shaw, University of Connecticut; James M. Larsen, U.S. Air Force; Peter K. Liaw, University of Tennessee; Masahiro Endo, Fukuoka University

Tuesday AM — Room: 215
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Morris E. Fine, Northwestern University; Reinhold H. Dauskardt, Stanford University

8:30 AM Invited
Mechanisms of Fatigue Damage Formation and Evolution in Zr-Based Bulk Metallic Glass: *Reinhold Dauskardt*[1]; Peter Hess[1]; Brian Menzel[1]; [1]Stanford University

The mechanisms of fatigue damage initiation and evolution in bulk metallic glasses are not well understood, limiting their use in structural applications. We present experimental and computational studies of initiation of fatigue damage obtained from stress-life experiments, and the growth of fatigue cracks measured under stable and transient cyclic loading conditions. The early stages of damage initiation and propagation resulting in the anomalously low reported fatigue endurance limits are discussed. To further elucidate the underlying shear processes, simulations of the effects of cyclic loading on the nature and propagation of shear bands was conducted using molecular dynamics computational models of a multi-component Leonard-Jones solid. The formation of shear bands and their evolution under the influence of alternating loads will be described. The evolution of fatigue damage was experimentally characterized in terms of both "small" and "long" fatigue crack growth rate behavior to elucidate the detailed mechanism of fatigue crack growth.

8:55 AM
Effect of Age-Hardening Condition on HCF Performance of Mechanically Surface Treated Al 2024: Tomasz Ludian[1]; *Wagner Lothar*[1]; [1]Clausthal University of Technology

The present work was aimed at evaluating the effects of shot peening and roller-burnishing on the fatigue performance of the well known age-hardening aircraft aluminium alloy Al 2024 (T6). Shot peening to full coverage was performed using spherically shot (SCCW14) with an average shot size of 0.36 mm and an Almen intensity of 0.20 mmA. Roller-burnishing was done using a conventional lathe and a hydraulically driven device with a 3 mm diameter hard metal ball pressed onto the specimen surface at a constant rolling force. After applying the various mechanical surface treatments, the changes in the surface and near-surface layer properties such as surface topography, micro-hardness and residual stress-depth profiles were determined. In addition to the mechanically surface treated specimens, electrolytically polished conditions were used to serve as reference. The fatigue results will be interpreted in terms of the process-induced changes in the resistances to crack nucleation and micro-crack growth.

9:20 AM
Effect of Localized Precorrosion on Initial Fatigue Damage Process in Al 7075-T6511: *Sang-Shik Kim*[1]; James Burns[2]; Richard P. Gangloff[2]; [1]Gyeongsang National University; [2]University of Virginia

In the present study, detailed micrographic and fractographic analyses were conducted on Al 7075-T6511, precorroded on L-S and L-T plane, respectively, in EXCO solution for 6 h, and fatigued at σmax of 150 and 240 MPa (R=0.1), respectively, in a controlled-moist environment with intermittent marker band cycling. The AFGROW fracture mechanics based program was used to predict the corrosion modified (CM)-equivalent initial crack size (EIFS), and the predicted CM-EIFS was compared with the measured CM-EIFS. The fatigue cracks in precorroded Al 7075-T6511 specimen were found to be initiated from the semi-elliptical shaped pit clusters with low a/c ratios. The AFGROW modeling of the fatigue life of precorroded Al 7075-T6511 specimens could provide reasonable CM-EIFS and full S-N relationships with account taken for varying a/c ratio of initial semi-elliptical surface crack during fatigue crack propagation and the initiation cycles, Ni, required to form a sharp crack from the precorrosion damage.

9:45 AM
Density of Fatigue Weakest-Links in an Al-Li Alloy: *Tongguang Zhai*[1]; [1]University of Kentucky

A hot cross-rolled Al-Li 8090 alloy was fatigued in different orientations at 20 Hz, room temperature, R=0.1, in air, using a self-aligning four-point bend rig. It was found that the number of cracks initiated on the sample surface varied with the maximum stress level applied in the fatigue tests and was a Weibull distribution function of this stress. Such a Weibull function represented the distribution of the strength of fatigue weakest-links (i.e., the preferred sites for crack initiation) under certain applied stress level in this alloy. This weakest-link strength distribution and the density of the fatigue weakest-links can be used as a material property. It was found that they were different when measured in different orientations in the 8090 alloy.

10:10 AM Break

10:25 AM Invited
Fatigue-Property Enhancements by Glass-Forming Metallic Films: *F. X. Liu*[1]; C. L. Chiang[2]; D. Smith[1]; J. P. Chu[2]; P. Rack[1]; P. K. Liaw[1]; [1]University of Tennessee; [2]National Taiwan Ocean University

Zr- and Cu-based glass-forming metallic films were deposited onto different substrates by magnetron sputtering. Four-point-bending fatigue tests were conducted on these coated materials. It was shown that the fatigue life and the fatigue-endurance limit of the materials could be considerably improved, depending on the films, the substrates, and the maximum applied stresses. Fractographic studies indicated the good adhesion between the film and the substrate. Surface-roughness measurements showed the improved surface conditions after film deposition. Residual stresses were determined by the curvature measurement. Analyses showed that the good adhesion of the film to the substrate, the improved surface conditions, and the compressive residual stress were the main reasons for the enhancement of the fatigue properties. The high strength and moderate bending ductility of the glass-forming metallic films might also play beneficial roles, revealing the deposition of the films as a novel and effective method to improve fatigue properties of structural materials.

10:50 AM
Fatigue and Fracture of LIGA Ni MEMS Thin Films: *Winston O. Soboyejo*[1]; [1]Princeton University

This paper presents the results of an experimental study of fracture and fatigue in LIGA Ni MEMS thin films. The mechanisms of crack nucleation and crack growth are elucidated under monotonic and cyclic loading. In-situ crack-tip strain measurements are also used to identify the extent to which traditional crack-tip fields can be used to characterize the crack driving forces. The results suggest that the crack-tip fields for LIGA Ni films should include a plasticity length scale to account for strain gradient plasticity effects. The fracture resistance curves and fatigue crack growth rate data are presented along with the underlying fatigue and fracture modes. These reveal that the mechanisms of crack growth behavior are influenced by stress state and microstructure effects. The nucleation of fatigue cracks is shown to be dominated by persistent slip band (PSB) induced oxide thickening mechanisms that give rise to surface/near-surface crack formation.

11:15 AM
Investigation of Fatigue in Nano-Layered Cu/Nb Thin Films: *Yun-Che Wang*[1]; John Greg Swadener[1]; Tobias Hoechbauer[1]; Tim Darling[1]; Richard Hoagland[1]; Amit Misra[1]; [1]Los Alamos National Laboratory

It has been shown nano-layered thin films exhibit ultra-high yield strength and large plastic deformation without formation of dislocation cell structures. Fatigue properties of these films are of interest. Multilayered Cu/Nb thin films with a nano-size individual layer thickness were manufactured via sputter deposition. We developed a novel experimental method for fatigue measurements with R = -1. Our experimental method employed a bimorph piezo actuator to drive a cantilever beam sample in a moderate vacuum environment. A feedback system using fiber optic measured displacement was utilized, and stress was determined. Resonant frequency shifts and SEM images of fatigued samples were studied to determine fatigue crack propagation. We obtained S-N curves for thin films with various individual layer thicknesses. By systematically studying the S-N curves versus individual layer thickness and comparing results from bulk materials, the effects of nano-size layers on fatigue endurance in thin films were obtained.

General Abstracts: Extraction and Processing Division: Copper and Nickel Hydrometallurgy

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Aqueous Processing Committee, TMS: Copper and Nickel and Cobalt Committee, TMS: Lead and Zinc Committee, TMS: Precious Metals Committee, TMS: Process Fundamentals Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Pyrometallurgy Committee, TMS: Recycling Committee, TMS: Waste Treatment and Minimization Committee, TMS: Materials Characterization Committee
Program Organizers: Thomas P. Battle, DuPont Company; Michael L. Free, University of Utah; Boyd R. Davis, Kingston Process Metallurgy

Tuesday AM	Room: 203A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Corby G. Anderson, Montana Tech of the University of Montana

8:30 AM
Fundamental Kinetics of the Ferrous Regeneration for Alternate Anode Reaction Technology in Copper Hydrometallurgy: *Emily A. Sarver*[1]; Maurice Fuerstenau[2]; Scot Sandoval[3]; Gregory Adel[1]; [1]Virginia Polytechnic Institute and State University; [2]University of Nevada, Reno; [3]Phelps Dodge Mining Company

The Fe(II) regeneration process is an important aspect of Alternate Anode Reaction Technology (AART) using a Fe(II)/Fe(III)-SO_2 (FFS) for copper hydrometallurgy. The process is basically Fe(III) reduction by $SO_{2(aq)}$, which is catalyzed by activated carbon particles. For the current work, experiments have been conducted to examine the fundamental kinetics of the process, including the primary reaction mechanism and the effects of four variable factors – carbon particle size, flow rate, and initial Fe(III) and SO_2 concentrations – on the Fe(II) regeneration rate. As expected, the regeneration reaction is mass transfer-controlled, and the rate is limited by the diffusivity of Fe(III), which is $1.1x10^{-7}$ cm^2/s under the tested conditions. Carbon particle size and initial Fe(III) are the most influential factors under the conditions tested. Additionally, initial SO_2 concentration has been determined to be insignificant to the reaction rate, and flow rate affects the reaction rate via its affects on diffusivity.

8:55 AM
Development of a Hydrometallurgical Process for Copper Concentrate Treatment by Sumitomo Metal Mining: Masaki Imamura[1]; *Kenji Takeda*[1]; Koji Ando[1]; Noriyuki Nagase[1]; Yasuo Ojima[1]; [1]Sumitomo Metal Mining

A novel hydrometallurgical process for copper concentrate treatment has been developed in Niihama Research Laboratories of Sumitomo Metal Mining Co., Ltd. The process has numerous advantages with respect to operational and environmental aspects. For example, copper is leached effectively from chalcopyrite concentrates by chlorine gas under atmospheric pressure. Then copper in the leach solution is separated from iron and silver by solvent extraction and electrowon to produce highly pure copper metal without further purification. Iron metal can be produced by electrowinning from the purified raffinate to avoid generating a large volume of goethite or hematite, which is a solid waste for iron rejection in common hydrometallurgical processes. This process also enables significantly efficient recovery of precious metals. Results in batch and continuous testworks are presented in the paper.

9:20 AM
Effect of Additives on the Electrolytic Processing of Dilute Effluents Containing Copper: Ran Ding[1]; Daniel Chapman[1]; *James W. Evans*[1]; Fiona M. Doyle[1]; [1]University of California

The use of copper as the interconnect material in integrated circuits has resulted in the generation of increasingly complex copper-bearing effluents in the semiconductor industry. Those effluents associated with copper electrodeposition or electroless deposition are usually concentrated, with extreme pH. Organic additives are present to allow superfilling of trenches and control the deposit quality. Effluents from chemical mechanical planarization usually contain only trace levels of copper, but may contain significant amounts of complexing agents. In principle, electrodeposition, especially with porous and particulate electrodes, is an attractive means for recovering copper from these effluents, to allow either recycling of the process streams or safe discharge. However, the effect that additives can have on the kinetics of electrodeposition has important ramifications on cell design. Here we report studies on the effect of additives such as polyethylene glycol and glycine, and the sensitivity of their effects to the presence of other electrolyte components.

9:45 AM Break

10:00 AM
The Effect of Additives on Morphology of Copper Electrodeposits from Halide Media: *Aphichart Rodchanarowan*[1]; Michael L. Free[1]; [1]University of Utah

The effect of organic additives on surface morphology of copper electrodeposit from halide media (0.10 mol/L CuCl, 4.0 mol/L NaCl, and 0.010 mol/L HCl) was studied under current controlled conditions. The copper was electrodeposited on a rotating disc electrode made from 99.999% pure copper. Surface roughness was characterized by a series of CCD camera images combined with computer analysis of the images. The effect of additives was analyzed for C10 TAB, Triton X 100, thiourea, polyethylene glycol, and gelatin. It was found that gelatin gave more uniform deposits compared to those obtained without any additives. Surface roughness evaluations were carried out by varying electrodeposition time, current density, and concentration for gelatin. The addition of gelatin helped to achieve smooth electrodeposit at long deposition time. The surface roughness in solution containing gelatin slightly increased with the increase in i/iL ratio. Gelatin concentration did not affect the change in the surface roughness.

10:25 AM
Electrodeposition of Copper from Halide Media: *Ravindra J. Bhide*[1]; Michael L. Free[1]; [1]University of Utah

The effect of mass transport on the surface roughness of copper electrodeposits, obtained from a bath containing 0.1 mol/L CuCl, 4.0 mol/L NaCl and 0.01 mol/L HCl was studied under controlled current conditions. Copper was electrodeposited on a rotating disc electrode made of 99.999% pure copper. Surface roughness was characterized using CCD camera images combined with their computer analysis. The roughness of the electrodeposits was examined as a function of rotational speed of the electrode, current density and pulsed current transients. It was found that the roughness of the deposit decreased with increase in rotational speed of the electrode. Applied current transients also affected the morphology of electrodeposits. For current controlled transients, lower i/iL values gave electrodeposits with lower surface roughness where i is applied current and iL is the limiting current density for given experimental conditions. Pulsing also gave smoother electrodeposits compared to those obtained under direct current conditions.

10:50 AM
High Aspect Ratio Nickel Structures Fabricated by Electrochemical Replication of Hydrofluoric Acid Etched Silicon: *Xi Zhang*[1]; K. N. Tu[1]; [1]University of California, Los Angeles

A technique for fabricating free-standing high-aspect-ratio Ni structures has been developed using wet Si processing. Si (100) was initially etched in HF solution to form macropores with micron-sized diameter. The aspect ratio can exceed 200: 1. Immersed in aqueous Ni solution containing no reducing agent but fluoride, metallic Ni was rapidly reduced onto the sidewalls by oxidizing the Si. With longer duration, the Si sidewalls were gradually converted to become total Ni while the deep macropore structure was still maintained. Electron microscopic analyses reveals that fluoride in solution helps dissolve the fully oxidized Si and leave the Ni deposits on the sidewall loosely arranged during the process, which can explain the replication. It is known that close-packed submicron Si pillars can be formed by oxidizing and subsequent etching of the macropore sidewalls. Treated in the same Ni solution, high-aspect-ratio Ni pillars were correspondingly emerged by replicating the original Si structure.

General Abstracts: Materials Processing and Manufacturing Division: Novel Processing Methods

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee, TMS: Global Innovations Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Thomas R. Bieler, Michigan State University; Ralph E. Napolitano, Iowa State University; Fernand D. Marquis, South Dakota School of Mines and Technology

Tuesday AM — Room: 211
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Thomas R. Bieler, Michigan State University; Robert E. Barber, Texas A&M University

8:30 AM
Spectral Methods for Capturing Crystallographic Texture Evolution during Large Plastic Strains in HCP Metals: *Xianping Wu*[1]; Surya Kalidindi[1]; [1]Drexel University

Recently Kalidindi et al. [Acta Materialia 2005; 53: 3613-3623] explored the novel concept for capturing and simulating crystallographic texture evolution during large plastic strains on metals using an efficient spectral representation of the orientation distribution function. The good prediction and efficiency of this new method was evaluated by using fcc metals in the same publication. In this paper, the similar concept was extended to simulate the crystallographic texture evolution at large deformation on hcp metals where both slip and deformation twinning are plastic deformation modes. Good predictions of the overall texture evolution in a number of different deformation paths were obtained using this newly developed framework.

8:50 AM
Electroless Copper Plating on Graphite Powder Surface: *Liu Wei*[1]; Guangchun Yao[1]; Yihan Liu[1]; [1]Northeastern University

In order to solve the wetting quantity of graphite/copper interface and improve the capability of graphite/copper composite material, we adopted bluestone as main salt, zinc as reductant to plate copper on graphite power surface. The results indicate that there is only copper but zinc in the electroless plating and the electroless plating is hard to be oxidated. This paper emphasize studied the factors such as the pro-treat process, main salt, reductant, temperature, loading capacity and reaction time that effect the electroless plating in order to confirm the optimizing process. Through the optimizing process without additive the copper-coated graphite assumes rose color, and the electroless plating unites tightly with graphite power by analysis of SEM technique.

9:10 AM
Modeling of Deformation and Bonding of Composite Particle during Cold-Spray Deposition: *Ivica Smid*[1]; Gaurav Aggarwal[1]; Albert Segall[1]; [1]Pennsylvania State University

Cold-gas spray is a low temperature coating process by which coatings can be produced without significant heating of substrate or powder, therefore permitting the use of thermally vulnerable lubricants. Nickel, nickel coated boron nitride and molybdenum disulphide, and granulated nickel-solid lube mixes were cold-spray deposited on Ti-6Al-4V substrates. The thermo-mechanical response has been modeled using finite elements. This allowed the estimation of the maximum impact pressures, deformation rates, and deformation kinetics during the impact. The velocity of the impacting particles was taken into account by applying a transient force along the particle equator; the resulting strains were converted to temperature. Local heating and deformation were experimentally validated. The modeling results were assessed with respect to thickness of composite particle encapsulation, critical particle velocity, bonding success and its quality, and deposition efficiency. On the basis of this criterion, optimum particle design and the respective critical velocities can be predicted.

9:30 AM
Non-Isothermal Shearing in Friction Stir Welding: *Patricio F. Mendez*[1]; Thomas J. Lienert[2]; [1]Colorado School of Mines; [2]Los Alamos National Laboratory

A coupled model of the deformation and heat transfer in Friction Stir Welding (FSW) is presented and general expressions are obtained. These expressions are useful to understand FSW in well known alloys such as aluminum-base ones and extrapolate that understanding to higher temperature alloys. The methodology consists of determining asymptotic regimes from which to generate scaling laws. In the analysis of this problem, a threadless pin is considered and the effects of the pin and shoulder are separated. The thickness, temperature, and shear rate of the region surrounding the rotating pin are predicted using an ordering analysis inspired in the bondary layer analysis in fluid mechanics. The model presented helps understand the torque, temperatures, and deformation history of the material near the rotating pin, but it does not address the mechanical mixing ocurring behind the pin. Predictions of torque, temperature, and shear region thickness agree with experiments.

9:50 AM Invited
Friction Stir Processing TiB2 into the Surface of Class 40 Grey Cast Iron to Improve the Wear Resistance: *Uma Ramasubramanian*[1]; William J. Arbegast[1]; Glenn J. Grant[2]; Glen A. Stone[3]; [1]Advanced Material Processing Center; [2]Pacific Northwest National Laboratory; [3]South Dakota School of Mines and Technology

The objective of this work is to use Friction Stir Processing (FSP) to improve the wear resistance of class 40 grey cast iron. FSP is used to process TiB2 particulate into the surface of the cast iron to produce a wear resistant layer. The optimization of the process was achieved by placing TiB2 powders on the surface of a cast iron plate then covering the powder

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with a 0.080 in thick mild steel sheet and subsequently plunging and translating a stir tool across the surface. Microstructural characterization of the optimized welds was carried out using optical and SEM. Comparison before and after the process show significant wear and deformation of the PCBN step spiral pin tool. Data on the wear characteristics and friction coefficient of Friction Stir Processed, TiB2 reinforced cast iron will also be presented.

10:10 AM Break

10:30 AM
Performance Improvement with Advanced Materials at High Temperatures in the Hot Zone Components of Gas Turbines: *Ramarao Adapa*[1]; D. N. Reddy[2]; K. V. Sharma[3]; [1]VNR VJIET; [2]Osmania University; [3]Jawaharlal Nehru Technological University

Gas Turbine (G.T) is basically a prime mover for power generation. To get enhanced performances, this Product Research work with F – Technology is presented which means to increase the Fire Point which in turns to increase the Turbine Inlet Temperature (TIT) there by Heat Energy (HE) is increased in the combustion chambers and converts increased Kinetic Energy (K.E) to Mechanical Energy (M.E). To withstand the very high temperatures in the hot zone compartments in the G.T, the advanced materials like Hastally in combustion chambers, FSX 414 for Nozzles, IN 738 for Turbine buckets, ASTM A336 and AISI 316 with plasma coating for shrouds and Ni Cr Mo V forged steels for Rotor discs and for guide blades in the stator 403-SS with Ni Cd Coating are preferred. The improved performance are recorded and presented with the practical results.

10:50 AM
An Electroplating Technique to Obtain Copper Coating on Carbon Fibers: *Zhuokun Cao*[1]; Yihan Liu[1]; Guangchun Yao[1]; [1]Northeastern University of China

Carbon fibers coated with copper are wildly using in many fields because of its special qualities. In this study, carbon fibers were pretreated in both air at high temperature and HNO3, and the change of the fibers' surface was detected by X-ray photoelectron spectroscopy (XPS). A simple electroplating technique in a sulfate bath was used. Through changing the concentration of H2SO4 and CuSO4 and adding in addition agents, uniform and smooth copper coating were obtained on carbon fibers. And the effects of H2SO4 and addition agents were discussed. Different thickness of copper coating was also obtained by change the compositions of the solution and technological parameters. And the copper coating was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD).

General Abstracts: Structural Materials Division: Advances in Steel

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizers: Rollie E. Dutton, U.S. Air Force; Ellen K. Cerreta, Los Alamos National Laboratory; Dennis M. Dimiduk, U.S. Air Force

Tuesday AM — Room: 218
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Warren M. Garrison, Carnegie Mellon University

8:30 AM
Compact Strip Production - Challenges and Solutions: *Carl-Peter Reip*[1]; Tilmann Böcher[1]; Rolf Hagmann[1]; Wolfgang Hennig[1]; Joachim Ohlert[1]; [1]SMS Demag Aktiengesellschaft

It has been particularly in recent years that owners of CSP plants have made considerable efforts to significantly expand their product range in response to changing market demands. In addition to steel grades, the focus now is also on ensuring the product quality in terms of microstructure, mechanical properties and strip surface, as well as the reproducibility of these parameters. Much attention has been paid to the introduction of the melting and casting technology and the pass-schedule design and cooling strategy for materials such as dual-phase steel, TRIP and API grades up to X80. For the large-scale production of TWIP, the metallurgical basis and process parameters have been worked out for the CSP technology and an initial test production prepared. New findings have been processed to achieve the best surface quality by means of a change in the oscillation strategy, casting powder and cleaning of the thin slabs.

8:55 AM
A Discussion of the Spacing of Inclusions and the Spacing of Inclusion Nucleated Voids on the Fracture Surfaces of Ultra-High Strength Steels: *Warren M. Garrison*[1]; [1]Carnegie Mellon University

For some steels increasing the inclusion spacing at constant inclusion volume fraction is believed to increase the toughness. This work considers a series of low alloy medium carbon martensitic steels in which high toughness has been achieved by increasing the inclusion spacing. The objectives of this work were to determine the spacing of inclusion nucleated voids on the fracture surface and to determine how this spacing related to the spacing of the inclusions in the volume and to assess the degree to which inclusions were actually retained on the fracture surface. While the spacing of the inclusion nucleated voids is comparable to the spacing of the inclusions in the volume the spacing of the inclusions on the fracture surface is much greater than would be observed if half of the inclusion nucleated voids contained inclusions. This suggests that not all inclusions are retained on the fracture surface.

9:20 AM
Dry Sliding Friction and Wear in High Nitrogen Austenitic 18Cr-18Mn-2Mo-0.9N Steel: *Yong-Suk Kim*[1]; Seung Duk Kim[1]; Sung-Joon Kim[2]; [1]Kookmin University; [2]Korea Institute of Machinery and Materials

Dry sliding friction and wear characteristics of a high nitrogen austenitic 18Cr-18Mn-2Mo-0.9N steel has been investigated at room temperature. Wear tests of the steel were carried out using a pin-on-disk wear tester against AISI 52100 bearing steel balls. Two different heat treatments (solution treatment alone and isothermal aging after solution treatment) were performed on the steel and the effect of the heat treatments on the wear were investigated. The wear rate of the solution-treated steel was low initially, but increased abruptly at loads above a critical value. Wear rates of the isothermally aged specimen were low and increased gradually with the applied load. Worn surfaces, their cross sections, and wear debris of the steel specimens were examined with an SEM. Phases of the wear debris were identified using XRD to explore wear mechanism of the steel. Effects of strain-induced phase transformation and Cr_2N precipitates on the wear were discussed.

9:45 AM
Effect of Water Vapor on Metal Dusting Behavior of Ferrous Alloys: *Aditya Putrevu*[1]; Shailendra K. Varma[1]; Zuotao Zeng[2]; K. Natesan[2]; [1]University of Texas; [2]Argonne National Laboratory

The metal dusting behavior of ferrous alloys T22, T91 and 800 in a H_2 $+CO_2+CO+x.H_2O$ atmosphere was investigated. The transition from a combination of accelerated metal dusting and/or extensive pitting to delayed metal dusting attack as vapor content in the atmosphere was increased from 2.3% to 23% was observed. Decreasing H_2O content in the gas increases the carbon activity leading to accelerated carbon deposition. While the increased carbon deposition in the low chromium alloys (T22 and T91) leads to rapid surface and mechanical degradation of the alloys, in high chromium alloys it is more localized in the form of pitting (800). XRD, AES, XPS and EDS have been used to characterize the metal dusting products at micro and nanoscale. Optical microscopy, SEM and TEM were used to study the microstructural issues. The role of alloying elements and mechanisms of growth of carbon nanotubes during metal dusting will also be discussed.

10:10 AM Break

10:25 AM
The Effects of Nickel, Chromium and Carbon on the Strength and Toughness of a Martensitic Precipitation Strengthened Stainless Steel: *Warren M. Garrison*[1]; Aytekin Hitit[2]; Piyamanee Komolwit[1]; [1]Carnegie Mellon University; [2]Afyon Kocatepe University

The effects of nickel, chromium and carbon content on the strength and toughness of a martensitic precipitation strengthened stainless steel having a composition (in wt. %) of 0.005C/12Cr/12Co/5Mo/4.5Ni have been investigated. All alloys were solution treated, oil quenched, refrigerated in liquid nitrogen and then aged. Nickel, chromium and small additions of carbon all increase the strength substantially after aging in the range 500°C to 550°C. Also, these additions all increase the room temperature Charpy impact energy. These additions all result in a marked decrease in the martensite start temperature and an increase in the retained austenite content. Fractography suggests that these additions improve the room temperature toughness because the increase in retained austenite content lowers the ductile-to-brittle transition temperature.

10:50 AM
Influence of Microstructural Anisotropy on Shear Localization in 1080 Eutectoid Steel: *Qing Xue*[1]; George T. Gray[1]; [1]Los Alamos National Laboratory

The influence of microstructural anisotropy on adiabatic shear localization in 1080 rail steel has been studied. A forced shear technique and "hat-shaped" specimens were utilized on a compression split-Hopkinson pressure bar to generate localized deformation. The microstructure was examined using both optical and transmission electron microscopy (TEM). Elongated MnS stringers are observed to be resident within the crystallographically isotropic eutectoid steel. The orientation of these inclusion stringers relative to the shear band direction was found to substantially affect the shear localization behavior. The debonding between the inclusions and the matrix appears to strongly influence the formation of shear bands. The TEM results indicate that the substructure within the shear bands consists of nanoscale subgrains. The fine pearlitic substructure in the steel constrains the dislocation motion and thus increases the propensity of instable deformation.

11:15 AM
On the Correlation of Fracture Toughness and Yield Strength in (15-5PH) Stainless Steel: Mina Samir Abdelshehid[1]; Kosha Mahmodieh[1]; Kyle Mori[1]; Leo Chen[1]; *Omar S. Es-Said*[1]; John Ogren[1]; Richard Clark[2]; [1]Loyola Marymount University; [2]College of the Canyons

Stainless Steel 15-5 PH (UNS number S15500) was thermally exposed and tested for fracture toughness and yield strength. The specimens were received in blank form. They were then austenized to Condition A form (1900°F (+/-25°F) for 1 hour), and then tempered at 925°F (+/-10°F) for 2 hours. 36 samples were machined as round-threaded tensile bars and 18 C(T) (compact tension) specimens for fracture toughness testing. The round tensile bars were tested using a Tinius Olsen frame. The C(T) specimens were analyzed on an MTS hydraulic frame and all had FFC(T) (front-face compact tension) geometry and an L-T (longitudal-transverse) crack plane orientation. It was found that if the tolerances for austenization and tempering temperatures are not strictly controlled, the KIc value decreases significantly, while the yield strength remains virtually unaltered.

11:40 AM
The Effect of Cobalt Additions on the Strength of a Martensitic Precipitation Strengthened Stainless Steel: *Warren M. Garrison*[1]; Piyamanee Komolwit[1]; [1]Carnegie Mellon University

The effect of cobalt additions of 3, 6, 9 and 12 wt. % cobalt on the strength of a martemsitic precipitation strengthened stainless steel having a nominal composition (in wt. %) of 0.005/12Cr/9Co/5Mo/1.5Ni has been investigated. All alloys were solution treated, oil quenched and then refrigerated in liquid nitrogen and then aged. None of the alloys contained retained austenite or delta ferrite. The tensile properties of these alloys have been determined for the as-quenched condition and for aging temperatures from 200°C to 600°C. The aging temperature of peak yield strength was 550°C to 575°C for all of the alloys. Cobalt additions can substantially increase the strength of these materials and the increase in yield strength per wt. % cobalt increases with increasing cobalt content. At the highest cobalt level yield strengths of over 1800 MPa can be achieved.

Granulation of Molten Materials: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Copper and Nickel and Cobalt Committee, TMS: Lead and Zinc Committee, TMS: Pyrometallurgy Committee
Program Organizers: Cameron L. Harris, HG Engineering Ltd; Hani Henein, University of Alberta

Tuesday AM — Room: 7B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Anthony E. M. Warner, HG Engineering Ltd

8:30 AM Introductory Comments by Tony Warner

8:35 AM
Common Granulation System Problems with a Focus on Non-Ferrous Applications: *Art Cooper*[1]; [1]Carlingview Technologies Ltd.

Granulation systems are customarily viewed as problem areas in metallurgical operations. The inherent risk of explosion when combining a high temperature melt with water creates a safety hazard. Separation of the granulated material from the quenching medium generally involves equipment that can have significant maintenance and operating costs. Granulation systems' reliability and availability can negatively affect the plant production. Against this background, granulation systems have been treated by design firms and producers as an add-on and a necessary evil. The technology is becoming better understood and solutions to common problems are being found. This paper will discuss typical problems in common systems, and some of the improvements that have been made, with particular reference to non-ferrous applications.

9:00 AM
Atomisation as Solidification Process Alternative from Pyrometallurgy to Hydrometallurgy and as a One Step Process for Flash Furnace Feed: *David Norval*[1]; [1]Bateman Engineering

In the Smelting of Ni, Co, Cu and Pt, mattes are a common material that requires granulation of the molten matte and milling of resultant granules for further processing. This route is fine if the material is brittle (friable) which is a property associated with the higher sulphur mattes. For lower sulphur levels, where the matte is less amenable to milling, the costs of the milling increases. This paper will compare water granulation with certain dry granulation processes and the downstream processes required for Hydrometallurgy. It will further introduce and expand on atomisation which offers a further safety benefit in the control of the metal flow into the 'wet zone' with the use of a tundish flow control also reducing the conversion from the matte to powder in one fairly straightforward operation.

9:25 AM
The Generation of Mono-Dispersed Granules from Melts: *Hani Henein*[1]; [1]University of Alberta

Recent innovations in atomization of melts have occurred in the area of single fluid atomization. Impulse Atomization (IA), one of the single fluid techniques, provides unique capabilities to study different regimes of atomization in a controlled environment. It has been observed that by changing processing variables, a mono-dispersed distribution of droplets above one millimeter in diameter can be produced. This is accomplished through proper characterization of the forces influencing the process. This paper will discuss the approach and illustrate results for various metallic melts.

9:50 AM Concluding Comments by Hani Henein

Hume Rothery Symposium: Multi-Component Alloy Thermodynamics: Alloy Thermodynamics I: Experiment and Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Y. Austin Chang, University of Wisconsin; Rainer Schmid-Fetzer, Clausthal University of Technology; Patrice E. A. Turchi, Lawrence Livermore National Laboratory

Tuesday AM — Room: 202A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Rainer Schmid-Fetzer, Clausthal University of Technology

8:30 AM Invited
Phase Modeling and Thermodynamic Simulations in the Y-Si-C-O System: *Hans J. Seifert*[1]; [1]University of Florida

Recent experimental information was used for the thermodynamic phase modeling in the quaternary Y-Si-C-O system and a new dataset with Gibbs free energy descriptions was developed. The dataset can be used for the simulation of complex multi-component, multiphase reactions in the system. The development of yttrium silicate coatings for the oxidation protection of C/SiC-based composites and the liquid phase sintering of SiC with yttria as an additive will be discussed. Besides the liquid and the gas phases, the following binary and ternary solid phases were taken into account: Y_5Si_3, Y_5Si_4, YSi, Y_3Si_5 (α, ß), YSi_2 (α, ß), γ yttrium carbide, YC_2, Y_2C_3 (α, ß), $Y_{15}C_{19}$ (α, ß), Y_2O_3 (α, ß), SiC (α, ß), SiO_2 (cristobalite, tridymite, quartz), $Y_5Si_3C_2$, $Y_2Si_2O_7$ (α, ß, γ, δ), Y_2SiO_5 (A, B) and γ-Y_2CO.

9:00 AM Invited
Thermodynamic Modeling of Ni-Based Alloys for Superconducting Coated Conductor Tapes: *Alexander Pisch*[1]; Jiasong Wang[2]; Jean-Louis Jorda[2]; [1]Institut National Polytechnique de Grenoble/Centre National de la Recherche Scientifique; [2]Universite de Savoie

Superconducting coated conductor tapes have a layered structure including a metallic substrate, an oxide buffer layer, the superconducting layer and a cap layer. To get good superconducting properties, the corresponding layer has to be highly textured. This can be achieved by texturing the substrate. A buffer layer is additionally introduced to prevent any reaction between the superconducting material and the metallic substrate. The substrate is a Ni-based alloy and the texture is achieved by rolling. The buffer layer is deposited by classical thin film deposition techniques. Another possibility is to obtain the buffer layer directly by oxidizing a substrate which contains the oxide forming element. In order to control the process, the relevant multi-component phase diagrams and the underlying equilibria have to be known. A thermodynamic modeling of the Ni-Cu-Ce and the Ni-Cu-Y system has been performed based on literature information to evaluate the feasibility of this in-situ oxidation process.

9:30 AM Invited
Thermodynamics of the Pt-Modified γ-Ni+γ'-Ni_3Al System and Resulting Implications on the Development of Novel High-Temperature Alloys and Coatings: *Brian M. Gleeson*[1]; Takeshi Izumi[1]; Chao Jiang[1]; Daniel Sordelet[1]; Shigenari Hayashi[2]; [1]Iowa State University; [2]Hokkaido University

This presentation will discuss the role of platinum addition in promoting the formation a highly adherent, slow-growing Al_2O_3 scale on γ'-Ni_3Al+γ-Ni alloys during high-temperature oxidation. It will be shown that this beneficial effect can be primarily ascribed to the fact that platinum is non-reactive and its addition decreases the chemical activity of aluminum in γ+γ' alloys. Related to the latter, Pt partitions almost solely to the Ni sites in the $L1_2$ crystal structure of γ', which has the effect of increasing the Al:Ni atom fraction on a given crystallographic plane containing both Al and Ni. Moreover, intensive first principles calculations showed that Pt segregation on (100) and (111) surfaces is energetically favorable. The tendency of Pt to surface segregate and to have a Ni-site preference has the contributing effect of kinetically favoring the formation of Al_2O_3 relative to NiO on the γ' surface.

10:00 AM Break

10:20 AM Invited
Contribution to Phase Relationship Study in the Si-Tb-Ti System: *Jean-Claude Tedenac*[1]; Marina Bulanova[2]; Henri Noel[3]; Anton Samelyuk[2]; A. Pudovkina[4]; [1]Laboratoire de Physique de la Matière Condensée; [2]I.N. Frantsevich Institute for Problems of Materials Science; [3]Laboratoire de Chimie du Solide et Inorganique Moléculaire- UMR-CNRS6511; [4]National Technical University of Ukraine

The silicon-terbium-titanium phase diagram system is still unknown. It present a double interest: on the titanium rich side possible cold and hot structural materials and on the silicon rich side possible materials of electronics. The problem which limits all these applications is the brittleness of these silicides which limits the structural properties but also the other applications. Constitution of materials in term of microstructure (study of phase diagrams, crystal structure determination of phases), properties of alloys obtained by solidification, heat treatment and powder techniques need to be determined. Concerning the phase stabilities, the only information available in this system is the existence of two ternary compounds – TiTbSi and Tb2Ti3Si4. By using differential thermal analysis, metallography, microprobe and X-ray analysis of some sections of the Ti-Tb-Si system, the phase transformations are determined.

10:50 AM
Lattice Monte Carlo Simulations of Y-Ti-O Nanoclusters in Ferritic Alloys: Matthew J. Alinger[1]; *Brian D. Wirth*[1]; G. Robert Odette[1]; [1]University of California

Lattice-based Monte Carlo (LMC) simulations of the formation and stability of nanometer-scale Y-Ti-O clusters (NC) in ferritic alloys, with a nominal composition in the range of Fe-(14)Cr-(0-1)W-(0.25-1)Ti-(0.1-0.25)Y-(0.15-0.40)O are presented. The LMC simulations use pair bond energies calculated from the mixing enthalpies in a regular-solution thermodynamics model and validated by ab-initio calculations and are performed on a bcc lattice, with oxygen on an octahedral interstitial sublattice. The results show NC structure and composition evolution in terms of temperature and alloy composition, starting from random super-saturated solutions. The resulting precipitate compositions are compared to an extensive experimental database of transmission electron microscopy, small angle neutron scattering, atom probe tomography and positron annihilation spectroscopy. Combined, these results provide atomic-level insight into the NC structure and chemistry and provide a basis for optimizing alloy development as well as understanding the thermal and radiation stability of the NCs.

11:10 AM
Thermodynamic Assessment of the Nb-Mo-Si-B System: *Yali Li*[1]; Zhihong Tang[1]; Mufit Akinc[1]; Matthew J. Kramer[1]; [1]Iowa State University

Mo-Si-B system intermetallics have a great potential for high temperature application due to their good oxidation resistance. In order to improve mechanical properties, Nb is considered as a favorable addition because of excellent strength of Niobium silicides based alloys and the complete solid solution between Mo and Nb. A thermodynamic description of Mo-Nb-Si-B quartnery system is assessed to help design alloy composition with CALPHAD method using Thermo-Calc. First, a thermodynamic description for Nb-Si-B ternary system is assessed using all the existing phase diagram and experimental data. Then, the Mo-Nb-B and Mo-Nb-Si systems are developed with the similar method. Finally, the Nb-Mo-Si-B quaternary system is critically optimized by extrapolating thermodynamic data of four constituent ternary systems. Comparison between experimental phase equilibria data and thepredictions based on the assessed data is discussed.

11:30 AM
Solute-Solvent Interactions in fcc-Ni: *Tao Wang*[1]; Long-Qing Chen[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University

The effect of solutes on the structural and thermodynamic properties of an alloy is determined by the solute-solvent interactions. In this work, we used first-principles approach to study the effects of various solute atoms (Al, Co, Cr, Hf, Mo, Nb, Re, Ru, Ta, Ti and W) on fcc-Ni. The partial formation energy as well as the lattice distortions (macroscopic and local) of different solutes were calculated and compared with experimental data in literature. By analyzing the charge density distributions around the solute atoms, the effects of atomic size, electronic interaction, and magnetic spin direction on lattice distortion were discussed. By neglecting the interactions between different solute atoms, the formation energy and the lattice parameters in multi-component Ni-base superalloys were predicted using a phenomenological model. A comparison of predicted results with available experimental measurements shows and good agreements at relatively low solute concentrations.

Lead Free Solder Implementation: Reliability, Alloy Development, and New Technology: Interfacial Reactions and Role of Intermetallics

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Nikhilesh Chawla, Arizona State University; Srinivas Chada, Medtronic; Sung K. Kang, IBM Corporation; Kwang-Lung Lin, National Cheng Kung University; James Lucas, Michigan State University; Laura J. Turbini, University of Toronto

Tuesday AM — Room: 214A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: C. R. Kao, National Central University; Andre Lee, Michigan State University

8:30 AM
Whisker and Hillock Formation on Sn, Sn-Cu and Sn-Pb Electrodeposits: *William J. Boettinger*[1]; Gery R. Stafford[1]; Christian E. Johnson[1]; Maureen E. Wiliams[1]; Kil-Won Moon[1]; Leonid A. Bendersky[1]; [1]National Institute of Standards and Technology

Bright Sn, Sn-Cu and Sn-Pb layers, 3, 7 and 16 µm thick were electrodeposited on phosphor bronze cantilever beams in a rotating disk apparatus. In several days, the surfaces of the Sn-Cu deposits, which have the highest compressive stress, develop contorted hillocks and whiskers, pure Sn deposits develop compact conical hillocks, and Sn-Pb deposits, which have the lowest compressive stress, remain unchanged. The differences between the initial compressive stresses is due to the rapid precipitation of Cu_6Sn_5 or Pb particles, respectively, within supersaturated Sn grains produced by electrodeposition. Over longer time, analysis of beam deflection measurements indicates that the compressive stress is augmented by the formation of Cu_6Sn_5 on the bronze/Sn interface. Uniform creep occurs for Sn-Pb because it has an equiaxed as-plated grain structure. Localized creep in the form of hillocks and whiskers occurs for Sn and Sn-Cu because both have columnar as-plated structures.

8:50 AM
Effect of Cu_6Sn_5 Doping on Microstructure and Strength for the Sn-Ag-Cu Solder Joint with Al/Ni UBM: *Guo-Jyun Chiou*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University

Intermetallic compound of Cu_6Sn_5 plays an important role in the interfacial reaction between SnAgCu solder and Al/Ni UBM during reflowing in electronics packaging. In this study, Cu_6Sn_5-contained SnAgCu solder paste was produced by mixing Cu_6Sn_5 nano powder into commercial SnAg solder paste. The Al/Ni UBM was first deposited onto the silicon wafer, and the Cu_6Sn_5-contained solder paste was then stencil printed on the UBM and reflowed at 240°. The interfacial microstructure and elemental distribution were evaluated was studied with FE-SEM and FE-EPMA. During different reflow times, the thickness of IMCs in Cu_6Sn_5-contained solder joints was thinner than that of commercial SnAgCu solder. To realize the effect of Cu_6Sn_5 nano powder doping, the shear strength of the joint was further investigated. In addition, the fracture mode of SnAgCu solder joints was probed with the aid of the fracture surface, interfacial morphology, and shear strength.

9:10 AM
Effect of Phosphorus Content on Cu/Ni-P/Sn-3.5Ag Solder Joint Strength under Multiple Reflow: *Zhong Chen*[1]; Aditya Kumar[1]; M. Mona[1]; [1]Nanyang Technological University

Electroless Ni-P with three different P contents (6.1, 8.8, and 12.3 wt%) was deposited on copper substrates. Sandwiched specimens of Cu/EN/Au/Sn-3.5Ag/Au/EN/Cu were prepared and were subjected to multiple reflows at 250°C. Tensile test was performed to investigate the effect of P content on the joint strength. It was found that the low-P samples exhibited the highest joint strength after multiple reflows, while the strengths of the medium and high-P samples decreased more rapidly. From cross sectioned views, Ni-Sn IMC formed at the interface of the low-P sample was found to be more stable, while the one of the medium and high-P samples spalled into the molten solder. The IMC spallation speeded up Ni-P consumption, leading to the formation of Cu-Sn IMCs. Fractographic and microstructural analyses showed that the degradation was due to full consumption of EN and the formation of Cu-Sn IMCs at the interface.

9:30 AM
Effects of the Interfacial Reactions of Sn-8Zn-3Bi Solder Joints on Their Electrical and Mechanical Properties: *Won Kyoung Choi*[1]; Changyoul Moon[1]; Sung K. Kang[2]; Da-Yuan Shih[2]; [1]Samsung Advanced Institute of Technology; [2]IBM Corporation

Sn-8Zn-8Bi solder has been reported to show improved their joint properties in terms of interfacial reactions, wetting and mechanical properties and long-term reliability comparing to the Sn-Zn binary system. In this study, the effects of the interfacial reactions on the joint properties of Sn-8Zn-3Bi were investigated as a function of reflow time, aging time, and surface finish (Cu vs Ni). The interfacial microstructure was investigated using EPMA and XRD to identify the phases of the interfacial intermetallic compounds formed. The electrical and mechanical properties were measured using a model joint made of two L-coupons. These properties revealed a unique behavior comparing to other Pb-free solder joints. The changes in the electrical and mechanical properties of Sn-8Zn-3Bi joints were also explained based on their interfacial reactions with a corresponding surface finish.

9:50 AM
Elemental Diffusion Behavior for the Sn-3.0Ag-(0.5 Or 1.5) Cu Solder Bump with Cu/Electroless Ni-P/Immersion Au Bonding Pad during Aging: Guh-Yaw Jang[1]; *Yung-Chi Lin*[1]; Jenq-Gong Duh[1]; Mysore A. Dayananda[2]; [1]National Tsing Hua University; [2]Purdue University

Isothermal interdiffusion in Sn-3.0Ag-(0.5 or 1.5)Cu solder joints with electroless Ni/immersion Au surface finish after aging at 150°C was investigated. The diffusion structures and concentration profiles were examined with a newly developed Field-Emission Electron Probe Microanalyzer. Intermetallic compounds of $(Cu_{1-y},Ni_y)_6Sn_5$, $(Ni_{1-x},Cu_x)_3Sn_4$ and P-rich layer formed between the solder and the EN layer in both Sn-Ag-Cu joints during aging. For the Sn-3.0Ag-0.5Cu joints after more than 2000 h aging, $(Ni_{1-x},Cu_x)_3Sn_4$ IMC gradually grew. The interdiffusion fluxes and effective interdiffusion coefficient of Cu, Ni, and P were calculated from the concentration profile with the aid of Matano plane evaluation. Values of J_{Cu}, J_{Ni} and J_P decreased with increasing aging time. The average effective interdiffusion coefficients in the order of 10^{-14} cm^2/s were also evaluated within the diffusion zone. In addition, the effect of Cu content in solders on diffusion behaviors of Cu in Sn-3.0Ag-(0.5 or 1.5)Cu joints were probed and discussed.

10:10 AM Break

10:25 AM
Fracture Mechanics Approaches to the Interfacial Fracture of a Solder Joint and a Solder Bump: *Woong Ho Bang*[1]; Choong-Un Kim[1]; Myung-Woon Moon[2]; Kyu Hwan Oh[2]; [1]University of Texas at Arlington; [2]Seoul National University

Using finite element methods, we have analyzed the interfacial fracture mode of a solder joint in a tensile test and that of a solder bump in a ball-shear test. The cracking site has been defined at interfacial IMC layer (Cu6Sn5) between solder and Cu metallization, where we have estimated stress intensity factors 'K' under those specific testing methodologies. Analysis of the tensile test has shown that tensile load applied to a solder joint develops both crack-opening KI mode and crack-shearing KII mode at crack tip in IMC layer. Through the FEM simulation on a bump shear test, a crack-opening KI has been observed as a dominant mode of the bump fracture. The present talk will discuss in detail current results.

10:45 AM
Revealing the Interfacial Reaction of Novel Solders Developed from Sn-3.5Ag-0.5Cu Nanoparticles on Electroless Ni-P/Al UBM: *Li-Yin Hsiao*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University

To meet the requirements for future electronic packaging technology, an ultrasmall solder bump is a tendency. With the reduced size of solder bumps, finer grain in solder paste is crucial for fine pitch bumping. Currently, near-eutectic SnAgCu alloys are developed as the lead-free solder. In this study, the Sn-3.5Ag-0.5Cu nanoparticles synthesized by chemical reduction method were introduced to prepare the lead-free solder. The secondary particle size of Sn-3.5Ag-0.5Cu nanoparticles was in the range of 40 nm. The detailed interfacial reaction of lead-free solders derived from the Sn-3.5Ag-0.5Cu nanoparticles on EN(P)/Al under bump metallization (UBM) was investigated after reflows. After morphological observation and quantitative analysis with a field-emission electron probe microanalyzer, the influence of the particle size for solders on the formation of IMCs at SnAgCu solder/electroless Ni-P interface was probed and discussed.

11:05 AM
Study of Coupling Effect by Using Sandwich Structures of Ni/Sn/Cu and Au/Sn/Cu: *Snen-Jie Wang*[1]; Cheng-Yi Liu[1]; [1]National Central University

The coupling effects between Cu-Sn and Ni-Sn soldering reactions have been investigated in this study. The interfacial Ni-Sn reaction would strongly be affected by the interfacial Cu-Sn reaction in the opposite side. The preliminary results did show that an unusual growth of ternary Cu-Ni-Sn compound at the Sn/Ni interface and a huge consumption of Cu in the opposite side. FE-EMPA results showed that the content of Ni in the Cu-Ni-Sn ternary phase varied along its thickness. The detail kinetics model for the ternary Cu-Ni-Sn phase formation will be presented in this talk. Beside, we will also report the coupling effect between Cu-Sn and Au-Sn interfacial reactions by using the Au/Sn/Cu sandwich structures. Again, we observed that the interfacial Cu-Sn reaction would be significantly influenced by the opposite Au-Sn interfacial reaction. The detail experimental observation on the microstructure and phase formation at both interfaces will be reported.

11:25 AM
Study of the Reactions between Cuxniy Alloy UBM and Sn-Ag (-Cu) Solder: *Hun Han*[1]; Y. C. Sohn[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology

A reaction study of CuxNiy alloy (x=0.2~0.95) under bump metallization (UBM) with Sn-Ag-zCu solder (z=0~0.7) was conducted. Formation and separation of intermetallic compounds (IMCs), effect of Cu addition to the CuxNiy alloy and the solders, and compatibility of reaction products with currently available phase diagrams are intensively investigated. Optimum range of Cu concentration, over which thick IMCs formed at interface without separation, was found for IMC separation behavior. Addition of Cu to the solder fasten IMC growth and CuxNiy (x=0.2~0.4) consumption though the reverse trend was true of most solder reactions in literature. The amounts of Ni and Cu dissolved to the solders were calculated, and the reaction products were compared with Cu-Ni-Sn ternary phase diagrams available. Experimental results in the present study suggest a need for modification of the Cu-Ni-Sn diagrams.

11:45 AM
Cu Content and Size Effects upon the Reaction Layer Detachment in the Sn-Cu/Ni Couples: *Chao Hong Wang*[1]; Sinn-Wen Chen[1]; [1]National Tsing Hua University

The effects of Cu contents and the size of solder volumes upon the reaction layer detachment are investigated. Sn-Cu/Ni couples are reacted at 250oC and the alloys are with 0.4, 0.7, 1.0 and 2.0wt% Cu. The amounts of Sn-Cu alloys are 2g and 10g. Except for the 0.4wt%Cu couples, the product in the beginning is the Cu6Sn5. The Cu6Sn5 reaction layer detaches in the 2g Sn-Cu couples after 2-hour reaction. The detached layer decomposes in the molten solder, and Ni3Sn4 forms after a prolonged reaction time. Higher amount of Cu and larger amount of solders delay the layer detachment. Microstructural analysis reveals the detached and the attached layers have different morphologies. The Cu contents in the solders also influence the morphologies of the Cu6Sn5 phase. The detachment is related with the formation of the Ni3Sn4 phase, and is influenced by both the Cu contents and the amounts of solders.

12:05 PM
Morphology of Intermetallic Compounds Formed between Lead-Free Sn-Zn Based Solders and Cu Substrate: Chia-Wei Huang[1]; *Kwang-Lung Lin*[1]; [1]National Cheng Kung University

The morphologies of intermetallic compound formed between Sn-Zn based solders and Cu substrate were investigated in this study. The investigated solders were Sn-9Zn, Sn-8.55Zn-0.45Al, Sn-8.55Zn-0.45Al-0.5Ag solders in the weight percent. The experimental results indicated that the Sn-9Zn solder formed the Cu5Zn8 and CuZn5 compounds on Cu substrate, while the Al-containing solders formed the Al4.2Cu3.2Zn0.7 compound. The addition of Ag to the Sn-8.55Zn-0.45Al solder resulted in the AgZn3 compound formed at the interface between Al4.2Cu3.2Zn0.7 compound and solder. Furthermore, it was found that the cooling rate of the specimen after soldering had an effect on the quantity of AgZn3 compound formed at the interface. The AgZn3 compound with air-cooling condition exhibited rougher surface and larger size than with water-quenched condition. It was believed that the formation of AgZn3 compound at the interface was enhanced by heterogeneous nucleation during solidification.

Magnesium Technology 2006: Casting and Solidification II

Sponsored by: International Magnesium Association, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Alan A. Luo, General Motors Corporation; Neale R. Neelameggham, US Magnesium LLC; Randy S. Beals, DaimlerChrysler Corporation

Tuesday AM — Room: 6A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: David H. St. John, University of Queensland; John E. Allison, Ford Motor Company

8:30 AM
Characterization of Cavity Pressure in Squeeze Casting of Magnesium Alloys: *Fang Alfred Yu*[1]; Naiyi Li[2]; Henry Hu[1]; [1]University of Windsor; [2]Ford Motor Company

In squeeze casting processes, cavity pressures directly affects the heat transfer coefficients between casting and mold, and subsequently the solidification behavior of castings. For process optimization, it is very important to understand the distribution of applied pressure in a die cavity. Very often in the past, the distribution of the cavity pressure in either process design or numerical simulation of squeeze casting was considered uniform in the die cavity. Rare experimental work on measuring the cavity pressure has been conducted. In this study, an attempt has been made in characterizing the pressure distribution in the die cavity. A piezoelectric quartz pressure transducer was integrated into a die cavity with different geometrical shapes. The experiments have been carried out to reveal that the pressure distribution changes with the cavity geometry. Also, the change of the local cavity pressure at various locations in the duration of casting solidification was observed.

8:50 AM
Effect of Power Ultrasound on Grain Refinement of Magnesium AM60B Alloy: *Xiaogang Jian*[1]; Thomas Tom Meek[1]; Qingyou Han[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

In the past decades, several grain-refining techniques have been reported for magnesium – aluminum based alloys, namely superheating, carbon inoculation, and Elfinal process. This article describes an experimental attempt to refine the solidification structure of magnesium AM60B alloy using high intensity ultrasonic vibrations. Ultrasonic energy up to 1500 W was injected into the alloy during its solidification. Casting temperature was varied in order to obtain the optimal effect on grain refinement. The experimental results indicated that grain refinement was readily achievable for this alloy with ultrasonic vibrations. When the casting temperature was 675°C, globular grains were obtained. The grain size can be as small as 30 to 40 µm, which was over an order of magnitude smaller than the dendritic grains obtained without using ultrasonic vibrations.

9:10 AM
Heat Treatments and Mechanical Properties of a Rheocast AM60 Magnesium Alloy: *Emanuela Cerri*[1]; Pasquale Cavaliere[1]; Pier Paolo De Marco[1]; Paola Leo[1]; [1]University of Lecce

A study on the heat treatment response and the mechanical properties of a rheocast AM60 magnesium alloy was performed. The alloy was aged in the as-rheo condition and after high temperature exposure (395 and 415°C). The aging temperatures were in the range of 170-220°C. The hardness values and electrical conductivity measurements of aged samples show no substantial difference between solution treated and as-rheo specimens. Tensile tests on samples heat treated in different conditions according to solution and aging temperatures presented above were also investigated. The mechanical properties like yield strength, ultimate tensile strength and strain to failure are discussed as a function of solution and aging temperature and time. The grain size remains stable when the material is exposed at high temperature (395 and 415°C).

9:30 AM
Microstructural Refinement of Magnesium Alloy by Electromagnetic Vibrations: *Kenji Miwa*[1]; Yoshiki Mizutani[1]; Takuya Tamura[1]; [1]National Institute of Advanced Industrial Science and Technology

We have developed the new process for refinement of metallic materials during solidification without addition of refiners or without rapid cooling. This process uses electromagnetic body force based on the vibrations caused by simultaneous imposition of direct magnetic field and alternative electric current on the alloy melt during solidification. The vibrations create cavitation in the melt and it breaks out during growth of it. Then explosive force is released toward the surroundings such as the primary solid particles and they are fractured finely. Finally fractured solid particles solidified as very fine grains. This process was applied to AZ91D alloy. Primary magnesium dendrite particles decreased from about 1800 micron to about 100 micron. This process was also useful to refinement of primary particle in pure magnesium.

9:50 AM
Microstructure and Mechanical Properties of Die Cast Magnesium Alloy AM50 with Varying Section Thicknesses: Ming Zhou[1]; Henry Hu[1]; *Naiyi Li*[2]; Alfred Yu[1]; [1]University of Windsor; [2]Ford Motor Company

Understanding of the effect of section thicknesses on mechanical behavior of AM50 is critical for proper design of different applications with varying cross-section thicknesses due to its extensive use in automobiles. In the present study, magnesium alloy AM50 was high pressure die cast into rectangular coupons with section thicknesses of 2, 6 and 10 mm. The prepared coupons were tensile tested at room temperature. Microstructure analysis and porosity measurement were performed on the representative specimens. The results show that their tensile properties decreases with an increase in section thicknesses of die cast AM50. Microstructure and porosity analyses indicate that the tensile behavior of die cast AM50 is primarily attributed to the level of porosity which resulted from entrapped gases during die casting process. The preliminary results of numerical simulation on mold filling imply that the porosity content of casting coupons is significantly influenced by the design of gating systems.

10:10 AM Break

10:30 AM
On Liquidus and Solidus Temperatures in AZ and AM Alloys: *Munekazu Ohno*[1]; Djordje Mirkovic[1]; Rainer Schmid-Fetzer[1]; [1]Clausthal University of Technology

The liquidus and solidus data reported for commercial AZ and AM alloys are generally based on thermal analysis. In the present study, the following two points have been clarified by performing own experiments and Calphad-type thermodynamic calculations: (i) The measured 'liquidus" temperature generally does not represent the actual equilibrium one, in other words, the primary precipitate for these Mg-alloys cannot be detected in the thermal analysis. (ii) The measured "solidus" does not correspond to the equilibrium solidus and not even to the end of non-equilibrium solidification process. The measured "solidus" is often associated with the precipitation of Mg17Al12 phase and, importantly, the solidification process of these Mg-alloys ends at much lower temperature. This work is supported by the German Research Foundation (DFG) in the Priority Programme "DFG-SPP 1168: InnoMagTec."

10:50 AM
Vertical Direct Chill (VDC) Casting of a Novel Magnesium Wrought Alloy with Zr and RE Additions (ZK10): Alloying Issues: *Michael Kettner*[1]; Franka Pravdic[1]; Werner Fragner[1]; Karl Ulrich Kainer[2]; [1]ARC Leichtmetallkompetenzzentrum Ranshofen GmbH; [2]GKSS Research Center

The present paper describes the microstructure of a new magnesium wrought alloy (ZK10) in as cast condition. Furthermore the experimental procedures and the evaluation of the results are presented. Zirconium master alloys of two different suppliers were analyzed in alloying experiments in laboratory scale. The master alloy showing better qualities was subjected to a VDC casting trial in industrial scale at LKR. Billets of the alloy containing zirconium and rare earth metals were cast in a diameter of 185 mm and a length of 2000 mm for further extrusion trials. The master alloys, the samples from the alloying experiments as well as the cast billets were investigated using metallography, scanning electron microscopy and segregation analysis.

11:10 AM
Warm Water Scale Model Experiments for Magnesium Die Casting: Mohamed Hassan[1]; Kazunori Kuwana[1]; Valerio Viti[1]; *Adrian S. Sabau*[1]; Kozo Saito[1]; [1]University of Kentucky

High Pressure Die Casting (HDCP) involves the filling of a cavity with the molten metal through a thin gate. High gate velocities yields jet break-up and atomization phenomena. In order to improve the quality of Mg parts, the mold filling pattern, including atomization phenomena need to be understood. The goal of this study was to obtain experimental data on jet break-up characteristics for conditions similar to that of a Mg HDCP, and measure droplet velocity, size and size distribution. A scale analysis is first presented in order to identify appropriate analogue Mg materials. Based on the scale analysis, warm water was chosen as a suitable analogue and different nozzles ere manufactured. A 2-D component Phase Doppler Particle Analyzer (PDPA) and 2-D component Particle Image Velocimetry (PIV) were then used to obtain fine particle diameter and velocity distributions in 2-D plane.

11:30 AM
Mechanical Properties of Lost Foam Cast Magnesium Alloys AZ91E, AM50 and ZE41: *Festus A. Fasoyinu*[1]; Peter Newcombe[1]; Mahi Sahoo[1]; [1]Materials Technology Laboratory

As part of an ongoing research program at CANMET (Canada Centre for Minerals and Energy Technology) Materials Technology Laboratory (MTL) to develop a viable lost foam casting technology for magnesium-base alloys, the mechanical properties of test bars machined from foam pattern castings were evaluated. This paper provides tensile properties data in the as-cast condition and after heat treatment, and the issues encountered during the casting trials are also described.

Magnesium Technology 2006: Microstructure and Properties I

Sponsored by: International Magnesium Association, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Alan A. Luo, General Motors Corporation; Neale R. Neelameggham, US Magnesium LLC; Randy S. Beals, DaimlerChrysler Corporation

Tuesday AM — March 14, 2006
Room: 6B — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jian-Feng Nie, Monash University; Menachem S. Bamberger, Technion

8:30 AM
Effect of Hot Torsion Deformation on Microstructure in AZ31 Magnesium Alloy: *Faramarz M. Zarandi*[1]; Stephen Yue[1]; Ravi Verma[2]; [1]McGill University; [2]General Motors

Strength and ductility are desired characteristics in sheet materials for applications in the auto industry. Such characteristics are developed during hot and cold rolling of thick as cast materials. Therefore, it is important to understand the evolution of microstructure during rolling. In terms of large strains and high strain rates applied during rolling, torsion could be an adequate laboratory deformation process to simulate the industrial rolling practice. In this work, an as cast AZ31 magnesium alloy was deformed in torsion under different thermomechanical conditions and the resultant microstructures were examined. At high temperatures, discontinuous recrystallization is the principal mechanism for microstructure evolution with nucleation of new grains at as cast grain boundaries. On the contrary, continuous recrystallization contributes to the evolution of microstructure at low temperatures. It is observed that the effect of strain rate on the microstructural development is not as important as those of temperature and strain.

8:55 AM Cancelled
Microstructure and Mechanical Property of Extruded Mg-Zn-Cu-Gd Based Alloy Reinforced by Quasicrystals and Laves Phases

9:20 AM
Microstructure Study of Magnesium Alloys at Various Stages of Creep Using EBSD: *Takanori Sato*[1]; Barry L. Mordike[2]; Jian-Feng Nie[3]; Milo V. Kral[1]; [1]University of Canterbury; [2]TU Clausthal; [3]Monash University

Magnesium and common Mg-Al based alloys exhibit constant creep rates during secondary creep stage, typically described as power law creep. Diffusion-induced dislocation motion and grain boundary sliding are believed to be the key mechanisms of creep in magnesium. Furthermore, with addition of aluminum, the development of coarse intergranular and fine coherent intragranular β ($Mg_{17}Al_{12}$) precipitates is known to affect the creep rate at temperatures over 150°C. In this research, wrought pure magnesium and binary Mg-Al alloys were creep tested at various conditions of temperature and stress. The tests were interrupted periodically and sample surface microstructure was characterized using orientation microscopy via SEM/EBSD to provide a sequential microstructural analysis. This enabled an historical observation of deformation accumulation, precipitation, and grain boundary interactions as creep progressed.

9:45 AM
Design Perspectives for Creep Resistant Die-Cast Magnesium Alloys: *Bimal Kad*[1]; Qingyou Han[2]; Srinath Viswanathan[3]; [1]University of California-San Diego; [2]Oak Ridge National Laboratory; [3]Sandia National Laboratories

Die cast magnesium alloy design for elevated temperature creep is evaluated from the perspective of the particular contributions of alloying, the nonuniform microstructure and eutectic constituents. Thermodynamic simulations provide the necesssary insight into solute segregation, solidus temperature, and the corresponding homologous temperature distribution in the a(Mg) phase for Mg-Al alloys with ternary additions. Experimetnal and computational results suggest that significant creep deformation occurs in the a(Mg) phase in and adjacent to the eutectic regions with the low solidus constituent while deformation in the primary a(Mg) dendrites is less pronounced. Microstructural design efforts that increase the homologous temperature, reduce eutectic volume or reinforce the eutectic a(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 development. REF: Han, Kad and Viswanathan (2004) Philos Mag, Vol.84, p.3843-60.

10:10 AM Break

10:30 AM
Creep Transition Behavior of Pure Magnesium Poly and Single Crystals: *Jaesin Park*[1]; Youngwon Chang[1]; [1]Pohang University of Science and Technology

The purpose of this study is to investigate the effect of barrier structure on the dislocation motion in hcp crystal, which is in turn believed to determine the permeability parameter(p) of plastic equation of state. It has also been intended to verify whether there exists a mechanical equation of state for Mg single and poly crystals. Mg single crystals with random orientations were grown form a commercial purity Mg poly-crystal using modified Bridgman furnace. A series of load relaxation tests was then conducted on these single and poly-crystal Mg. In addition, tests at various temperatures were performed to investigate the temperature effect on the flow behavior. The test results are then analyzed using the recently proposed internal variable theory, based on dislocation kinematics, which has been successfully applied to many other materials.

10:55 AM
Precipitations Hardening and Phase Formation in Mg-Sn-Zn-Al Alloys: Shlomo Haroosh[1]; Ginat Goren-Muginstein[1]; George Levi[1]; *Menachem S. Bamberger*[1]; [1]Technion

Based on a previous research, Mg-Zn-Sn alloys do not show promising behavior at higher temperatures. In order to increase the structural stability, 1%wt MM (50%Ce-25%La-20%Nd-5%Pr) was added. At the as-cast condition, SEM micrographs indicate a very fine micro-structure (DAS below 12 µm). The study focuses on the precipitation hardening, phase formation and structural stability during aging at elevated temperatures, of solution treated samples. After solution treatment (450 C) and aging at 225 C, Vickers hardness measurements show that this alloy maintains an increase of 30% in hardness for periods up to 32 days. Very fine homogenously distributed precipitations (less than 500nm) were found. EDS, XRD and Auger characterization methods were employed in order to identify the phase structure of the alloy and the origin of precipitates. There is no evidence for the presence of the deleterious γ-Al12Mg17 phase. EDS and Auger spectroscopy study showed that the RE precipitates are MgREAlZn.

11:20 AM
The Influence of Y Addition on Precipitation Sequence in Mg-Zn-Sn Based Alloy: Anton Gorny[1]; Ginat Goren-Muginstein[1]; Gerhard Dehm[2]; Boriana Rashkova[2]; *Menachem S. Bamberger*[1]; [1]Technion; [2]Max Plank Institute fuer Metallforschung

The main goals of this research are to investigate the precipitation sequence in Mg-Sn-Zn based alloy with the addition of Y. Further, the changes in mechanical properties during the precipitation process were studied. The results showed a huge difference between the alloys. New Mg-Sn-Y phase/compound appeared in the as-cast state. This phase/compound does not dissolve in the a-Mg during the solution treatment (440°C for 96 hrs) and aging up to 16 days at 225°C. The alloy exhibits high hardness levels for a long time at high temperature, i.e. it does not overage in contrary to the base alloy. It seems that the precipitates are Y free, however Y slows down the over aging process. In order to elucidate the contribution of Y to the structural stability of Mg-Sn-Zn based alloy, XRD, TEM, SEM and EDS analyses will be detailed.

Materials Design Approaches and Experiences II: Superalloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee
Program Organizers: Michael G. Fahrmann, Special Metals Corporation; Yunzhi Wang, Ohio State University; Ji-Cheng Zhao, General Electric Company; Zi-Kui Liu, Pennsylvania State University; Timothy P. Gabb, NASA Glenn Research Center

Tuesday AM Room: 202B
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Michael Fahrmann, Special Metals Corporation; Ji-Cheng Zhao, General Electric Company

8:30 AM Introductory Comments by M. Fahrmann, Special Metals Corporation

8:40 AM Invited
Accelerated Insertion of Materials and Processes for Aircraft Engine Applications: *Deborah DeMania Whitis*[1]; Andrew P. Woodfield[1]; J. Pfaendtner[1]; S. Srivatsa[1]; David P. Mourer[1]; D. Wei[1]; Michael F. Henry[2]; L. Jiang[2]; [1]GE Aircraft Engines; [2]GE Global Research Center

Material and process development for aircraft engines has, in the past, required long and costly experimental programs, imposing a significant barrier to the insertion and exploitation of new materials. With the advent of computer modeling and simulation of materials processing, and the accelerated insertion of materials (AIM) approach, we have begun to provide the tools to industrial materials designers that they need to increase productivity and reduce cost of alloy development. This presentation will provide an overview of the implementation of the AIM approach at GE Aircraft Engines. The integration of materials models, historical databases, and analysis tools have allowed us to more rapidly downselect new alloys and manufacturing processes, responding quickly to the design requirements of a particular component and engine environment. Current progress in applying these techniques to nickel-based superalloys and titanium alloys will be reviewed.

9:05 AM Invited
Transport Phenomena and Macrosegregation in VAR and ESR: *Matthew John M. Krane*[1]; Shawn A. Cefalu[1]; Kent J. VanEvery[1]; Dymtro Zagrebelnyy[1]; [1]School of Materials Engineering, Purdue University

The heat, mass and momentum transfer and electromagnetics present during electroslag and vacuum arc remelting (ESR and VAR) are modeled and sump profiles, macrosegregation patterns and local solidification times are predicted. These results are studied as functions of process parameters and ingot geometry. During ESR of superalloys, a maximum in macrosegregation is found as a function of filling velocity in the flow regime dominated by buoyancy, and the growing influence of electromagnetics at higher melt rates is discussed. Comparisons are made to experimental data from industrial ingots. During VAR of titanium alloys, DC current levels are generally much higher than the AC current in ESR, so the sump flow is controlled by Lorenz forces leading to different segregation patterns. Scaling analyses of the fluid flow in the sump give some insight into the effect of process parameters on the fluid velocities and the tendency to segregate.

9:30 AM
Semi-Experimental Approach of Optimizing Castability and Homogenisation of Ni-Base Superalloy: *Muthiah Ganesan*[1]; David Dye[1]; Peter D. Lee[1]; [1]Imperial College London

Increased creep rupture life and strength with acceptable scrap rates and cost are just some of conflicting requirements presently facing designers of heavily alloyed Ni-base superalloy single crystals. Both component castability and solution heat treatability have become increasingly difficult. We have recently proposed an improved statistical approach for accurately characterizing microsegregation in multicomponent alloys. Here, we demonstrate how applicability of this new approach can be further extended via predictive methods (e.g. thermodynamic and regression modeling) to help make improvements in the processing of existing and new alloys. Examples provided include assessment for thermosolutal convection and thus freckle formation, predictions of incipient melting temperatures and alloy susceptibility to topologically closed packed phase formation in a segregated alloy. Empirical equations predicting non-equilibrium phase transformation temperatures and segregation parameters are also provided and compared to CALPHAD-based thermodynamic predictions. Interactive effects between alloying elements are identified and the implications for alloy design discussed.

9:50 AM Invited
Phase Field Modeling Qualitative and Quantitative Microstructural Evolution in Ni-Base Alloys: *Jeff P. Simmons*[1]; Youhai Wen[2]; B. Wang[3]; Y. Wang[3]; [1]U.S. Air Force; [2]UES Inc; [3]Ohio State University

The Phase Field method has been an important and popular method in academia for simulating realistic microstructures because of its relative simplicity and its striking similarity to actual experimental observations. Recently, Phase Field methods have begun to be applied to phase transformations problems of industrial import. This presentation discusses successes and difficulties in applying the Phase Field method towards qualitative behavior and quantitative kinetic predictions in Ni-base alloys of practical interest. In particular, diffusion-limited transformations behavior, bimodal particle size distributions, quantitative mean particle sizes and number density evolution in time have been successfully modeled, as has microstructures formation under non-isothermal heat-treat conditions, where nucleation, growth, and coarsening all occur concurrently. Computations in this work were accomplished with the OpenPF open source Phase Field code, originally developed under contract to DARPA under the AIM program.

10:10 AM Invited
Integrated Modeling for the Manufacture of Ni-Based Superalloy Discs from Solidification to Final Heat Treatment: *Sammy Tin*[1]; Ahmad Kermanapur[2]; Peter D. Lee[2]; Malcom McLean[2]; Martin A. Rist[3]; [1]University of Cambridge; [2]Imperial College; [3]Open University

Process models of the various stages of gas turbine disc manufacture have been integrated to simulate the physical and microstructural transformations occurring within a nickel-based superalloy throughout the entire manufacturing route. Production of these critical rotating structural components requires several distinct processing stages: vacuum induction melting, vacuum arc remelting, homogenization heat treatment, cogging, forging, final heat treatment and machining. During the course of these consecutive manufacturing stages, the various thermal and thermomechanical processes lead to significant changes in both the microstructural characteristics and internal stresses in the alloy. Although separate models have previously been developed to simulate the individual processing stages, this talk describes how these models can be integrated to simulate the entire manufacturing process from secondary melting through to the final forging and heat treatment. The implications of such integrated modeling for quality assurance through process control are discussed.

10:35 AM Break

10:55 AM
Application of JMatPro in Alloy Design at ATI Allvac: *Wei-Di Cao*[1]; Ramesh Minisandram[1]; [1]ATI Allvac

The experience of applying JMatPro in alloy design at Allvac will be introduced in this presentation. JMatPro has been applied to the design of new Ni-base superalloy Allvac™ 718PlusTM and new ultrahigh stainless steel Allvac™ S240TM. The methodology was to trace the change of alloy behaviors by systematically changing the chemistry of model alloys. The behaviors include: 1. Type, Chemistry and quantity of alloy phases as function of temperature. 2. Precipitation and coarsening kinetics of various alloy phases. 3. Prediction of thermo-physical and mechanical properties of model alloys. Experiment works were carried out to validate the results, giving an opportunity to evaluate this tool. Examples of successful and unsuccessful experiences will be presented. Improvements desired will be suggested.

11:20 AM

Initial Assessments of Wrought Nickel-Base Superalloys for Space Applications: *Timothy P. Gabb*[1]; John Gayda[1]; [1]NASA Glenn Research Center

Plate superalloys which can be formed, brazed, and welded have potential applications in space, for ducts and heat exchangers in power generation systems. Some proposed applications would require sufficient strength and creep resistance for long term service at 800-930C, with service times to 10,000 h or more. Such long term service can severely tax alloy creep and phase stability, to the potential detriment of other mechanical properties. Several wrought plate superalloys including Hastelloy X and Inconel 617 are being screened with tensile and creep tests to compare their capabilities for these applications. Conventional tensile and creep tests were performed at temperatures up to 930C on specimens extracted from wrought plates. Microstructure and phase evaluations were then undertaken. The results will be discussed along with supporting comparative predictions of a modern alloy design tool.

11:40 AM

A Validation Study of the Predictive Capabilities of the Materials Properties Simulation Software JMatPro: *Michael G. Fahrmann*[1]; [1]Special Metals Corporation

JMatPro is one of the modern computational tools that allows for the simulation of selected thermodynamic, physical, and mechanical properties of various families of alloys. Based on computational thermodynamics and the best available composite models for the material's effective properties, it employs only a few adjustable parameters derived from benchmark alloys of industrial relevance. For an industrial user, the accuracy of the predictions and the range of validity are of paramount importance when utilizing this tool in actual alloy or process development. This paper presents the outcome of a validation study exercised on wrought Ni-base and Ni-Fe-base superalloys. Special emphasis is placed on JMatPro's predictive capabilities for yield strength, minimum creep rate, and stress rupture life. The importance of equilibrium assumptions and a detailed knowledge of microstructural features and length scales is stressed by parametric studies. JMatPro is a registered trademark of Sente Software, U.K.

Materials in Clean Power Systems: Applications, Corrosion, and Protection: Corrosion in Clean Coal Power Plants and Fuel Cells

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee

Program Organizers: Zhenguo Gary Yang, Pacific Northwest National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Michael P. Brady, Oak Ridge National Laboratory

Tuesday AM — Room: 212B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Peter Tortorelli, Oak Ridge National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory

8:30 AM Keynote

Simultaneous Oxidation and Carburisation of Chromia Forming Alloys: *David John Young*[1]; [1]University of New South Wales

Chromium forms both carbides and oxide at moderate and elevated temperatures. Because the carbides are less stable than Cr2O3, they form only when the chromia scale is permeable to a carbonaceous species, or when any scale is removed. On the other hand, because carbon has a much higher permeability in austenitic alloys than does oxygen, internal carburisation is much faster than internal oxidation. Reactions of chromium metal and model binary alloys with mixed gases at 900°C were used to identify condiitions under which chromia scales are permeable to carbon. Reactions of commercial alloys at 600-700°C with CO/CO2 gases demonstrated simultaneous internal precipitation of oxide and carbides.

9:15 AM Invited

Fireside and Steamside Corrosion of Alloys for USC Plants: *Ken Natesan*[1]; Jong-Hee Park[1]; [1]Argonne National Laboratory

A program on fireside and steamside corrosion, in support of ultrasupercritical (USC) plants, was conducted to evaluate the performance of several structural alloys in the presence of mixtures of synthetic coal ash, alkali sulfates, and alkali chlorides and in a steam environment. Experiments address the effects of deposit chemistry, temperature, and alloy chemistry on the corrosion response of alloys at temperatures in the range of 650-800°C. Materials selected for the study included intermediate-Cr ferritic steels, high-Cr austenitic alloys, and Ni-base alloys. Data were obtained on weight change, scale thickness, internal penetration, microstructural characteristics of corrosion products, mechanical integrity of the scales, and cracking of scales. Based on acquired corrosion information, we have proposed a mechanism for accelerated corrosion in the presence of a NaCl-containing oxidizing environment. Corrosion test data will be used to assess the corrosion performance of candidate materials for long-term service in USC plant environments.

9:45 AM

Deposition of Al-Si Precursor onto Mo-Si-B Turbine Materials: *Joshua E. Jackson*[1]; David LeRoy Olson[1]; Brajendra Mishra[1]; Ian Michael Solomon[1]; [1]Colorado School of Mines

The interfacial reactions of Mo-Si-B with Al-Si during processing to achieve mullite high-temperature corrosion protective coating for advanced turbine materials are discussed. Al-Si was deposited on the Mo-Si-B substrate material by both cathodic deposition and cryogenic immersion in liquid Al-Si alloy to achieve adhesion and compositional gradients across the interfacial region. This interfacial region, with Al-Si deposition, is the precursor to the formation of mullite by annealing and then oxidation. The optimum Al-Si content of this metallic layer, which is attained by deposition and silicon diffusion from Mo-Si-B, will be described. Characterization of the constitution and gradients across the interfacial region will be reported. Transport modeling of this precursor layer will be discussed.

10:10 AM

Control of Metal Dusting Corrosion in Fe- and Ni-Base Alloys: *Zuotao Zeng*[1]; [1]Argonne National Laboratory

Metal dusting is a major issue in plants used in the production of hydrogen, methanol-reformer systems, and syngas (H2/CO mixtures) systems that are pertinent to the chemical and petrochemical industries. Usually, metal dusting corrosion has two stages: incubation and growth resulting in propagation of metal dusting pits. The two stages were studied by scanning electron microscopy and Raman scattering to evaluate the scale of the surface oxide in the initiation and propagation of metal dusting attack. The initiation is dictated by the presence of defects and the propagation is determined by the diffusion of carbon into the alloy. These carbon diffusion pathways can be blocked by periodically oxidizing the surface of alloys at moderate temperatures in controlled atmospheres. It was concluded that metal dusting degradation by selecting an alloy with a long incubation time and subjecting it to intermediate oxidation steps.

10:35 AM Break

10:50 AM Invited

The Effects of Dual Environments and Chromia Vaporization on Metallic Interconnect Behavior: *Frederick S. Pettit*[1]; Wes Jackson[1]; Scot Laney[1]; Gerald H. Meier[1]; [1]University of Pittsburgh

In solid oxide fuel cells, metallic interconnects are exposed at 800°C to air at the cathode and fuel (hydrogen) at the anode. Moreover, in the case of metallic alloys that rely upon Cr2O3 for oxidation resistance, the vaporization of Cr2O3 has been shown to poison the cathode electrode. This paper is concerned with the effects of dual environments on the oxidation of alloys, as well as approaches that may be used to inhibit the adverse effects of Cr2O3 vaporization. The studies concerned with dual environments will use silver exposed to air on one side and an argon-hydrogen gas mixture on the other. Inhibition of Cr2O3 vaporization will be described by using nickel and iron base alloys containing chromium and titanium.

11:20 AM
High Temperature Corrosion Behavior of Metals and Alloys under Influence of a Hydrogen Gradient: *Z. Gary Yang*[1]; Gregg Coffey[1]; Dean M. Paxton[1]; Prabhakar Singh[1]; Jeff W. Stevenson[1]; Gordon G. Xia[1]; [1]Pacific Northwest National Laboratory

In clean power systems, components such as the metallic interconnects in high temperature fuel cells are often exposed simultaneously to a hydrogen or hydrogen-rich fuel at one side and air at the other, i.e. in a "dual" environment. Recently it has been found that the oxidation and corrosion behavior of the metals and alloys under the dual exposures can be significantly different from that in a single exposure, either an oxidizing or reducing atmosphere. The anomalous corrosion behavior that is attributed to hydrogen diffusion from the fuel side to the airside is a function of alloy composition and affected by thermal history and water vapor level in surrounding environments. This paper will present the details of our study on different group of metals and alloys, and discuss mechanistic understanding on the corrosion behavior of the metallic materials under the dual exposure conditions.

11:45 AM
Silver-Aluminum Based Air Braze for High Temperature Electrochemical Devices: *Jin Yong Kim*[1]; John S. Hardy[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

Recently, reactive air brazing (RAB) technique has been developed as an effective alternative for high temperature electrochemical devices such as solid oxid fuel cells. One of the concerns related to the silver-based RAB is its endurance under the dual atmosphere such as hydrogen on one side and oxygen on the other side. Since aluminum would be expected to work as an oxygen getter by forming oxides, aluminum was added to the silver-based reactive air braze (RAB) in order to improve the mechanical properties as well as dual atmosphere tolerance of the braze. In this study, the effects of aluminum content and processing conditions on the microstructure and mechanical properties have been investigated. The detailed results to date will be discussed.

Multicomponent-Multiphase Diffusion Symposium in Honor of Mysore A. Dayananda: Metals and Alloys

Sponsored by: The Minerals, Metals and Materials Society, ASM Materials Science Critical Technology Sector, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Yong-Ho Sohn, University of Central Florida; Carelyn E. Campbell, National Institute of Standards and Technology; Richard Dean Sisson, Worcester Polytechnic Institute; John E. Morrall, Ohio State University

Tuesday AM	Room: 203B
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Hiroshi Numakura, Kyoto University; Carelyn E. Campbell, National Institute of Standards and Technology

8:30 AM Invited
Analysis of Interdiffusion Data in Multicomponent Alloys to Extract Fundamental Diffusion Information: *Irina V. Belova*[1]; Graeme E. Murch[1]; [1]University of Newcastle

There is a very large quantity of interdiffusion data existing for multicomponent metallic systems but rather little has been done so far to extract from it fundamental atomistic diffusion information, e.g. defect-atom exchange frequencies, vacancy-wind factors and tracer correlation factors. This potentially rich source of information could be used to provide a sound foundation from which to tailor the microstructure by means of additives and heat treatment. In this paper, we review the new methods available and the recent progress that has been made to extract fundamental diffusion information from interdiffusion data. We focus on two ternary alloy systems Fe-Ni-Cr and Cu-Ni-Fe, both of which also have some tracer diffusion coefficients available for consistency checking.

9:00 AM
Self- and Interdiffusion Studies in Ternary Cu–Fe–Ni Alloys: *Robert Filipek*[1]; Marek Danielewski[1]; Sergiy.V. Divinski[2]; F. Hisker[2]; Christian Cherzig[2]; [1]AGH University of Science and Technology; [2]University of Münster

Interdiffusion in ternary Cu-Fe-Ni system was studied using diffusion couple technique and Darken model for multi-component systems. Nernst-Planck's flux formula assuming a chemical potential gradient as a driving force for the mass transport was used for computation of the diffusion flux in non-ideal multi-component systems. Lattice diffusion of 64Cu, 59Fe and 63Ni radiotracers has been measured in Cu-Fe-Ni alloys of different compositions at 1271 K. Most of the measured penetration profiles show both extended grain boundary-induced part and the initial bulk diffusion induced part. Grain boundary diffusion contribution was consistently taken into account when determining the bulk diffusivities of the components. For the computations the measured tracer diffusion coefficients of Cu, Fe and Ni and the literature data on thermodynamic activities for the Cu-Fe-Ni system were used. The calculated interdiffusion concentration profiles (diffusion paths) are compared with the experimental results.

9:25 AM
Multi-Component, Multi-Phase Diffusion in Metallic Nuclear Fuels: *Dennis D. Keiser*[1]; [1]Argonne National Laboratory

Multi-component, multi-phase diffusion is commonly observed in nuclear fuels during irradiation in nuclear reactors. This diffusion results in the formation of a variety of different phases that directly impact the performance of the fuel. Diffusion occurs within the nuclear fuel materials themselves (driven by things like temperature gradients), and also between the fuel alloy constituents and the cladding alloy constituents. To better understand the diffusion that occurs within these fuels during reactor operation, out-of-reactor studies have been conducted with various fuel and cladding alloys. These studies have generated useful information about the diffusion kinetics in specific fuel systems and about the types of phases that can form as a result of the diffusion. This paper will discuss results from some out-of-reactor studies that have been conducted to look at diffusion in metallic nuclear systems. Results from these studies will be compared with what has actually been observed in irradiated fuel.

9:50 AM
Interdiffusion in Ni-Cr-X (Al, Si, Ge or Pd) Alloys: *Narayana Garimella*[1]; Michael P. Brady[2]; Yong-Ho Sohn[1]; [1]University of Central Florida; [2]Oak Ridge National Laboratory

Interdiffusion in Ni-Cr (fcc γ phase) alloys with small addition of Al, Si, Ge, or Pd was investigated using solid-to-solid diffusion couples. Ni-Cr-X alloys having compositions of Ni-22at.%Cr, Ni-22at.%Cr-6.2at.%Al, Ni-22at.%Cr-4.0at.%Si, Ni-22at.%Cr-1.6at.%Ge and Ni-22at.%Cr-1.6at.%Pd were cast using arc-melt. The diffusion couples were assembled in Invar steel jig, encapsulated in Ar after several hydrogen flush, and annealed at 900°C in a three-zone tube furnace for 168 hours. Experimental concentration profiles were determined from polished cross-section of these couples by using electron probe microanalysis with pure standards. Interdiffusion fluxes of individual components were calculated directly from the experimental concentration profiles, and the moments of interdiffusion fluxes were examined to determine average ternary interdiffusion coefficients. Effects of ternary alloying additions on the diffusional behavior of Ni-Cr-X alloys are presented in the light of the formation and adhesion of protective Cr2O3 scale. This work was financially supported by CAREER award from National Science Foundation (DMR-0238356).

10:15 AM Break

10:25 AM Invited
Three-Dimensional Interdiffusion under a Stress Field in Fe-Ni-Cu and Fe-Ni-Cr Alloys: *Marek Danielewski*[1]; Bartlomiej Wierzba[1]; [1]AGH University of Science and Technology

The model of interdiffusion under the stress field base on the Darken concept of the drift velocity. Regardless of the common acceptance of the original Stokes interpretation, we propose that the velocity appearing in Newton's viscosity law should be the drift velocity. The key results lie in modification of the Navier-Lamé equations for systems, where the concentrations are not uniform and diffusion occurs. The presented coupling

TUESDAY AM

of the Darken method with mass and momentum balance equations allows for quantitative analysis of the transport processes. The diffusion of components depends on the chemical potential gradient and on the stress that can be induced by the diffusion and by the boundary conditions. The energy, momentum and mass transport are diffusion controlled and the fluxes are given by the Nernst-Planck formulae. It is shown that the Darken method is effective for modeling transport processes in Fe-Ni-Cu and Fe-Ni-Cr alloys.

10:55 AM
Diffusion in the Ir-Nb System: *Hiroshi Numakura*[1]; Tatsuru Watanabe[1]; Makoto Uchida[1]; Yoko Yamabe-Mitarai[2]; Eisuke Bannai[2]; [1]Kyoto University; [2]National Institute for Materials Science

The diffusion behaviour of Ir-rich Ir–Nb alloys has been studied by interdiffusion experiments. The chemical diffusion coefficient has been measured in the Ir-rich fcc solid-solution and the $L1_2$ ordered compound Ir_3Nb has been measured in the temperature range between 1750 and 1950°C, using Ir / Ir–8%Nb and Ir–26%Nb / Ir–28%Nb single-phase diffusion couples, respectively. While the diffusion coefficient in the solid-solution phase turns out to be similar in magnitude to the tracer self diffusion coefficient in pure iridium, the diffusion in the $L1_2$ compound has been found to be extremely slow: the diffusion coefficient is about 1/20. The low diffusion rate in the compound phase must be beneficial for high-temperature performance of refractory superalloys based on the Ir–Nb system.

11:20 AM
Diffusion in $Al_{80}Ni_{20}$-Melts: Experiments and Simulation: *Axel Griesche*[1]; Michael-Peter Macht[1]; Günter Frohberg[2]; S. M. Chathoth[3]; Andreas Meyer[3]; S. K. Das[4]; Jürgen Horbach[4]; [1]Hahn Meitner Institute; [2]Technical University Berlin; [3]Technical University Munich; [4]University Mainz

In this contribution we report about the comparison between diffusion measurements and molecular dynamic simulations (MDS) in Al-Ni melts. The direct long-capillary method was used to measure Ni self diffusion and to measure interdiffusion in liquid Al-Ni alloys. Convective contributions to the diffusion measurements were detected and could be corrected by measuring the time dependence of the diffusion coefficients. In addition the indirect quasi-elastic neutron scattering was used to measure convection-free Ni self diffusion. MDS were used to determine self diffusion of Al and to determine also interdiffusion. The comparison between experiment and simulation shows an excellent agreement of the interdiffusion coefficients as a function of temperature. The simulation results reflect also the experimentally found phenomenological relation between self diffusion, interdiffusion and thermodynamic forces in $Al_{80}Ni_{20}$, which was described originally by L.S. Darken.

11:45 AM
Diffusional Reaction Layers in the Rod-Type U-Mo/Al Dispersion Fuel during the Irradiation: *Ho Jin Ryu*[1]; Jong Man Park[1]; Chang Kyu Kim[1]; Yeon Soo Kim[2]; Gerard L. Hofman[2]; [1]Korea Atomic Energy Research Institute; [2]Argonne National Laboratory

U-Mo/Al dispersion fuel has been developed as one of the most promising candidates for a high performance research reactor fuel. Although a diffusional reaction between the U-Mo fuel and Al matrix results in complicated phenomena, it is necessary to understand the reaction layer growth behavior for the estimation of thermal performance and swelling behavior of the dispersion fuel. Reaction layers in rod-type U-Mo/Al dispersion fuels were observed after irradiation in the HANARO research reactor in this study. Scanning electron microscopy and electron probe microanalysis were used to observe microstructures and compositional profiles. The results from the in-reactor irradiation test were compared with out-of-pile results from diffusion couple tests and annealing tests. The effect of reaction layer on the thermal conductivity was analyzed for the calculation of the temperature profile of a fuel element.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials V: New Process for Cu Interconnects and Semiconductor Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee
Program Organizers: Katsuaki Suganuma, Osaka University; Douglas J. Swenson, Michigan Technological; Srinivas Chada, Jabil Circuit, Inc.; Sinn Wen Chen, National Tsing-Hua University; Robert Kao, National Central University; Hyuck Mo Lee, Korea Advanced Institute of Science and Technology; Suzanne E. Mohney, Pennsylvania State University

Tuesday AM — Room: 213B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Douglas Swenson, Michigan Technological; Masanori Murakami, Kyoto University

8:30 AM Invited
Reduction of Electrical Resistivity of Cu Interconnects: *Masanori Murakami*[1]; Miki Moriyama[2]; Susumu Tsukimoto[1]; Kazuhiro Ito[1]; Takashi Onishi[3]; [1]Kyoto University; [2]Toyoda Gosei Company, Ltd.; [3]Kobe Steel, Ltd.

Although Cu was found to be attractive as interconnect materials for ultra-large scale integrated (ULSI) Si devices, the electrical resistance of Cu films was found to increase significantly when the film thickness was thinner than 70nm. In this talk we will first review our recent results on (1) determination of the primary factor to increase the electrical resistivity of nano-scale Cu films, (2) development of a fabrication technique for low resistance Cu films, and (3) formation of extremely thin diffusion barriers between Cu films and insulators by adding a small amount of a second element to Cu interconnects. Then, we will propose a new fabrication process of Cu innerconnects using a sputter-deposition and high temperature reflow technique.

9:05 AM
Good Thermal Stability Cu Films for Advanced Barrierless Metallization: Jinn P. Chu[1]; *C. H. Lin*[2]; [1]National Taiwan Ocean University; [2]Chin-Min Institute of Technology

Copper has a lower electrical resistivity and better reliability to electromigration than aluminum. Therefore, Cu and its alloys have been widely used for metallization in microelectronics and TFT-LCD. However, Cu diffuses readily into Si and SiO_2, and copper silicide forms at low temperatures. Our prior studies showed that doping with a small amount of the insoluble elements such as W increased the thermal stability of Cu films. In this study, Cu films with dilute amounts of insoluble substances, such as WC and WNx, prepared using co-sputtering were characterized. It is found that the thermal stability of Cu has been greatly improved without a barrier layer on Si up to 600°C while maintaining the low resistivity. Various characterization techniques including XRD, FIB, SIMS, TEM, XPS, electrical resistivity and current-voltage (I-V) curve measurements are employed to evaluate the thermal stability of Cu films. The results are discussed.

9:30 AM
Structural and Passivative Behaviors of Cu(In) Thin Film: Jau Shiung Fang[1]; *Hsin Yi Hsieh*[1]; [1]National Formosa University

Self-passivated copper as a gate electrode in the form of $In_2O_3/Cu/In_2O_3/SiO_2$ has been obtained by annealing Cu (In) alloy film in this study. The deposition of a Cu (In) thin alloy film to cause the formation of a $In_2O_3/Cu/In_2O_3$ trilayer and nano structure with low Cu resistivity, good adhesion to SiO_2, and excellent passivation capability. Studied Cu (In) films were prepared by a cosputtering method on Corning Eagle^{2000} glass substrate, and annealing under ambient from 200-500°C for 10-30 min. Structural analyzing were performed by XRD, SEM and TEM. Only copper diffraction peaks can be detected for the as-deposited film. However, annealing the film at an elevated temperature induced a formation of In_2O_3

phase. Resistivity was decrease with increasing annealing temperature, thus induced a low resistivity below 5 μΩ-cm. Results of the preliminary study can be potentially adopted in the application of TFT-LCD gate electrode and copper metallization in integrated circuits.

9:55 AM
Atomic Bond and Property of Al-Cu Alloy with (Al2Cu): *Gao Yingjun*[1]; [1]Guangxi University

Atomic bond of (Al2Cu) in Al-Cu alloy is calculated by using empirical electron theory (EET) in solid. The results are showed that the Cu-Cu bond in phase is the strongest, while the second strongest bond is the Al-Cu bond. They were all stronger than the strongest Cu-Cu bond in pure Cu metal. These are the reason that only precipitation in Al-Cu thin-film alloy can be find when fewer vacancy being in the alloy in higher temperature. The electromigration lifetime of Al-Cu thin-film alloy for interconnects is concerned with the strong atomic bonding in particles which dispersed in matrix of alloy. The precipitation in Al-Cu thin-film alloy can enhance the strength of alloy and increased the electromigration lifetime for interconnects.

10:20 AM Break

10:30 AM
Metal-Semiconductor Amorphous+Nanoscale Ge Phase Composites Produced by Rapid Solidification and by Devitrification of an Amorphous Matrix: *Dmitri Louzguine*[1]; Akihisa Inoue[1]; [1]Tohoku University

Ge-rich alloys containing up to 70 at.% Ge were produced in Ge-Al-Cr-RE (RE-rare earth elements) systems while Si-based amorphous alloys containing up to 60 at.% Si were produced in S-Al-TM systems (TM-transition metals) by the melt-spinning technique. The samples are ribbons of 10-100 μm in thickness. In the present work we summarize our findings on the formation of the nanocrystal-amorphous composites containing nanoscale semiconductive Ge or Ge(Si) solid solution particles homogeneously distributed in the conductive amorphous matrix. Nanocomposites in Si-Al-TM-Ge (TM-transition metals) and some Ge-Al-Cr-RE alloys were produced directly by rapid solidification while in the Ge55Mg35Y10 alloy they were produced by devitrification of the glassy phase on heating. The semiconductive nanoparticles exhibit homogeneous distribution and their size is varied from about 5 to 20 nm. The structure, phase transformations and properties of the samples will be discussed.

10:55 AM
Insulator to Metal Phase Transformation of VO_2 Films upon Femtosecond Laser Excitation: *Sergiy Lysenko*[1]; Valentin Vikhnin[1]; Armando Rua[1]; Zhang Guangjun[1]; Felix Fernandez[1]; Huimin Liu[1]; [1]University of Puerto Rico

The laser induced insulator-to-metal (I-M) phase transition in VO_2 thin films was explored by femtosecond optical pump-probe spectroscopy. The 130 femtosecond laser pulses were applied to study of transient reflection and relaxation processes in VO_2 films with different concentration of oxygen vacancies, deposited by laser ablation on amorphous and crystalline substrates. I-M transition was observed on a 10^{-11}-10^{-9} sec temporal-scale where phase transition can be considered as a competitive process between thermal and light-induced phase transition mechanisms. The laser excitation is able to produce Frenkel and Wannier-Mott vibronic excitons in VO_2. Excited state dynamics in such a system are strongly dependent on the concentration of structural defects and excitation laser power. Analysis of transient reflectivity properties of qualitatively different VO_2 films allows suggesting the excitonic-controlled light-induced insulator to metal phase transition mechanism in VO_2 via an intermediate state.

11:20 AM
Photoinduced Reflectivity Transients in Vanadium Dioxide: *Sergiy Lysenko*[1]; Valentin Vikhnin[1]; Armando Rua[1]; Guangjun Zhang[1]; Felix Fernandez[1]; Huimin Liu[1]; [1]University of Puerto Rico

Vanadium dioxide is phase transition functional material which exhibits first order insulator-to-metal phase transition (PT) at the temperature about 340 K or under optical photoexcitation. The ultrafast pump-probe optical spectroscopy can provide unique information about transient structural and electronic properties of solid. We summarize the results of transient reflectivity for VO_2 thin films in semiconductor and metallic phases; the VO_2 reflectivity data for 130 femtosecond laser pulses was recorded at 295 K and 410 K. It was found that the relative reflectivity changes after photoinduced and thermally-induced PT are different. Such a difference can be related to formation of an excited state during photoinduced PT. Pump-probe reflectivity measurements of VO_2 in metallic phase shows nonlinear optical response with the two-stage relaxation process on the 10^{-9} and 10^{-6} temporal-scales. Structural and carrier dynamic in VO_2 will be discussed in the framework of excitonic model.

11:45 AM
High Reflection and Low Contact Resistance of Ni/Ag(Al) Contact to Thin-GaN LED: *Chiahsien Chou*[1]; Cheng-Yi Liu[1]; [1]National Central University

A high reflective and low contact resistance p-GaN electrode contact is required for n-side up thin-GaN LED structure. Conventionally, Ni-O/Au/Al or Ag metallization scheme was used as p-GaN contact, in which Al or Ag reflector served as a high reflective layer. However, it has been reported that this contact scheme would be significantly degraded during the chip and packaging processes. Especially, Au and Ni-O layer have high absorption coefficient for the ultraviolet region. In this study, we design a Ni/Ag(Al) metallization scheme for the p-GaN electrode contact. Our preliminary results show that this contact scheme has high thermal stability in reflectivity and contact resistance in UV regime. Also, this metal scheme can be used as the electrode contact for n-GaN.

Phase Transformations in Magnetic Materials: Magnetic Nanocrystals and Nanoparticles

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Raju V. Ramanujan, Nanyang Technological University; William T. Reynolds, Virginia Tech; Matthew A. Willard, Naval Research Laboratory; David E. Laughlin, Carnegie Mellon University

Tuesday AM — Room: 213A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Robert D. Shull, National Institute of Standards and Technology; Vincent G. Harris, Northeastern University

8:30 AM Introductory Comments Raju V. Ramanujan, M. Willard, W. T. Reynolds, Jr. and D. E. Laughlin

8:35 AM Invited
Microstructural Development and Influence on the Properties of Nanocrystalline Soft Magnets: *Matthew A. Willard*[1]; Todd Heil[1]; Ramasis Goswami[2]; [1]Naval Research Laboratory; [2]Geo-Centers

Soft magnetic materials consisting of nanocrystallites surrounded by a residual amorphous matrix provide excellent properties, including both low coercivity and high magnetization. They are produced by rapid solidification processing with devitrification by isothermal annealing. Ultimately, to obtain the highest permeability and lowest core losses, the microstructure must be successfully optimized, with grain diameters less than 10 nm and retained amorphous matrix. Typically, this microstructure is developed during an isothermal anneal between 450 and 650°C. This study will examine the influence of composition, kinetics, and microstructure on the magnetic properties of (Fe,Co,Ni)-Zr-B-Cu alloys. Differential thermal analysis, thermomagnetic analysis, x-ray diffraction, and transmission electron microscopy will be used to describe the phase transformations and the resulting structure/property relationships.

9:10 AM Invited
Microstructure-Property Relationships of Rare Earth Nanophase Magnets: *Kazuhiro Hono*[1]; [1]National Institute for Materials Science

Higher performance permanent magnetic materials are strongly desired for electric vehicles and aerospace applications. Candidates are either Nd-Fe-B or Sm-Co based rare earth magnets, the former is widely used for motors but the current sintered magnets do not have sufficient coercivity for the applications at 200C. The latter is for a higher temperature specifications for aerospace applications. In both systems, the control of nanoscale microstructures are essential to improve the properties. One approach is to develop exchange coupled nanocomposite magnets. We will be presenting some examples of microstructure-property studies of melt-spun $Fe/Nd_2Fe_{14}B$ nanocomposite magnets, but due to their isotropic nature, high performance cannot be achieved. To demonstrate the possibility of achieving higher performance magnets, we have fabricated an anisotropic $Sm(Co,Cu)_5/FeCo$ exchange coupled thin films. In addition, we will comment on the controversies on the phase transformation and coercivity in $Sm(Co,Cu,Fe,Zr)_{7.5}$ based magnets based on our recent microstructural investigations.

9:45 AM Invited

Structural Evolution and Process Refinement of Nanocrystalline Exchanged-Coupled Magnetic Films: Shashidhar Joshi[1]; Soack Dae Yoon[1]; Aria Yang[1]; Nian X. Sun[1]; Carmine Vittoria[1]; Ramarsis Goswami[2]; Matthew Willard[2]; *Vincent G. Harris*[1]; [1]Northeastern University; [2]Naval Research Laboratory

Nanocrystalline exchanged-coupled alloys represent the latest development of soft magnetic materials for inductor applications. Although these alloys have been refined for power electronic applications, little work has been done on the processing of these alloys as films. In this paper, we report on the processing and refinement of nanocrystalline exchange-coupled films having compositions $(Ni,Co,Fe)_{88}Zr_7B_4Cu_x$ (x=0,1). Films were processed at a range of temperatures and gas pressures using PLD. The softest magnetic properties (i.e. H_c <0.5 Oe) coincided to a deposition at 300°C in which an fcc grain size (D) was 6-8 nm separated by an amorphous phase of ~ 1 nm. A power law relationship is observed where $H_c \propto D^{3.5}$ for Cu containing alloys and $H_c \propto D^2 2$ for the alloy without Cu. These results are consistent with the nanostructure being dominated by an uniaxial anisotropy. The addition of Cu appears to play a minor role in the crystallization.

10:20 AM Invited

Phase Transformations in High Density Magnetic Nanoarrays: *Raju V. Ramanujan*[1]; Akhilesh Srivasatava[1]; [1]Nanyang Technological University

The development of high density periodic nanoarrays composed of magnetic nanoparticles has several applications in the areas of information storage devices, biosensors and photonics. XRD, SEM, TEM and VSM techniques were utilized in this study of the nanoarrays. Nanowires and nanobowls of cobalt have been prepared by chemical synthesis techniques using porous alumina and polystyrene sphere templates, respectively. Cobalt nanoparticles were synthesized using the reverse micelle technique, vacuum assisted infiltration in the presence of an external magnetic field was used to infiltrate cobalt nanoparticles within the template. Heat treatment of the nanoarrays was then utilized to produce the morphology, size and crystal structure of the array elements possessing the desired magnetic properties. A systematic study of the role of phase transformations occurring during heat treatment was conducted, these results will be presented and discussed.

10:55 AM Break

11:05 AM

Shape Effects in Magnetic Nano-Particles: Marco Beleggia[1]; *Marc J. DeGraef*[2]; [1]Brookhaven National Laboratory; [2]Carnegie Mellon University

It is well known that magnetostatic interactions between nano-particles are sensitive to the shape(s) of these particles. In this talk, we review recent progress in the description of magnetostatic interactions between uniformly magnetized particles of arbitrary shape. The assumption of a uniform magnetization state is justified by the fact that in the small particle limit, magnetic domain walls are energetically unfavorable. The uniformly magnetized state is, hence, a good first order approximation. A Fourier space representation of the particle shape leads to an explicit expression for the magnetometric tensor field, which takes the place of the conventional dipolar interaction field in energy computations. We will provide examples using cylindrical disks and rectangular prisms. It will be shown that, in a triangular array of three thin circular disks, shape-dependent interactions modify the ground state of the array.

11:25 AM Invited

The Effects of Processing Conditions on Chemically Prepared Magnetic Nanoparticles and Nanorods: *Lynn Kurihara*[1]; Michael M. Miller[1]; Marc P. Raphael[1]; David J. Pena[1]; Meghan P. McHenry[1]; [1]Naval Research Laboratory

Chemical routes have been used to prepare magnetic nanoparticles and nanorods. Nanoparticles of Fe, Co, Ni, Fe_xCo_{100-x}, Fe_xPt_{100-x}, $Fe_xNi_{100-x}$and Co_xNi_{100-x}, were prepared using the polyol method or a modified polyol method. Using the polyol process we are able to produce monodispersed particle sizes from a few nanometers to microns in diameter. Depending upon the their size and composition, these particles can span a range of magnetic behavior from superparamagnetic to ferromagnetic. Cobalt nanorods were prepared by electrodepositing the cobalt into alumina membranes.By varying the process conditions, cobalt nanorods can be prepared so that they are c-axis textured such that the hard magnetic axis is parallel to the principal axis of the rods. The phase purity and structure of the nanoparticles and rods were characterized using XRD. Morphologies were determined by AFM, SEM and TEM. Magnetic properties were measured using VSM and magnetic force microscopy (MFM).

Point Defects in Materials: Bulk Metal Diffusion

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Dallas R. Trinkle, U.S. Air Force; Yuri Mishin, George Mason University; David N. Seidman, Northwestern University; David J. Srolovitz, Princeton University

Tuesday AM — Room: 210B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: David N. Seidman, Northwestern University

8:30 AM Invited

Recent Progress in the Theory of Diffusion Kinetics in Materials: *Graeme E. Murch*[1]; Irina V. Belova[1]; [1]University of Newcastle

Diffusion kinetics is that part of diffusion theory that deals with models where the defect-atom exchange frequencies are specified and produces, as its main result, tracer and chemical diffusion coefficients and ionic conductivities. The theory can be used where the input defect-atom frequencies come from various types of simulations and diffusion coefficients are then derived or it can be used in reverse in order to extract defect-atom exchange frequencies and correlation factors from experimentally measured diffusion coefficients. In this paper, we review recent findings on a number of such models including the five-frequency solute model, the random alloy model, vacancy-pair model and the four-frequency interstitial model with examples drawn from metallic alloys, intermetallics, ceramic oxides/silicates and alkali halides.

9:00 AM

A Self-Consistent Mean-Field Model for Diffusion in Concentrated Alloys: *Vincent Barbe*[1]; Maylise Nastar[1]; [1]Commissariat à l'Energie Atomique, Service de Recherches en Métall. Phys.

Building a diffusion model for interacting concentrated alloys based on an atomic scale description of the jump mechanism remains a current challenge. The SCMF theory* calculates the transport coefficients of an alloy through a Master Equation and a self-consistent estimation of a non-equilibrium distribution function expressed in terms of an effective Hamiltonian. Successive upgrades recently proved their efficiency in strongly correlated systems and dilute alloys** for the vacancy mechanism, for which the effective Hamiltonian is directly related to the return paths of the vacancy. Diffusion by the dumbbell mechanism is also addressed, including a realistic description of the set of jump frequencies. The results of the SCMF are discussed on the basis of Monte Carlo simulations in interacting systems. *Nastar et al., Phil Mag A 80 (2000) 155, PCCP 6 (2004) 3611; **Nastar, Phil Mag, 2005, in press.

TUESDAY AM

9:20 AM
Formation Volume of Vacancies in Metals - Correlation with Poisson's Ratio: *Hiroshi Numakura*[1]; [1]Kyoto University

The formation volume of atomic vacancies is an important parameter, particularly in diffusion, but experimental evaluation remains rather difficult. We have calculated the vacancy formation volume in some metals of the cubic structure by molecular statics simulation using embedded-atom potentials. The formation volume is found to be well correlated with Poisson's ratio. For bcc transition metals, it is close to 1 atomic volume in materials with Poisson's ratio close to 1/4, while it decreases almost linearly to 0.6 times atomic volume for those with Poisson's ratio of about 0.4. The simple relation may be utilized to estimate an approximate value for which experimental data are not available.

9:40 AM Invited
Stability and Mobility of Vacancy- and Interstitial-Clusters in Iron from First Principles: *François Willaime*[1]; Chu-Chun Fu[1]; [1]Commissariat à l'Energie Atomique

The properties of point-defect clusters, with up to five vacancies or five interstitials, in iron are studied by DFT-GGA calculations performed on 128 atom supercells using the SIESTA code. The structure of the mono-interstitial is the <110> dumbbell in agreement with experiments and the migration energies of mono-vacancies and mono-interstitials are 0.67 and 0.34 eV against 0.55 and 0.30 eV experimentally. The structures of vacancy clusters are similar to that proposed from empirical potentials - with a second-neighbor configuration for the di-vacancy - and unexpected low migration energies are predicted for the tri- and quadri-vacancies (0.35 and 0.48 eV). Interstitial clusters are made of parallel <110> dumbbells, up to the quadri-interstitial, and of <111> dumbbells at larger sizes. Up to the tri-interstitial, they migrate by the translation-rotation Johnson mechanism. When used as input data in a Kinetic Monte Carlo simulation, these results successfully account for the kinetics of defects in irradiated iron.

10:10 AM Break

10:25 AM Invited
On the Diffusion of Substitutional Solutes in Nickel: *Roger C. Reed*[1]; Chong Long Fu[2]; Maja Krcmar[2]; [1]Imperial College; [2]Oak Ridge National Laboratory

Nickel alloyed with solutes from the d-block of transition metals form the basis of the superalloys - as used for jet engine and industrial gas turbine applications. Vacancy-assisted diffusion controls a number of important phenomena: dislocation creep, precipitate coarsening, oxidation. In this paper, we will report on recent experimental and theoretical work which has aimed to identify patterns of behaviour inherited from the periodic table. Our experimental work rests on the traditional approach of Boltzmann-Matano for the quantification of interdiffusion rates. The theoretical work - using first principles methods - has helped us to rationalise our results. Elements from the centre of the d-block (such as Re) diffuse slowly because of a high activation energy for solute/vacancy exchange. Correlation effects are important only for solutes from the far west and far east of the d-block, e.g. Hf and Au. Predictions for the 3d solutes will also be presented.

10:55 AM
***Ab Initio* Calculations of Point Defect Formation and Migration in FCC Metals**: *Julie D. Tucker*[1]; Dane D. Morgan[1]; Todd R. Allen[1]; Reza Najafabadi[2]; [1]University of Wisconsin - Madison; [2]Lockheed Martin Corporation

Point defects in metals, such as those created in radiation environments, can cause severe degradation of mechanical properties and changes in grain boundary compositions. While vacancy fomation and migration mechanisms are well established, quantitative studies on interstitial configurations and migration paths in FCC metal alloys are quite limited. In this work we study the Fe-Ni-Cr alloy system. For each pure element *ab initio* calculations were performed for vacancies and multiple possible interstitial configurations. The most probable diffusion mechanisms have been further investigated to locate the minimum energy migration paths and associated energy barriers for vacancies and interstitials. Many of these values are not available experimentally and *ab inito* calculations allow us to generate a consistent set of diffusion barriers for use in a kinetic model to simulate radiation induced segregation in Fe-Ni-Cr alloys.

11:15 AM
Point Defect Properties in bcc Fe from an Ab Initio Point of View: *Christophe Domain*[1]; Charlotte S. Becquart[2]; Edwige Vincent[1]; [1]Electricite De France; [2]University of Lille 1

Under ageing or irradiation, the changes of the macroscopic properties such as the hardness or the brittleness are partly due to the evolution of the microstructure, which is governed by the formation and diffusion of point defects (vacancies and interstitials). In order to model the defect evolution, with for instance kinetic Monte Carlo, the knowledge of these elementary properties is required. In this work, point defects as well as small point defect clusters in bcc Fe have been studied and characterised, in terms of their formation and migration energies using density functional theory calculations. The effect of solute atoms will also be discussed. The results are analysed in terms of diffusion properties in order to compare with experimental data and to get input data for upper scale simulations such as kinetic Monte Carlo. Furthermore, point defects properties in bcc Fe are compared to those in other bcc transition metals.

11:35 AM
Diffusion of Small Interstitial Clusters in Fe: Napoleon Anento[1]; *Anna Serra*[1]; Yuri N. Osetsky[2]; [1]Politechnical University; [2]Oak Ridge National Laboratory

Small clusters of self interstitial atoms (SIAs) in Fe can be stable as both <110>-dumbbells and <111>-crowdions. They are mobile and depending on the temperature and size may perform one-dimensional (1D) or three-dimensional (3D) diffusion. We present here the results of modelling of motion of clusters up to 7 SIAs over a wide temperature range. Particular attention was paid to the transition from 3-D to 1-D motion as a function of cluster size. Jump frequency, correlation factors, diffusion coefficient and activation energies for different mechanism were obtained. The results are discussed in application to microstructure evolution of neutron irradiated Fe. This change in orientation has implications in the global microstructure evolution. In this work we study the diffusion of small clusters by atomic computer simulation and we compare the results obtained with two potentials that show different stability for the single dumb-bell interstitial.

Polymer Nanocomposites: Session I

Sponsored by: The Minerals, Metals and Materials Society
Program Organizer: Devesh K. Misra, University of Louisiana at Lafayette

Tuesday AM — Room: 217A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Hilmar Koerner, University of Dayton Research Institute

8:30 AM Invited
3-D Morphology Control of Polymer Nanocomposites Using Electric Fields: *Hilmar Koerner*[1]; Evangelos Manias[2]; Wei Lu[3]; Devesh Misra[4]; Richard Vaia[5]; [1]University of Dayton Research Institute; [2]Pennsylvania State University; [3]University of Michigan; [4]University of Louisiana at Lafayette; [5]Air Force Research Laboratory/MLBP

The term 'nano-composites' invokes parallels to traditional fiber reinforced composite technology and the ability to spatially 'engineer, design and tailor' materials performance for given applications. At present, minimal methodologies exist to realize this degree of control of nanoparticle dispersion in polymers. Herein, AC electrokinetics will be discussed as an approach to provide non-invasive, spatial and orientational control of organically modified layered silicates in polymers. Experimentally, electric fields smaller than 0.5 V/microns align nanoparticles parallel to the applied field direction in an uncured epoxy matrix. Theoretical simulations reveal remarkably rich dynamics. Alignment and exfoliation behavior is significantly affected by particle shape and dielectric properties of nanoparticles and medium. As predicted, thermal coefficient of expansion, tensile strength and dynamic mechanical response depend on nanoparticle orientation and particle/matrix interface. These results present new possibilities for investigating mechanical and transport properties at interfaces and provide the initial step to a 'true' nano-composite paradigm.

9:00 AM Invited
Limitations of Mechanical Improvement for High-Stiffness Polymers Layered-Inorganic Nanocomposites: *Evangelos Manias*[1]; George Polizos[1]; Matthew Heidecker[1]; [1]Pennsylvania State University

Nanometer-thin inorganic fillers are currently being explored for the improvement of the mechanical properties of various polymers. Although the nanocomposite structure offers generally-applicable principles for such enhancements across all polymers, there exist realistic limitations for the extent of improvement that can be achieved. A comparative discussion, across several polymers reinforced by the same layered inorganic fillers, aims in revealing these limitations and tracing their molecular origins to the polymer/filler interactions and the filler characteristics. Simple theoretical arguments quantifying the relevant dependencies will also be discussed.

9:30 AM Cancelled
Catalyzing Carbonization of Polypropylene/Clay Nanocomposites for Synthesizing CNTs and Improving Fire Retardancy

9:55 AM
Development and Characterization of Nanocomposite Films for Extended Lunar and Mars Explorations: Iris V. Rivero[1]; Mark S. Paley[2]; Benjamin G. Penn[3]; Donald O. Frazier[3]; *Keith Jones*[1]; [1]Texas Tech University; [2]AZ Technology; [3]NASA/Marshall Space Flight Center

Gas barrier, chemical resistant, non-toxic ethylene-vinyl alcohol copolymer (EVOH) has been a subject of research at NASA for devising materials useful as liners in habitat structures for extended Lunar and Mars explorations. Specifically, the innate gas barrier property of this copolymer is attractive for habitat structures to secure the high level of hermeticity required in these extreme environments. In addition, factors such as the abrasiveness of Lunar and Martian regolith, micrometeorite showers, and launch and landing exhaust plume introduce the requirement for the liner material to be able to absorb impact-energy and to be tear resistant. An alternative to impart strength to EVOH while retaining its film flexibility is to embed EVOH with single-wall carbon nanotubes (SWCNTs) due to their high strength-to-weight ratio property. This research will present a methodology for dispersing SWNTs in EVOH films followed by an evaluation of the resultant nanocomposite's mechanical and microstructural properties.

10:20 AM
Effect of Stylus Radius on the Scratch Resistance of Polymer Nanocomposites: *Qiang Yuan*[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

The effect of stylus radius and temperature on the scratch resistance of various polymer nanocomposites is studied. Electron microscopy is used to study the micromechanism and the corresponding theoretical explanation is proposed. Moreover, the results are compared with that of the effect of notch radius on the toughness of the polymers and their blends.

10:45 AM Break

10:55 AM
Essential Fracture Work of Polymer-Silicate Nanocomposites: *Yu Qiao*[1]; Jigar K. Deliwala[1]; [1]University of Akron

In a fracture experiment, it was discovered that, when the cooling rate was increased, the fracture toughness of a polyamide 6-silicate nanocomposite increased by 50-100% in a broad range of silicate content up to about 4 wt.%. According to the x-ray diffraction analysis, a high cooling rate would promote the formation of gamma phase and suppress the growth of spherulites. The final failure was dominated by the microvoiding induced by intercalated nanolayer stacks. Further investigation showed that, while the background energy dissipation was strongly dependent on the morphology, the essential near-field fracture work was quite constant. Thus, the dependence of the fracture resistance on the cooling rate should be attributed to the variation in viscosity, instead of the change in crack-tip plastic deformation mechanisms.

11:20 AM
Hierarchical Structure, Properties, and Scratch Resistance of Melt Intercalated Polymer-Clay Nanocomposites: *Anand Mudaliar*[1]; Raghunath Rao Thridandapani[1]; Q. Yuan[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

The addition of nanoparticles or nanoclays to polymers has been extensively studied in recent years to examine the benefit of their addition in enhancing mechanical and thermal barrier properties. However, only a few studies have been devoted towards understanding the deformation behavior. In the present study the hierarchical structure of melt-intercalated polymer nanocomposites and the interaction of clay with the polymer matrix studied by high resolution transmission electron microscopy (HRTEM), x-ray diffraction, and Fourier-transform infrared spectroscopy (FTIR) is combined to establish relationships among scratch/mar resistance and deformation mechanism.

11:45 AM
Effects of Filler and Temperature on the Stability of B-Crystal of Glass Bead Filled Polypropylene: *Qiang Yuan*[1]; Wei Jiang[2]; Lijia An[2]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette; [2]Changchun Institute of Applied Chemistry

The effects of crystallization temperature (Tc), filler content and filler size on the b-crystal and structural stability of original b-crystal of glass bead filled polypropylene (PP) are studied. Differential scanning calorimetry (DSC) measurements indicate that the amount of b-crystal in PP crystals is a function of Tc as well as glass bead content. Additionally, disorder parameter deduced from DSC data is defined for the characterization of structural stability of original b-crystal. The results indicate that the increase of glass bead size would decrease the content and stability of b-crystals.

Sampling, Sensors and Control for High Temperature Metallurgical Processes: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Fundamentals Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Pyrometallurgy Committee
Program Organizer: Robert L. Stephens, Teck Cominco Metals Ltd

Tuesday AM — Room: 8B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Thomas P. Battle, DuPont Company

8:30 AM
Advanced PC Programmed Level Control for Quality Casting Using Precimeter Control System - Part I: Principle and Devices for Flow Control: *Masao Suzuki*[1]; Hans Wilhelm[1]; Jan Strombeck[1]; [1]AI Tech Associates

In order to cast aluminum and its alloys or other metals, it is necessary to accurately control the flow of the metal out of the furnace. This is achieved by measuring and controlling the level of the molten metal surface downstream. Precimeter Control AB has studied and developed intelligent and affordable solutions to make these operations automatic for implementation in automated facilities and quality assurance processes. This principal is based on laser triangulation using digital camera technology. This combination is unique and it features many advantages like a high accuracy and reliable function in difficult industrial environments. The Precimeter Control System consists of a ProH digital camera sensor and incorporates an actuator equipped with a control unit of a tap-out rod, pin-plug rod and or gate-valve. First, the ProH sensor measures the level or height displacement. This measured signal is then converted to a control signal of 4-20mA.

8:50 AM
Advanced PC Programmed Level Control for Quality Casting - Part II: Examples for Casthouse Application Using Precimeter Control System: *Masao Suzuki*[1]; Hans Wilhelm[1]; Jan Strombeck[1]; [1]AI Tech Associates

In order to cast aluminum and its alloys or other metals, it is necessary to accurately control the flow of the metal out of the furnace. This is achieved by measuring and controlling the level of the molten metal surface downstream. Precimeter Control AB has studied and developed intelligent and affordable solutions to make these operations automatic for implementation in automated facilities and quality assurance processes. This principal is based on laser triangulation using digital camera technology. This combination is unique and it features many advantages like a high accuracy and reliable function in difficult industrial environments. The Precimeter Control System consists of a ProH digital camera sensor and incorporates an actuator equipped with a control unit of a tap-out rod, pin-plug rod and or gate-valve. First, the ProH sensor measures the level or height displacement. This measured signal is then converted to a control signal of 4-20mA.

9:10 AM
Challenges in Sampling, Metallurgical Accounting and Reconciliation of Primary Smelting Furnaces: *Jacques J. Eksteen*[1]; Chris Cutler[1]; [1]University of Stellenbosch

Primary smelting furnaces pose unique difficulties in the sampling and materials mass flow and inventory measurement. This paper reviews the requirements for good sampling practice, and how conflicting requirements for good furnace operation and design often makes reliable closure of the metallurgical balance tenuous at best. Design and smelter operation requirements for metallurgical accountability will be reviewed. Typical physical causes of variance and bias will be identified together with methods to eliminate or minimize them.

9:30 AM
Development and Field Testing of a Prototype Aluminum Tapping Camera: *David L. Death*[1]; Luke J. Pollard[1]; Craig A. Rogers[1]; Paul Gwan[2]; Simon Maunder[2]; Flavio Giurco[3]; Peter Herd[4]; Andrew Johnston[5]; [1]CSIRO Minerals; [2]CSIRO Industrial Physics; [3]Aloca; [4]Tomago Aluminium; [5]Comalco

The Tapping Camera Prototype (TCP) is a prototype instrument developed to record and analyse video of the molten metal stream entering the tapping crucible as it is tapped from an aluminium reduction cell. The TCP mounts a wireless camera and periscope to view the molten metal stream via a peephole in the tapping crucible. Real time video is recorded to either digital video tape or computer disk for subsequent video analysis to quantify the bath material drawn into the tapping crucible with the molten aluminium. The images transmitted by the camera are available on a handheld LCD video screen. A peephole in the back of the periscope assembly allows the operator to directly view the tapping stream.

9:50 AM
Local Temperature Control for Die Casting Processes: *Tiebao Yang*[1]; Henry Hu[1]; Xiang Chen[1]; Yeou-Li Chu[2]; Patrick Cheng[2]; [1]University of Windsor; [2]Ryobi Die Casting (USA), Inc.

In high pressure die casting processes, a die plays a critical role in removing heat from the molten metal during the cavity filling and solidification stages. Proper control of die temperature is essential for producing superior quality components and yielding high production rates. This paper proposes a local temperature control scheme for die thermal management based on a computerized intelligent real-time monitoring and control system (IRMCS). In this scheme, cooling water lines are controlled by a pump and solenoid valves. A controller is designed according to the law of die temperature fluctuations. The results obtained from the experiments carried out on a laboratory die casting process simulator indicate that the controller can limit the local die insert temperatures in a given band. Hence, the desired thermal pattern of the die insert becomes achievable.

10:10 AM
Sensors, Laboratory Results and Modern Automatic Control Techniques: *Florian Kongoli*[1]; I. McBow[1]; R. Budd[1]; S. Llubani[1]; [1]FLOGEN Technologies Inc

The data provided by high temperature sensors as well as laboratory results are indispensable for an effective control of high temperature smelting processes. However the difficulty with sampling, the lack of timely laboratory measurements and the availability of proper sensors has impeded the effective use of these data for an efficient control. As a result, the control has been carried out mostly in a classical and static way. It has been always attempted to find the best unique set of feed composition and conditions where the process could run smoothly. However this is associated with natural difficulties and some times has proven impossible due to various reasons. In this work a new approach for an effective control of these processes has been discussed. This not only allows a better control but also a continuous optimization of the processes and can serve as an adequate means for automation.

10:30 AM
Variance and Heterogeneity in Melt Samples from Industrial Ferro-Alloy Furnaces: *Jacques J. Eksteen*[1]; Elton L. Thyse[1]; Makiwe Nkohla[1]; [1]University of Stellenbosch

Any effort to improve the metallurgical control of industrial furnaces employed in the primary extraction of high carbon ferroalloys are bound to fail unless the variability of the melt is not fully accounted for. A number of sampling campaigns were performed on both 3phase AC submerged arc and single phase DC open arc furnaces at different smelter sites in South Africa where High Carbon Ferrochrome is produced. It was found that the heterogeneity between multiple samples during a single taps were much higher that for heterogeneity found within a sample. Moreover, this heterogeneity (as quantified by the relative standard deviation per tap) varied with the operating conditions of the furnace, the element monitored and the type of furnace. A relationship was found between the relative standard deviation of certain elements and the degree of subcooling of the melt.

10:50 AM
Modeling and Control of Magnetite Behavior in Copper Isasmelt Smelting Slag: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Xstrata Copper

One copper Isasmelt furnace has been operated in Xstrata Copper at Mount Isa in Australia since 1992. A thermodynamic model has been developed to simulate the magnetite behavior in copper Isasmelt furnace. The effects of the process parameters on the content of magnetite in copper slag have been predicted and discussed. These process parameters includes SiO2/Fe, matte grade, coke or coal, size of coke or coal, temperature, slag concentrate in the feed, oxygen enrichment, and CaO content in slag.

11:10 AM
Modeling of SEMTECH OPC Signals in P-S Converter: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Xstrata Copper

The SEMTECH OPC system, developed by Semtech AB in Sweden, provides an indication of the endpoints in both converting stages by use of on-line spectroscopy. SEMTECH OPC measures the levels of PbO and PbS emission in the gaseous flame created during the slag and copper making stages of copper conversion. A telescope positioned in the line of sight of the flame detects PbO/PbS emissions. Focused light is transmitted by fiber-optic cable to the server cabinet that contains the spectroscopy hardware. Spectroscopic information is processed, stored, and sent to client screens in the converter control cabins. A thermodynamic mode has been developed to model the SEMTECH signals, in order to determine the slag chemistry, and skimming operations. Some useful information to the operators has been presented.

11:30 AM
Modeling, Control and Optimization of Chemistry and Viscosity of Copper Converter Slag: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Xstrata Copper

The control of converter slag chemistry and viscosity has played an important role in optimizing converter operations. A thermodynamic model of copper P-S converter and a viscosity model of converter slag have been used to predict the chemistry and viscosity of converter slag. The effect of fluxing strategy, the ratio of SiO2/Fe in slag, copper and anode skim charges, and temperature on viscosity and skimming operation of converter slag has been discussed. Some improvements of the industrial operations have been presented.

Simulation of Aluminum Shape Casting Processing: From Alloy Design to Mechanical Properties: Alloy Design and Treatment

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Aluminum Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Solidification Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Qigui Wang, General Motors Corporation; Matthew Krane, Purdue University; Peter Lee, Imperial College London

Tuesday AM — Room: 6D
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Y. Austin Chang, University of Wisconsin

8:30 AM Invited
Atomistic and Microstructural Modeling in Aluminum Cast Alloy Applications: *Christopher M. Wolverton*[1]; [1]Ford Motor Company

The role of atomic-scale computations in the Integrated Computational Materials Science methodology applied to Al castings at Ford will be described. First-Principles atomic-scale computational methods are becoming increasingly used in industry due to their predictive power. However, their computational complexity often limits their use to relatively simple systems. Hence, we have focused on coupling first-principles methods with other computational approaches such as phase-field microstructural evolution models, computational thermodynamics or CALPHAD methods, and cluster expansion methods and kinetic Monte Carlo. The resulting combined models have been applied to alloys in the Al-Si-Cu-Mg system, and applications to precipitation, heat treatment, microstructural evolution, and yield strength have proved fruitful.

8:55 AM Invited
Multi-Scale Structural Design and Processing for Increased Strength in Aluminium Alloys: *Barry C. Muddle*[1]; Jian-Feng Nie[1]; Christopher R. Hutchinson[1]; [1]ARC Centre of Excellence for Design in Light Metals, Monash University

A design protocol for optimizing structural parameters that may be manipulated to maximize strength in aluminium alloys will be outlined. Attention will be directed to alloy composition, grain size, inclusion content and distribution, and dispersed strengthening constituents. In the case of heat-treatable alloys, recent work in wrought materials to rationalize the roles of precipitate identity, form, crystallography and distribution, and the control of dispersed phases will be reviewed. The potential for extending the strategies utilized for strengthening wrought alloys to existing and emerging cast alloys will be examined. The design analysis will be accompanied by a review of existing and emerging approaches to materials processing that permit implementation and control of desired structural changes. Emphasis will be given to multi-stage heat treatments and to the potential for achieving efficiencies in thermal treatment utilising continuous processing. Issues associated with structural stability and the maintenance of property profiles will be discussed.

9:20 AM Invited
Advances in Computational Modeling of Microstructure Evolution in Solidification of Aluminum Alloys: *M. F. Zhu*[1]; C. P. Hong[2]; D. M. Stefanescu[3]; Y. A. Chang[4]; [1]Southeast University; [2]Yonsei University; [3]Ohio State University; [4]University of Wisconsin-Madison

During the last decade, extensive efforts have been dedicated to explore simulation models of microstructure formation in solidification of alloys. Numerical simulation of microstructure evolution can provide us the information for predicting and controlling solidification microstructures, and also the information for improving the fundamental understanding of solidification mechanism. This paper presents an overview of cellular automaton based models and of derived front tracking models, for the simulation of microstructure evolution in solidification of aluminum alloys. The models can be used for modeling meso-scale and micro-scale solidification microstructures. The main achievements in this field are addressed by presenting examples encompassing a wide variety of problems including dendritic growth in binary and ternary alloys, dendritic growth with melt convection, and eutectic and peritectic solidification. Particular emphasis is made on the quantitative aspects of simulations. Finally, future prospects and challenges of solidification microstructure modeling are also discussed.

9:45 AM Invited
CALPHAD/First-Principles Hybrid Approach: The Study of Phase Equilibria and Solidification Behavior of the Al-Ni-Y System: *Dongwon Shin*[1]; William Golumbfskie[1]; Raymundo Arroyave[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University

Al-Ni-Y alloys show excellent glass forming ability and are very promising for structural materials. In order to study the phase equilibria of this ternary system the CALPHAD (Computer Coupling of Phase Diagrams and Thermochemistry) approach is combined with results from first-principles calculations coupled with experimental data and existing CALPHAD models for the constituent binaries. The finite temperature thermodynamic properties including both enthalpy and entropy of ten ternary compounds in this system have been obtained through harmonic and quasi-harmonic approximations. The calculated formation enthalpy and lattice parameters showed good agreement with experiments. From the constructed thermodynamic database, phase equilibria and the liquidus projections were calculated. The solidification sequences for several alloys in this system were also studied through Scheil simulations and were compared with available experimental information.

10:10 AM Break

10:25 AM
Evolution of the Morphology of the Remaining Liquid Pool during the Last Stages of Solidification: *Hongbiao Dong*[1]; Helen Atkinson[1]; [1]University of Leicester

The morphology of the remaining liquid pool at the last stages of solidification is a critical factor affecting the formation of casting defect, such as hot tears. The evolution of morphology has been related to the dihedral angle (2q) in previous studies. In this paper, the effect of solute diffusion in solid phase on the morphology of the remaining liquid and the area fraction of solid phase covered by liquid phase is described for a two dimensional hexagonal network, by assuming a constant dihedral angle. Predicted microsegregation using a numerical model including the proposed description shows a better fit with experimental observation in Al-Cu alloys.

10:50 AM
Modeling of Hydrogen in Aluminum Foundry Melt Treatment Operations: Christian Simensen[1]; *Oyvind Nielsen*[1]; Jan Ove Løland[2]; Steinar Sælid[3]; [1]SINTEF; [2]Alcoa Automotive Castings; [3]Prediktor

Variations in hydrogen content in aluminium melts due to variations in weather, alloy, and process conditions constitute a significant part of the large variability of properties in shaped castings. On one hand, it is well-known that the combination of hydrogen and oxide inclusions promotes the formation of gas porosity in castings, which has a negative impact on mechanical properties. On the other hand, a stable "non-zero" hydrogen content may significantly reduce the reject rate in structural castings by dispersing the macroshrinkage in poorly feed regions into micropores. Thus, the prediction of the hydrogen content in the melt to be cast would be useful for reducing the variability in foundry processes. In the present work, a semi-empirical model describing the evolution of hydrogen in various foundry melt treatment operations is presented. Furthermore, the implementation of this model in an online processes simulation installed at a LPDC foundry is described.

11:15 AM
Validation of Microsegregation Models by the Point-Matrix Approach: *Muthiah Ganesan*[1]; David Dye[1]; Peter D. Lee[1]; [1]Imperial College London

Experimental characterization by the point-matrix approach has proven to be an effective means of validating microsegregation models. The accuracy of this method however depends strongly in the manner in which measured data are subsequently treated to yield segregation profiles. Here, an improved alloy-independent data treatment algorithm recently proposed by the authors for the treatment of randomly sampled data has been applied to accurately examine and validate CALPHAD-type Scheil-Gulliver model predictions of the influence of grain refinement and/or Sr-modification on elemental segregation in an A319-type aluminum alloy. Comparison is also made to other available data sorting strategies and it is demonstrated that the normal procedure of independently sorting elemental data leads to inaccurate estimation of the solidification path of multicomponent alloys. The influence of non-equilibrium interdendritic particles on the consequent sorted profiles is examined.

11:40 AM
Pressure Losses during Pressure Filtration Tests of Liquid Aluminium Alloys: *Xinjin Cao*[1]; [1]Institute for Aerospace Research

There has been an increasing tendency to determine liquid metal quality using the pressure filtration methods in casting industry. However, little has been known about the pressure losses and the distributions as well as their influence on the structures of formed cakes. In this work, the pressure losses across the cakes and filter media during the Prefil Footprinter tests of liquid aluminium alloys have been calculated. The pressure losses and their distributions at the three phases (i.e. initial transient, steady and terminal transient stages) are discussed in details.

Solidification Modelling and Microstructure Formation: A Symposium in Honor of Prof. John Hunt: Columnar to Equiaxed Transition

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Solidification Committee
Program Organizers: D. Graham McCartney, University of Nottingham; Peter D. Lee, Imperial College; Qingyou Han, Oak Ridge National Laboratory

Tuesday AM — Room: 6C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: P. Lee, Imperial College; G. Lesoult, Ecole des Mines de Nancy

8:30 AM Invited
Columnar-to-Equiaxed Transition Revisited: Selim Mokadem[1]; *Wilfried Kurz*[1]; [1]Swiss Federal Institute of Technology

The original Hunt model on the columnar to equiaxed transition (CET) from 1984 allowed for the first time an understanding of this important microstructure transition. In experiments on laser repair of SX superalloys it became evident that even using more precise dendrite models Hunt's approach did not always lead to the right results. Two cases will be presented: (i) Dendritic growth: in off heat flow axis growth a columnar to columnar transition may occur, i.e. the new columnar structure reorients itself by competition into the heat flow direction; (ii) Cellular growth: even if no CET is expected when growth is in the heat flow direction, in off heat flow axis growth nucleation ahead of a divergent front is often observed. These two cases will be analysed and modelled. A model for the CET accompanied by a phase change will also be presented.

8:55 AM Invited
Influence of Macrosegregation on Developing Grain Structures: A Comparison of Predictions with Experimental Observations: Gildas Guillemot[1]; *Charles A. Gandin*[1]; [1]Ecole des Mines de Paris

Following Hunt's model to predict the extent of columnar and equiaxed grain structures in casting, determinisitic approaches have been developed. The main limitation of the deterministic models is yet due to the grain shape approximations. Thus the impingement of an equiaxed grain with a growing front and its further preferred development in the direction of the temperature gradient is not accounted for. This limitation is overcome by considering the development of each individual grain. This is possible using stochastic models in which the growth front of each crystal is considered. Comparison between predictions and in-situ observations of the solidification of a binary alloy in the casting configuration proposed by Hebditch and Hunt is presented. The effect of macrosegregation on the development of the structure is demonstrated. The model offers the possibility to quantify the approximation of using a model in which no consideration of the grain structure is accounted for.

9:20 AM
Recent Developments on Deterministic Models for the Description of the Columnar-to-Equiaxed Transition (CET): *Menghuai Wu*[1]; Andreas Ludwig[1]; [1]University of Leoben

Since the first analytical model of J.D. Hunt in 1984, the so-called columnar-to-equiaxed transition (CET) has become one of most important subjects in solidification research. Today, there exist stochastic and deterministic models for CET predictions. In the present paper an overview on recent developments on deterministic models is given. It is demonstrated that with appropriated multi-phase volume-averaging approaches it is possible to account for nucleation and growth of equiaxed grains ahead of a growing columnar front in the presence of shrinkage-induced feeding flow, thermo-solutal buoyancy driven flow and grain sedimentation. Special modeling assumptions ensure that both CET mechanisms namely 'hard' and 'soft' blocking are described. It is demonstrated that although these models are highly sophisticated, under special conditions they still get similar results as Hunt's classical approach.

9:45 AM
Analysis of Columnar – Equiaxed Transition and Equiaxed Growth of Al – 3.5 Wt% Ni Alloys by In Situ Synchrotron X-Ray Radiography: *Guillaume Reinhart*[1]; Henri Nguyen-Thi[1]; Nathalie Mangelinck-Noël[1]; Thomas Schenk[2]; Joseph Gastaldi[3]; Bernard Billia[1]; Jürgen Härtwig[4]; José Baruchel[4]; [1]L2MP (Laboratoire Matériaux et Microélectronique de Provence); [2]Laboratoire de Physique des Matériaux ; [3]CRMCN; [4]ESRF

Physical mechanisms involved during grain growth and Columnar to Equiaxed Transition (CET) are related to dynamical events during solidification. Therefore, in situ and real time studies are required to improve our understanding of those phenomena. For metallic alloys, the use of X-ray sources provided by 3rd generation synchrotron allows such study.In this communication, we present results of x-ray radiography performed during experiments at the European Synchrotron Radiation Facility (ESRF). Directional solidifications of Al-Ni alloys were analysed to determine growth conditions of dendritic columnar or equiaxed regime. CET was studied by following the microstructure evolution and its characteristics induced by successive jumps of the pulling rate on refined samples. Several outstanding results are obtained: i) blocking mechanism of the columnar structure by equiaxed grains nucleation, ii) propagation of an effective front for fully equiaxed growth, iii) influence of the pulling velocity on the fully equiaxed microstructure morphology and grain density.

10:10 AM Break

10:25 AM Invited
Aspects of Nucleation Mechanisms on the CET in Al Alloys: *Peter Schumacher*[1]; [1]University of Leoben

The columunar to equiaxed transition is central to the casting behaviour of foundry alloys. In particular the phenomenon of grain refinement is best described with the CET. Within the models of CET assumptions on the nucleation mechanism are contained which need to reflect the nucleation mechanism on added heterogeneous substrates. This paper will give an overview of nucleation mechanism observed in aluminium alloys on added grain refining particles and discusses their effect on the CET.

10:50 AM Invited
Two Versus Three Dimensional Simulation of the Columnar-to-Equiaxed Transition: *Hongbiao Dong*[1]; Peter D. Lee[2]; [1]University of Leicester; [2]Imperial College London

The influence of simulating the solute fields in the primary dendrite tip region during the transition from columnar to equiaxed growth in two dimensions versus three is investigated. A mesomodel that combines a cellular automaton description of solid growth together with a finite difference computation of solute diffusion was used. The variations in solute concentration and tip undercooling between 2D and 3D simulations is compared and contrasted to data from prior experimental observations and models. The importance of stereological effects on solute diffusion and the undercooling are discussed.

11:15 AM
Modelling Convection and Grain Impingement in Alloy Solidification Using Front-Tracking: Jerzy Banaszek[1]; Shaun A. McFadden[1]; *David J. Browne*[1]; [1]University College Dublin

Modelling of columnar and equiaxed growth into undercooled metallic liquid is carried out using a front-tracking technique. The methodology is a 2D variant of the models of John Hunt and Steve Flood developed in the 1980s, and here progress on the inclusion of natural convection and grain impingement is reported. Via solution of the flow field occurring due to natural thermal convection in interdendritic and bulk liquid, it is predicted that such flow affects undercooling ahead of the growing columnar front and therefore changes the conditions created for equiaxed solidification. Modelling the growth of such equiaxed grains, both hard and partially soft impingement is simulated. In hard impingement there is no interaction with the solutal or thermal fields of neighbouring grains, but in semi-soft impingement the effects of latent heat evolving from nearby grains is simulated. It is shown that the interaction of such thermal fields alters dendritic growth conditions.

11:40 AM
Influence of Solidification Thermal Parameters on the Columnar to Equiaxed Transition of Al-Zn and Zn-Al Alloys: Alicia Esther Ares[1]; Sergio Fabian Gueijman[2]; Ariane Bonczok[2]; Adriana Candia[2]; Miguel Angel Alterach[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[2]; [1]CONICET/UNAM; [2]University De Misiones; [3]DEMA-FEM-UNICAMP

The prediction of the columnar to equiaxed transition (CET) is of great interest for the evaluation and design of the mechanical properties of castings products. Experiments are conducted to analyze the CET during the upward unsteady state directional solidification of Al-Zn and Zn-Al alloys, under different conditions of superheat and heat transfer efficiencies at the metal/mould interface. A combined theoretical and experimental approach is developed to quantitatively determine the solidification thermal parameters: transient heat transfer coefficients, growth rates, thermal gradients and cooling rates. A numerical procedure combined with experimental results does not give support to CET criteria based either on growth rate or on temperature gradients. Rather, the analysis has indicated that a more convenient criterion should encompass both thermal parameters through the cooling rate. The effects of solidification parameters like cooling rate, growth rate, temperature gradient, melt superheat and solute concentration on the CET position are investigated.

Surfaces and Interfaces in Nanostructured Materials II: Nano-Structured Metals and Oxides

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
Program Organizers: Sharmila M. Mukhopadhyay, Wright State University; Narendra B. Dahotre, University of Tennessee; Sudipta Seal, University of Central Florida; Arvind Agarwal, Florida International University

Tuesday AM — Room: 209
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Narendra B. Dahotre, University of Tennessee

8:30 AM
A Cross-Strip Comparison Study of Cu Surface/Interface Electromigration: *Jee Yong Kim*[1]; Nancy Michael[1]; C.-U. Kim[1]; [1]University of Texas at Arlington

With the use of Cu interconnects in integrated circuits, several new technical and theoretical challenges are emerging. One of these is the effect of the interface on Cu electromigration. In this study, the effect of interface materials on electromigration rate at the Cu surface/interface is investigated using a simple cross-strip configuration which enables a direct comparison of interface and surface electromigration rates. Surprisingly, the polarity of void and hillock formation appears to indicate a higher electromigration rate at the Cu/Ta interface than at the Cu/Si3N4 interface, with both being higher than that of the adjacent Cu/ambient surface. Variation in cross-strip configuration, material, thickness, length, test ambient, temperature and current density affect marker formation and indicate that competing mechanisms are active.

8:50 AM Invited
The Secret Life of Nanoparticles: Characteristics of Nanoparticles and Nanostructured Materials that are Frequently Forgotten or Ignored: *Donald Baer*[1]; M. H. Engelhard[1]; A. S. Lea[1]; D. G. Gaspar[1]; K. Pecher[1]; C. -M. Wang[1]; J. E. Amonette[1]; A. A. El-Azab[2]; S. Kuchibhatla[3]; Sudipta Seal[3]; [1]Pacific Northwest National Laboratory; [2]Florida State University; [3]University of Central Florida

The literature is increasingly filled with images of nanoparticles and nanostructured materials and along with descriptions of their synthesis and aspects of their properties. Properties of nanoparticles are often influenced by particle size. However, it is possible to read many papers on nanoparticles before leaning that many nanoparticles are structurally unstable and that aspects of their history, environment and even proximity to other nanoparticles may also alter their physical or chemical properties. The dynamic properties of these particles can explain some altered chemical properties. Although it is generally accepted that nano-structured materials are mostly surface or interface, the impacts of the nature of that surface are often ignored or minimized. The properties of individual nanoparticles or isolated nanoparticles can also be altered when they are assembled or packed into aggregate systems. Some of these effects will be described based on studies of iron oxide and ceria oxide nanoparticles.

9:30 AM
N-Implantation Induced Phase Separation in Zr-Cu Film: *Hirono Naito*[1]; Shinji Muraishi[1]; [1]Tokyo Institute of Technology

The phase-transition, -separation in proportion to N has been investigated for Zr-Cu alloy by means of N-implantation. Since Zr shows negative enthalpy change with N, whereas Cu shows positive, the different sign of formation and mixing enthalpy would yield selective nitriding of Zr against Cu and simultaneous phase-separation structure consisting of ZrN with metallic ZrCu phase. The 200 nm thick of Zr and Zr-10at%Cu films have been prepared and N-implantation has been conducted with beam energy of 100keV and dose of 1.0x1017 ions/cm2. Selective nitriding behavior in Zr-Cu has been made by XPS chemical shift analysis. The evolution structure with lattice constant change in proportion to N has been observed by TEM to evaluate phase separation sequence in Zr-Cu-N system.

9:50 AM Invited
Nickel Nano-Interlayers for CrN Coatings on Steels: *Ray Y. Lin*[1]; Pravahan Salunke[1]; [1]University of Cincinnati

Effects of nickel nano interlayers on the property of CrN coated steels were investigated. The nickel interlayer was prepared with electroless plating to various thicknesses of up to 100 nm. CrN coatings were sputter deposited over the nickel interlayer. The coating crystal structures, microhardness, and scratch resistance were determined. Results showed that the nickel interlayer improved the microhardness and scratch resistance of CrN coated steels. To further enhance coating performance, an infrared heat treatment at 400°C and 2 minutes was carried out. With infrared heat treatments, the beneficial effect of the Ni interlayer was clearly exhibited. Without the Ni interlayer, after heat treating the CrN coated steel, severe cracks on coatings were observed. Heat treatment induced cracks on the CrN coatings were completely eliminated with a thin Ni interlayer.

10:30 AM
Oxidation of Ta at Low Temperatures and Its Implications for Cu Interconnect Technology: *Nancy L. Michael*[1]; Dongmei Meng[1]; Choong-Un Kim[1]; [1]University of Texas at Arlington

Ta is considered a stable material and is commonly used as a barrier for nanometer range Cu interconnects. While it is true that the Cu/Ta interface shows little intermixing under normal conditions, the presence of oxygen leads to formation of Ta_xCu_yO compounds and degradation of Cu. Because low dielectric constant materials allow ambient gasses to reach the Ta barrier, Ta degradation through oxidation is possible within the structure. The ability of nanoscale Ta films (~20nm) to protect Cu in the presence of oxygen is the subject of this study. Using both blanket wafers and real interconnect structures, the influence of oxygen on Ta and the Ta/

Cu interface is investigated at temperatures below 350°C. In this study we find that even at these low process temperatures Ta is readily oxidized beyond its native oxide thickness. Using XPS and TEM, the sequence of Ta film oxidation and Cu degradation is developed.

10:50 AM
Surface Amorphization in Zr Alloy Films via Ni Implantation: *Shinji Muraishi*[1]; Hirono Naito[1]; Tatsuhiko Aizawa[2]; [1]Tokyo Institute of Technology; [2]Asia SEED

Multi component Zr based glassy alloy is promising candidate for structural material and protective coating because of its high strength and good surface roughness. The good fabrication nature and stability is derived from enhanced mixing enthalpy and radius differences between selected constituents. Present work focuses on surface amorphization behavior in Zr and Zr-Cu films in proportion to Ni implantation. TEM observation revealed that Ni-implantation induced surface amorphization of Zr alloy films with thickness of 100nm. Compositional gradient of penetrated Ni has been measured by XPS to exhibit critical concentration for c-a transition is 20at%Ni for pure Zr, 10at%Ni for Zr-10at%Cu. The decrease of Ni concentration in Zr-Cu film for c-a transition indicates that induced amorphization is mainly attributed to chemical interaction between constituents with large negative enthalpy of mixing.

11:10 AM
Void Formation at Aluminun-Spinel Interface under the Influence of an Electrical Field at 620°C: *Gurpreet Singh*[1]; Myongjai Lee[1]; Rishi Raj[1]; Yeonseop Yu[2]; Jeremy Mark[2]; Frank Ernst[2]; [1]University of Colorado at Boulder; [2]Case Western Reserve University

We show that voids can be formed at an aluminum-spinel interface by the application of an electrical potential difference. The mechanism is related to the change in the vacancy potential at the interface when an electrical field is applied. The polarity of the applied electrical potential can either enhance or weaken the vacancy potential. Accordingly, the voids are nucleated when the aluminum film is the positive electrode. The experimental findings will be followed by a unified model for void nucleation which articulates the equivalence of electrical potentials as well as mechanical stress. The pathway for the unified model is the use of electrochemical potential of the atomic and defect species at interfaces.

11:30 AM Invited
Polar-Surface Induced Novel Growth Configurations of Piezoelectric Nanobelts: *Zhong Lin Wang*[1]; [1]Georgia Institute of Technology

The wurtzite structure family has a few important members, such as ZnO, GaN, AlN, ZnS and CdSe, which are important materials for applications in optoelectronics, lasing and piezoelectricity. The two important characteristics of the wurtzite structure are the non-central symmetry and the polar surfaces. The structure of ZnO, for example, can be described as a number of alternating planes composed of tetrahedrally coordinated O2- and Zn2+ ions, stacked alternatively along the c-axis. The oppositely charged ions produce positively charged (0001)-Zn and negatively charged (000-1)-O polar surfaces, resulting in a normal dipole moment and spontaneous polarization along the c-axis. This polar surface gives rise a few interesting growth features. In this presentation, we will focus on a few growth phenomena that are closely related to the polar surface. Some details will be given about the analysis of the nanobelt based materials.

12:10 PM
AFM Characterization and Surface Modification of Nanocrystalline Ni Films: *Kevin Stevenson*[1]; Frederic Sansoz[1]; [1]University of Vermont

Electroplated Ni for thin films and nanowires exhibit improved mechanical properties due to the increased volume fraction of crystal interfaces and surfaces. Advanced mechanical testing at the nanoscale requires a comprehensive understanding of the relation between fabrication and surface properties. The objective of this paper is to obtain a correlation between electrodeposition parameters and grain size using high-resolution atomic force microscopy (AFM). The surface structure of electroplated Ni films was investigated to determine the ideal electrochemical environment to produce grain sizes between 8-80 nm. Pulsed-current deposition and organic additives were critical for obtaining grain sizes less than 30 nm. Standard AFM cantilevers (tip radius~10nm) were found to be insufficient for imaging grains smaller than 30 nm. However, high-resolution AFM cantilevers (tip radius~1nm) enabled accurate surface and grain analysis over the range of produced sizes. This non-destructive approach provided additional insight into the structure-property relationship of thin films at the nanoscale.

The Brandon Symposium: Advanced Materials and Characterization: Atom Probe

Sponsored by: The Minerals, Metals and Materials Society, Indian Institute of Metals, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Srinivasa Ranganathan, Indian Institute of Science; Wayne D. Kaplan, Technion; Manfred R. Ruhle, Max-Planck Institute; David N. Seidman, Northwestern University; D. Shechtman, Technion; Tadao Watanabe, Tohoku University; Rachman Chaim, Technion

Tuesday AM — Room: 206B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: David Seidman, Northwestern University; Danny Shechtman, Technion - Israel Institute of Technology

8:30 AM Invited
3-D Atomic-Scale Analysis of Interfaces in Thin-Film Materials and Devices: *Alfred Cerezo*[1]; [1]University of Oxford

The 3-dimensional atom probe (3DAP) permits the nanochemistry of materials to be observed in 3 dimensions with near-atomic spatial resolution. Although the primary application of the technique has been to the study of engineering alloys, developments in specimen preparation and the use of laser pulsing allow analysis of thin-films and semiconductor devices. With the 3DAP, asymmetry has been observed in the atomic-scale roughness and interdiffuseness of the interfaces in CoFe/Cu/CoFe structures, similar to those used as giant magnetoresistance (GMR) read heads in hard disks. Molecular dynamics (MD) simulations have shown a good match to the atomic-scale chemical data from the 3DAP. The chemistry and morphology of barrier layers in tunnel magnetoresistance (TMR) films has also been investigated as a function of oxidation and annealing, again showing good agreement with MD modelling. Preliminary results have also been obtained on composition fluctuations and interface sharpness in III-V semiconductor multiple quantum-wells.

8:55 AM Invited
Atomic Scale Characterization of Advanced Materials Using 3D Atom Probe: *Muraleedharan Kuttanellore*[1]; Balamuralikrishnan Ramalingam[1]; Mohan P. Pathak[1]; Konduri Satya Prasad[1]; [1]DMRL, Hyderabad India

Our laboratory is engaged in the development of several advanced materials for structural applications at ambient and at high temperatures. These include high strength aluminium alloys, ultra high strength high toughness steels and nickel base superalloys. In each of these classes of materials, a significant contribution to the understanding of the microstructure-property-processing correlations can be made by the use of the unique atomic-scale characterization capability of the 3D atom probe: the morphology and composition of fine precipitates and nano-scale clusters in modified 7010 high strength aluminium alloys; the evolution of the fine carbides and their chemical composition as a function of tempering temperature and times in the ultra high strength high toughness steel; and the alloy partitioning behaviour between γ and γ' in multi-component nickel base superalloys. The presentation will provide an overview of our efforts at addressing these issues.

9:20 AM
A Comparison of the Early-Stages of γ'-Precipitation in Two Ni-Al-Cr Superalloys: *Chantal K. Sudbrack*[1]; Jessica A. Weninger[1]; Chris Booth-Morrison[1]; Ronald D. Noebe[2]; David N. Seidman[1]; [1]Northwestern University; [2]NASA

Atom-probe tomography (APT) is a direct-space post-mortem imaging and chemical analysis technique that characterizes chemical heterogeneities with sub-nanometer resolution in 3D. The nanostructural evolution in two model Ni-base superalloys, Ni-5.2 Al-14.2 Cr and Ni-7.5 Al-8.5 Cr atomic %, with similar solute supersaturations is followed temporally with APT, as the alloys decompose isothermally at 873 K for times varying from 0.17 to 1024 hours. Particular emphasis is placed on the influence of solute composition on the nanostructural properties and phase compositions as they evolve temporally. Small spheriodal precipitates with radii ranging from < 1 nm to 10 nm are observed at a high number density (ca. 10^{23}-10^{24} m^{-3}). Over the time scale investigated, nucleation, growth and coarsening of the γ'-phases is observed. The decomposition behavior is analyzed with established models utilizing nondilute thermodynamics.

9:35 AM
Microstructural Evolution of Nanoscale Precipitation-Strengthened Aluminum Alloys: *Keith E. Knipling*[1]; David C. Dunand[1]; David N. Seidman[1]; [1]Northwestern University

This research is toward developing a new castable and heat-treatable precipitation-strengthened aluminum alloy having coarsening- and creep resistance beyond 375°C. Decomposition of supersaturated Al(Zr) solid solutions occurs initially by the formation of metastable cubic $L1_2$ Al_3Zr precipitates, which are small (ca. 20 nm), coherent, and resist coarsening at temperatures up to 425°C. However, segregation incurred during peritectic solidification results in an inhomogeneous dendritic distribution of Zr, creating a network of interdendritic precipitate-free zones in the precipitated microstructure. By adding a eutectic ternary solute, we exploit the natural tendency for solute partitioning during solidification to obtain a homogeneous dispersion of strengthening precipitates. Specifically, Sc is added which forms nanoscale $L1_2$ Al_3Sc precipitates. Moreover, in the presence of Zr, $Al_3(Sc,Zr)$ precipitates form with substantially improved thermal stability compared to binary alloy, α-Al plus Al_3Sc. Relationships between the microstructure (precipitate size, morphology, distribution, and chemistry) and the observed mechanical properties are discussed.

9:50 AM Break

10:20 AM Invited
Recent Applications of Field Ion Microscopy and 3D-Atom Probe Analysis: *Reiner Kirchheim*[1]; Talaat Al-Kassab[1]; [1]Institut Fuer Materialphysik

(1) Nucleation and growth have been studied in supersaturated Cu-Co alloys using the 3d-atom probe (TAP). Besides the known spherical nuclei cylindrically shaped particles with an aspect ratio of more than 10 were found. The long axis is directing in the soft (100) direction. These findings are supported by 3d-field ion microscopy. (2) Using TAP data and a statistical evaluation of atomic positions in the intermetallic alloy Ti(Nb)Al revealed that Nb-atoms are occupying sites of the Ti-sublattice. (3) Growth and coarsening of oxide particles was studied in internally oxidized Ag-Mg and Ag-Mn alloys by using both TAP and SANS. It could be shown that growth and coarsening are strongly affected by the composition of the oxide/Ag-interface. (4) By using a Cu-Bi bicrystal and a focussed ion beam a TAP-sample containing the corresponding grain boundary was prepared and the Bi-excess of the boundary was measured with the 3d-atom probe.

10:45 AM Invited
Atom-Probe Tomographic Studies of the Temporal Evolution of Nanostructures in Multicomponent Alloys: *David N. Seidman*[1]; [1]Northwestern University

Atom-probe tomographic (APT) and transmission electron microscopy (TEM) studies of the temporal evolution of nanostructures of multicomponent nickel-based alloys, Ni-Al-Cr, are studied in concert with lattice kinetic Monte Carlo (LKMC) simulations. Additionally, Al-Sc-X (X = Mg, Zr or Ti) are studied employing APT, TEM and high-resolution electron microscopy (HREM). The experimental studies involve temporally following the mean radius of precipitates (second phase), number density of precipitates, mean compositions of the precipitates and the matrix, volume fraction of precipitates, and precipitate morphologies; thereby obtaining a complete experimental description of how a multicomponent alloy evolves towards its thermodynamic equilibrium state when aged in a two-phase region. The experimental results are compared with predictions of extant models for nucleation, growth or coarsening, and with LKMC simulation results. It is shown that LKMC simulations in concert with the experimental results allows for a deeper physical understanding of the temporal evolution than either approach alone.

11:10 AM Invited
Solute Segregation at Crystal Lattice Defects Studied by 3-D Atom Probe Analysis: *George David William Smith*[1]; [1]Oxford University

The segregation of solute atoms at crystal defects such as dislocations and grain boundaries can have a profound effect on the properties of materials. Until recently, the techniques available to characterise these segregation processes have been extremely limited. The development of the 3-D atom probe has enabled valuable new information to be obtained regarding the atomic-scale distribution of solute species, including interstitial atoms such as boron, carbon and nitrogen. This presentation will review recent work carried out at Oxford University on the segregation of carbon to dislocations in iron, the segregation of phosphorus to grain boundaries in nanocrystalline nickel, and the segregation of interstitial and substitutional solutes to grain boundaries in iron. Applications to the design and development of new advanced engineering materials will be emphasised.

11:35 AM Invited
Atom Probe Tomography Characterization of Solute Segregation to Dislocations and Interfaces: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory

The level and extent of solute segregation to individual dislocations and interfaces may be visualized and quantified by atom probe tomography. The large volume of analysis and high data acquisition rate of the local electrode atom probe (LEAP) enables the solute distribution in the region of and along the core of dislocations to be estimated. Solute segregation along and normal to interphase interfaces and grain boundaries may also be estimated. Solute segregation at precipitate-matrix interfaces of precipitates as small as 2 nm diameter may be quantified. Examples will be presented of solute segregation in superalloys, neutron irradiated pressure vessel steels and mechanically alloyed, oxide dispersion strengthened (MA/ODS) ferritic alloys. Research at the SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

The James Morris Honorary Symposium on Aluminum Wrought Products for Automotive, Packaging, and Other Applications: Fundamental Studies

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Recycling Committee
Program Organizers: Subodh K. Das, Secat Inc; Gyan Jha, ARCO Aluminum Inc; Zhong Li, Aleris International Inc; Tongguang Zhai, University of Kentucky; Jiantao Liu, Alcoa Technical Center

Tuesday AM — Room: 207A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Subodh K. Das, Secat Inc; Tongguang Zhai, University of Kentucky

8:30 AM Introduction to Prof. Morris by Dr. Subodh Das

8:40 AM Invited
Anisotropy of Aluminum and Aluminum Alloys: *William F. Hosford*[1]; [1]University of Michigan

A Taylor-Bishop & Hill model describes the plastic anisotropy of aluminum single crystals under constrained deformation. A yield criterion with an exponent of eight describes the anisotropy and is useful in metal forming analyses of aluminum. Anisotropy has less effect on the limiting drawing ratio in cupping than predicted by the quadratic Hill yield criterion. The effect of texture on forming limit diagrams is negligible. Predictions of strain-path changes on forming limit curves of aluminum alloy sheets are in reasonable agreement with experiments. Second phase precipitates greatly modify the form of plastic anisotropy. In age-hardened Al-4% Cu single crystals the anisotropy of hardening caused by thin θ' platelets on {100} planes is explained by their rotation as well as deformation. Tension during ageing causes the precipitates to form primarily on the {100} planes normal to the stress whereas compression causes precipitation on the other two sets of {100} planes.

9:05 AM Invited
Recrystallization Kinetics and Texture Development in Aluminum: *Anthony D. Rollett*[1]; Mohammed Alvi[1]; Abhijit Brahme[1]; Hasso Weiland[2]; Jaakko Suni[2]; [1]Carnegie Mellon University; [2]Alcoa Technical Center

Both automated EBSD and computer simulation has been used to study the recrystallization behavior of several aluminum alloys including both kinetics and texture development. In Al-1050, the kinetics are dominated by nucleation and growth from sites close to boundaries in the deformed structure, which has highly elongated grains. These characteristics, combined with site saturated nucleation conditions, leads to exponents in KJMA plots close to unity. The texture development is dominated by strong growth in the cube component, which is found to nucleate in cube oriented bands. The cube grains grow most strongly into the S deformation component, whereas the brass deformation component is the slowest to recrystallize. Computer simulation demonstrated that several factors were significant to texture development. In approximate decreasing order of importance were oriented nucleation, stored energy varying with component, oriented growth (anisotropic boundary mobility), and anisotropic boundary energy. Results for 3003 and 5005 will also be reported.

9:30 AM Invited
Recrystallization Kinetics in Cold-Rolled Aluminum: *Dorte Juul Jensen*[1]; [1]Riso National Laboratory

This presentation focuses on crystallographic orientation aspects of growth during recrystallization. By the electron back scattering pattern method it is shown that cube grains may grow faster than grains with other orientations and that this growth advantage cannot be explained a 40° <111> misorientation relationship. 3 Dimensional X-ray Diffraction (3DXRD) microscopy confirms that cube grains on average grow the fastest but also that there is a large variability in growth rates even among grains with the same orientation. This can strongly affect the overall recrystallization kinetics. Besides, in-situ recording of the kinetics of individual bulk grains, 3DXRD can also be used to map the complete 3D grain shape evolution while annealing the sample on-line in the x-ray beam. These measurements are also presented and used for an analysis of orientation relationships for grain boundary migration during recrystallization.

9:55 AM Invited
Simulation of Microstructure and Texture in Al-Mg-Mn Sheet: *Juergen R. Hirsch*[1]; [1]Hydro Aluminium AS

The microstructure and texture evolution during industrial production of Al-Mg-Mn alloy sheet and the resulting properties are shown and analyzed, based on recently developed integrated process simulation tools. This alloy group is used for beverage cans where anisotropy is strictly controlled, but also for many other applications where optimum forming conditions are required and sophisticated forming simulation methods – including detailed microstructure information - is often applied. The characteristic texture effects and related deep drawing cup ("earing") profiles are analyzed systematically for various initial hot band microstructures achieved by different rolling reductions. Method for the evaluation of the characteristic forming behaviour and earing profiles in Al-sheet are applied which allow the description of cup profiles and their transition in a simple quantitative way. The variation of initial cube texture and the cold rolling texture evolution, incl. minimum rolling strain are predicted to ensure optimum strength and anisotropy.

10:20 AM
Sheet Metal Formability for Anisotropic Materials Based on Stress-Based Forming Limit Curve: *Jeong Whan Yoon*[1]; Thomas B. Stoughton[2]; Robert E. Dick[3]; [1]Alcoa Technical Center; [2]General Motors R&D Center; [3]Alcoa Rigid-Packaging

Since the trend today is to utilize models with full anisotropy in order to more accurately capture the physics of material behavior, the issue of anisotropy of forming limits must also be addressed. A solution to assess formability for an anisotropic material is proposed that rescales the stresses by a factor so that the scaled stresses have the same relationship to a single forming limit curve in a 2D plot in stress-space, as the actual stresses have to the true anisotropic forming limit in 3D space. The rescaling enables engineers to accurately view the formability of all the elements at the same time for a given finite element analysis of an application. Several applications in rigid-packaging are presented to show the validify of the suggested model.

10:40 AM Break

10:50 AM
Rolling History Microstructure-Property Relations of 6022 Aluminum Sheet: *Christina Burton*[1]; [1]Mississippi State University

This paper covers the microstructure analysis and correlating mechanical properties of 6022 aluminum alloy at various stages of the rolling process. The history effect will provide a database for material modeling. The rolling steps comprise reductions of a 6-inch billet to a 3-inch billet to a 1/8-inch sheet, an annealing process of the 1/8-inch sheet, and finally a reduction to the 1/32-inch finish gauge sheet. The microstructural content will be obtained through scanning electron microscope (SEM) analyses, optical microscopy (OM) analyses, and X-ray tomography. Mechanical tensile tests and microhardness tests will be performed on specimens retrieved from the various stages in the rolling direction and short and long transverse directions of the material. 6022 aluminum sheet is used in the automotive industry primarily in closure panel applications.

11:15 AM Invited
The Effects of Transition Metal Element Additions on Concurrent Precipitation and Recrystallization in Al Alloys: *Tongguang Zhai*[1]; Qiang Zeng[1]; Xiyu Wen[1]; Jin Li[2]; [1]University of Kentucky; [2]Beijing Jiaotong University

Continuous cast Al alloys, such as AA5083, AA5754, high Fe-containing AA5754 and AA3004, were studied in this work in terms of their recrystallization behaviors, especially the effects of transition metal element additions in these alloys. Concurrent precipitation occurred during recrystallization, which gave rise to the formation of special textures in these deformed alloys that had a high level of solid solution. It was found that Mn resulted in a strong P {011}<122> texture, Cr led to a strong texture of {113}<110>, and Fe had no significant effect on the concurrent precipitation in these alloys. The recrystallized grain structure was elongated in the rolling direction because of the concurrent precipitation effect in these alloys. TEM observations revealed that fine precipites pinning dislocations was likely to be the mechanism for the formation of these special textures in these alloys.

11:40 AM Invited
Anisotropic Ultrasonic Attenuation in an AA 5754 Aluminum Hot Band: *Chi-Sing Man*[1]; Zhiqiang Cai[1]; Kevin D. Donohue[1]; [1]University of Kentucky

Ultrasound resonance spectroscopy of through-thickness stress waves is an emerging technique for in-line monitoring of texture and microstructures in sheet metals. In this paper a simple phenomenological theory involving an effective viscosity tensor is adopted for the analysis and interpretation of spectroscopic data of through-thickness resonance. Ultrasonic attenuation of through-thickness shear and longitudinal waves in sheet samples of a continuous-cast AA 5754 aluminum hot band and its O-temper counterpart was measured at room temperature with resonance EMATs (electromagnetic acoustic transducers). The frequency range in question was 2 to 10 MHz. For the hot band, the attenuation of each mode was found to be predominantly proportional to the square of the frequency. The dissipation constant pertaining to the fast shear mode was about 4 times that of the slow shear mode, which suggests the presence of a highly anisotropic dislocation structure that resulted from the hot rolling.

12:05 PM
Nanoindentation Study of Strain Gradient Plasticity in Aluminum Alloy 2024: *Yun Jo Ro*[1]; Matthew R. Begley[1]; Sean R. Agnew[1]; [1]University of Virginia

This study was motivated by the hypothesis that strain gradients play an important role in determining the crack growth resistance. Fatigue crack growth resistance of AA2024 within inert vacuum environments degrades with aging. Therefore, the effect of aging on the so-called length-scale parameter, which governs the impact of strain gradients on the plastic flow stress, has been studied using nanoindentation. Measured hardness vs. depth data are plotted according to the Nix and Gao model, and the results show a specific trend in the length-scale parameter with aging. The length scale is large at short aging conditions (< 1 hour at 190°C) and decreases rapidly with increasing aging, until essentially reaching a plateau at the peak aged condition (~ 6 hours.) Speculations as to why an increase in strain gradient effects would correlate with increased crack growth resistance will be offered.

12:25 PM
Modelling Recovery, Recrystallization and Precipitation in AA6111: *Johnson Go*[1]; Warren J. Poole[1]; Matthias Militzer[1]; Mary A. Wells[1]; [1]University of British Columbia

A systematic study has been carried out to examine the microstructure evolution in cold rolled AA6111 during isothermal annealing. Prior to cold rolling, the alloys was severely overaged for 7 days at 250 and 325°C. Experimental results indicate the recrystallization kinetics is controlled by the spatial distribution of precipitates and the extent of recovery in the deformed matrix. At lower annealing temperatures, an extensive amount of recovery occurs before the onset of recrystallization. In addition, clusters of precipitates were found to pin grain boundaries during recrystallization. A model was subsequently developed by combining models for recovery, recrystallization and precipitation. Particular attention was taken to capture the interaction between the three phenomena. The precipitation model takes into account the dissolution or growth and coarsening of precipitates. It will be demonstrated that the model is capable of describing the softening response of the alloys which has been cold rolled 40% thickness reduction.

12:45 PM
Thermodynamic Investigation of the Effect of Na on High Temperature Embrittlement of Al-Mg Alloys: *Shengjun Zhang*[1]; Qingyou Han[2]; Zi-Kui Liu[1]; [1]Pennsylvania State University; [2]Oak Ridge National Laboratory

Sodium is an undesired impurity element in Aluminum-Magnesium alloys. Despite trace amounts of content, it leads to high temperature embrittlement (HTE) due to intergranular fracture which is prone to edge cracking during hot rolling. In the present work, thermodynamic investigation was carried out to reveal the correlations between HTE, phase relations, temperature and Na content of the Al-Mg alloys. The HTE sensitivity and safe zones of the Al-Mg alloys were determined. The effect of Na on hot ductility loss was also studied. It was found: (i) The liquid miscibility gap associated with Na is closely related to HTE; (ii) HTE ascribes the formation of the intergranular Na-rich liquid phase which significantly weakens the strength of the grain boundary; (iii) The Na content should be controlled below the critical content and different proper hot-rolling temperature should be chosen with different Na content in order to suppress HTE and avoid cracking.

The Rohatgi Honorary Symposium on Solidification Processing of Metal Matrix Composites: Processing and Microstructure of MMCs - II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Solidification Committee
Program Organizers: Nikhil Gupta, Polytechnic University; Warren H. Hunt, Aluminum Consultants Group Inc

Tuesday AM — Room: 207B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: David C. Dunand, Northwestern University; Adam Loukus, GS Engineering, Inc

8:30 AM Invited
Structure and Properties of Pressure-Infiltrated Aluminum/Mullite-Microsphere Syntactic Foams: Dorian K. Balch[1]; Olivier Couteau[2]; *David C. Dunand*[2]; [1]Sandia National Laboratory; [2]Northwestern University

Aluminum syntactic foams with densities of 1.4-1.6 g/cm^3 are fabricated by liquid metal infiltration of aluminum into a packed bed of hollow mullite-silica spheres with diameters of about 50 micrometers. The foam structure is first presented, e.g. matrix/sphere interface, infiltration of spheres and chemical reaction between spheres and matrix. The foam compressive behavior measured at room temperature is then discussed in terms of stiffness, strength, damage, energy absorption and load transfer between matrix and spheres. Finally, the foam thermal expansion and creep behavior are presented and discussed within the framework of metal matrix composites.

8:55 AM
Effects of Interfacial Reactions during Solidification on Mechanical Properties in Short Fiber Reinforced AlSi12CuMgNi Composites: Yuanding Huang[1]; *Norbert Hort*[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre

The addition of short fibers to AlSi12CuMgNi matrix alloys causes interfacial reactions between fiber and matrix during casting. These reaction products are influencing the matrix composition as well as the physical metallurgy of the matrix, such as microstructure, aging behavior and mechanical properties. The present work investigates the interfacial reactions of Saffil®- and Kaowool®-fiber reinforced AlSi12CuMgNi composites by means of SEM and TEM techniques. It is shown that the interfacial reaction largely depends on the fiber composition, especially the content of SiO2 in the fiber. The reaction occurring during solidification can drastically influence the subsequent mechanical behavior of the investigated composites. The relationship between the interfacial reaction and mechanical behavior in accordance to ageing is discussed based on the obtained experimental results. The ageing hardening is deteriorated due to the depletion of the alloying element magnesium caused by the interfacial reaction.

9:20 AM
Microstructure Characterization of Al-7%Si-10%B4C Die Casting Composite: *Zhan Zhang*[1]; X.-G. Chen[2]; A. Charette[1]; [1]University of Quebec at Chicoutimi; [2]Alcan International Limited

Recently, Alcan has developed a liquid mixing process to economically produce Al-B4C particulate reinforced composites. Al-7%Si-10%B4C is a composite developed for shape casting. On the microstructure characterization of a die casting part (a box type) with optical and electron microcopies, attention is particularly focused on the distribution of particulates and interactions between B4C particles and matrix. Quantitative analysis results show that solidification process affects the distribution of the reinforcement particulates. Basically, more particulates are located on final solidified zones, such as the section center of the part. However, in this case, the filling mould process seems not to significantly impact on the distribution of particles. Moreover, a reaction layer containing titanium and silicon is detected on the surface of boron carbide particulates. This layer would play an important role in the limitation of interfacial reactions. The mechanism of interfacial reactions and their prevention in this composite will be discussed.

9:45 AM
Solidification Structures and Properties of Zinc-Aluminium/SiC(MMC) Alloys: Alicia Esther Ares[1]; Rubens Caram[2]; *Carlos Enrique Schvezov*[3]; [1]CONICET/UNaM; [2]DEMA-FEM-UNICAMP; [3]UNaM

The Zinc-Aluminum (ZA) alloys can be cast using a variety of casting methods; they can also be forged, extruded and laminated. The fabrication cost is competitive and at present it is used in some vehicle components in transmission and suspension systems. In the present investigation Zinc – Aluminum alloys containing Silicon, Copper and reinforcing particles of Silicon Carbide were cast and analyzed. The analyses were done performing hardness test (HV), quantitative metallography, and dimensional stability tests. Composition analysis of the different alloying elements along the cast samples were done by SEM (Scanning Electron Microscopy) and EDAX (Energy Dispersive X-Ray Microanalysis). Several correlations between properties, structures and compositions were found, showing the increase in hardness with the increase in the volume fraction of SiC particles. The alloys tested for dimensional stability during 1000 hr at 165 ± 2,5°C were very stable.

10:10 AM
Aluminum Composite Castings Incorporating Used and Virgin Foundry Sand as Particle Reinforcements: *Michael A. Belger*[1]; Pradeep

Rohatgi[2]; Nikhil Gupta[3]; [1]Citation Corporation; [2]University of Wisconsin - Milwaukee; [3]Polytechnic University

Incorporating virgin and used sand from foundries into aluminum castings serves as a means toward recycling waste sand, reducing the cost of castings, and improving selected mechanical properties of castings. The research conducted differs from other researcher's prior experimentation in this field because higher volume percentages of finer silica particles were incorporated in aluminum alloys in the virgin and used state. Stir mixing can be used to incorporate up to twenty volume percent of sand and pressure infiltration can be used to incorporate above fifty volume percent of sand. SEM and XRD show some of the silica in the sand is converted to alumina, leading towards higher hardness, stiffness, and abrasion resistance in the castings. Simulations of the compositions used in these studies were conducted using ProCAST to further understand the dispersed sand particle's influence on fluidity of the melt, thermodynamic changes in the system, and fill characteristics.

10:35 AM Break

10:50 AM
Microstructural Analysis of Ni Coated Carbon Fiber Reinforced Al-2014 Composites: Pradeep Rohatgi[1]; *Vindhya Tiwari*[1]; Nikhil Gupta[2]; [1]University of Wisconsin-Milwaukee; [2]Polytechnic University

Ni coated carbon fibers were infiltrated with Al-2014 melt using conventional and a modified squeeze cast process. The process modification involved extending the fibers out of the mold and being air cooled during solidification. The process modification was aimed at reducing the damage to the Ni-coating during the infiltration process. The mismatch between the coefficient of thermal expansion of Ni-coating and carbon fiber was found to be the primary reason leading to the damage, which was minimized by adjusting the cooling rates. This process resulted in significant improvement in retention of the Ni-coating compared to the unmodified process. The solidification pattern in the matrix was established by measuring the copper and nickel contents at various distances from the fiber surface. The contents for both nickel and copper in the matrix decreased with increase in the distance from fiber surface suggesting that solidification of primary α-Al started in the interfiber region.

11:15 AM
Reactions in Cast Aluminum/Silica Composites and Their Effect on Physical and Mechanical Properties: *J. B. Ferguson*[1]; Ben Schultz[1]; Pradeep Rohatgi[1]; Simon Alaraj[2]; [1]University of Wisconsin - Milwaukee; [2]Birzeit University

In order to gain a better understanding of the reactions and strengthening mechanisms in cast aluminum/silica composites synthesized by stir mixing, a critical review of recent work was undertaken and experiments were conducted to incorporate virgin foundry sand into aluminum composites by stir mixing with the use of Mg as a wetting agent. It was determined that composite density increased with increasing reaction time and SiO_2 content. Reactions between the particles and both the wetting agent and the matrix result in extensive conversion of $SiO_2$2 to $MgAl_2$2O_4 and some $Al_2$2O_3 releasing Si into the matrix. The change in mechanical properties with composition and time is also described. A reaction mechanism is proposed to account for these changes which indicates that both physical and mechanical properties can be controlled by controlling the Base Alloy/$SiO_2$2/Mg chemistry and reaction times.

11:40 AM Invited
Development of Innovative Magnesium Based Composite Formulations Using Disintegrated Melt Deposition Methodology: *Manoj Gupta*[1]; S. F. Hassan[1]; Wai Leong Eugene Wong[1]; [1]National University of Singapore

Magnesium based materials are subject of extensive research investigations due to their ability to significantly reduce the weight of engineering devices. For example, magnesium has the capability to reduce 35% of the weight if it replaces aluminium in engineering applications. To make inroads in the weight-critical area currently dominated by aluminium based materials, it is important that new magnesium based materials to be developed with comparable or better properties than aluminium based materials and conventional magnesium alloys. To realize that, experimental investigations were made to reinforce magnesium with metallic particles such as copper, nickel, titanium and molybdenum. Disintegrated melt deposition coupled with hot extrusion was used as the processing methodology to produce near dense samples. Primary focus was to make matrix-reinforcement interface relatively more compatible and to enhance the properties as a result of that. Very interesting and promising results were obtained from these formulations.

12:05 PM
Microstructural Evolution of TiC Particulate Reinforced Mg-13.5Al-0.6Zn Matrix Composite during Partial Remelting: *Hui-Yuan Wang*[1]; Lin Huang[1]; Qi-Chuan Jiang[1]; [1]Jilin University at Nanling Campus

Microstructural evolutions during partial remelting of Mg–13.5Al–0.6Zn alloy and TiC/Mg–13.5Al–0.6Zn composite are investigated by the isothermal treatment at the temperature of 510°. Compared with the unreinforced matrix alloy, the remaining liquid dramatically decreases in the composite; furthermore, also the solid α-Mg grain size in the composite significantly reduces in the partial remelting microstructure. The addition of cerium (Ce), which is combined with Al to form AlxCe compounds during solidification, has little contribution to the entrapment of TiC by the α-Mg grains during partial remelting process. As a result, in addition to very small amount of them eventually entrapped within theα-Mg grains, most of the TiC particulates are present in the remaining liquid phases distributing at the grain boundaries after partial remelting process.

Titanium Alloys for High Temperature Applications - A Symposium Dedicated to the Memory of Dr. Martin Blackburn: Processing of High Temperature Titanium Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Michael W. Peretti, Lyondell Chemical Company; Daniel Eylon, University of Dayton; Ulrike Habel, Munich; Guido C. Keijzers, Del West USA; Michael R. Winstone, DSTL

Tuesday AM — Room: 201
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Andrew P. Woodfield, GE Transportation; Daniel Eylon, University of Dayton

8:30 AM Invited
Processing to Maximize High Temperature Performance of Titanium Alloys: *Stephen P. Fox*[1]; [1]TIMET

The high temperature performance of titanium alloys is a function of chemistry and microstructure, which are in turn a function of the alloy formulation, melting, conversion and heat treatment. Optimization of these processes can be quite different for an application where a balance of strength, fatigue and creep resistance is required, as compared to optimization for ultimate creep performance. Similarly where the critical requirements are oxidation performance, a very different approach to alloying and processing can be required depending on the specific requirements of the application. The different approaches used will be discussed and placed in context of aerospace and automotive applications comparing the different characteristics of TIMETAL®834, TIMETAL®1100, TIMETAL®21S and TIMETAL®XT.

9:00 AM
Microstructural Development and Modeling the Relationships between Microstructure and Tensile Properties of Timetal 550 Using Bayesian Neural Networks: Eunha Lee[1]; *Gopal B. Viswanathan*[1]; Sujoy Kar[1]; Santosh Kaduri[1]; Peter Collins[1]; Hamish L. Fraser[1]; [1]Ohio State University

Alloy Timetal-550 is heat-treated using the Gleeble thermal-mechanical simulator to obtain various microstructures. Samples from each heat-treated condition were tested in tension at RT. Various microstructural features of the tested samples, spanning large length scales, are quantified using stereological procedures. The database consisting of these quantified microstructural features together with the corresponding tensile properties are used to develop a Bayesian Neural Network (BNN) model to predict the relationship between microstructure and RT mechanical properties: yield strength, ultimate tensile strength and elongation. Results indicate that α lath thickness has the most influence on tensile properties considered. The prior β grain size has a smaller effect on UTS and YS, but has a significant effect on the elongation. Grain boundary α layer-width has a significant effect on the UTS and YS. The model will be presented and the effect of individual microstructural features on the RT tensile properties will be discussed.

9:30 AM
A Combinatorial Approach to the Elemental Optimization of a Beta Titanium Alloy Using Directed Laser Deposition: *Ben Peterson*[1]; Peter C. Collins[1]; Dan Evans[2]; Pat Martin[2]; James Williams[1]; Hamish Fraser[1]; [1]Ohio State University; [2]Air Force Research Laboratory

The composition of the alloy Timetal 21S has been selected as a baseline for the development of a new high temperature beta titanium alloy. A combinatorial approach employing directed laser deposition of elemental powders has been used to develop rapidly the new alloy. Subsequently, the tensile properties are assessed in a rapid manner for simulated high-temperature exposures, and the associated microstructures are characterized. These data populate the databases used to train and test fuzzy logic based motels for predicting the mechanical properties. In addition to the base elements (Ti, Mo, Nb, Al, and Si), neutral elements (Zr and Sn), ß-stabilizers (W), and dispersoid formers (B, C, Ge) are being tested as alloying additions. The most promising alloying additions have been identified. 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.

10:00 AM Break

10:30 AM Invited
The Burn-Resistant Alloy Ti-25Cr-15V-2Al-0.2C and Its Potential Application in Aeroengines: *Xinhua Wu*[1]; Michael Loretto[2]; Wayne Voice[1]; [1]Rolls-Royce plc; [2]University of Birmingham

The burn-resistant alloy Ti-25Cr-15V-2Al-0.2C(wt%) was developed in the Materials IRC and has a tensile strength >1200MPa, ductility >20% and is thermally stable up to 500°C. The alloy is commercially competitive because vanadium and chromium are introduced using aluminium-containing master alloys rather than using expensive pure elements. The material has been used to manufacture aeroengine components through forging and powder processing and some examples of applications will be given. Extensive work has been carried out to understand the effect of various elements on the microstructure and mechanical properties. It has been found that too much aluminium gives rise to embrittlement associated with the formation of B2. Carbon reduces oxygen matrix content and grain boundary segregation through transformation of Ti2C carbide that subsequently retards the precipitation of alpha thus confering thermal stability. The resultant Ti(CO) carbide also leads to a finer grain size after thermo-mechanical processing and higher creep resistance.

11:00 AM
The Role of Jominy Tests in Understanding Transformations in TiAl-Based Alloys: *Michael H. Loretto*[1]; David Hu[1]; Xinhua Wu[1]; Aijun Huang[1]; [1]University of Birmingham

The influence of composition and of grain size on the transformation of the high temperature alpha phase during cooling a number of TiAl-based alloys has been studied using standard end-quench Jominy tests. Longitudinal sections of these quenched samples provide information over a wide range of cooling rates of the influence of composition and of the alpha grain size on the solid state transformations which lead either to two phase structures (lamellar, Widmanstatten, feathery) or to single phased structures (massive gamma or to retained alpha). The alloys which have been studied include alloys such as Ti46Al8Nb with and without addition of boron. The observations will be discussed in terms of the factors which favour the massive transformation over those transformations which result in two phase microstructures of gamma and alpha and on the response of the massive microstructure to subsequent aging.

11:30 AM
Development of Damage Tolerant Titanium Aluminides by Equal Channel Angular Extrusion Processing: *Shankar M. Sastry*[1]; Rabindra N. Mahapatra[1]; [1]Washington University

Equal Channel Angular Extrusion (ECAE) processing was used to produce fine grained microstructures in several titanium aluminides including the most recent iterations of the commercial titanium aluminide compositions. ECAE was performed successfully below the eutectoid isotherm which resulted in the conversion of gamma+alpha2 grains into fine, equiaxed grains. The lamellar microstructure was the most difficult to break down. Upon ECAE processing, the intense shearing during BECAE produced duplex grains of size 0.5-2mm. Annealing the ECAE processed alloys at temperatures in the lower a+g region resulted in the formation of fine, duplex grains. Room temperature ductility values, as measured by the percentage elongation before fracture, were 50-90% higher than in conventionally processed alloys. Furthermore, an increase in room temperature yield strength of up to 50% was observed the ECAE processed alloys.

Ultrafine Grained Materials - Fourth International Symposium: Processing and Microstructures II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
Program Organizers: Yuntian T. Zhu, Los Alamos National Laboratory; Terence G. Langdon, University of Southern California; Zenji Horita, Kyushu University; Michael Zehetbauer, University of Vienna; S. L. Semiatin, Air Force Research Laboratory; Terry C. Lowe, Los Alamos National Laboratory

Tuesday AM — Room: 217D
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Ruslan Z. Valiev, UFA State Aviation Technical University; Kazuhiro Hono, National Institute for Materials Science; Zenji Horita, Kyushu University; K. Ted Hartwig, Texas A&M University

8:30 AM Invited
The New SPD Processing Routes to Fabricate Bulk Nanostructured Materials: *Ruslan Z. Valiev*[1]; [1]UFA State Aviation Technical University

Since the mid-1990's the fabrication of bulk nanostructured metals using severe plastic deformation (SPD) has been evolving as a rapidly advancing direction of modern materials science that is aimed at developing materials with new mechanical and functional properties. The principle of these developments is based on grain refinement down to nanoscale level by various SPD techniques. However, within recent years SPD techniques have been applied for producing bulk nanomaterials using some other principles, namely, SPD-consolidation of powders, including nanostructured ones, as well as SPD-induced nanocrystallization of amorphous alloys. This paper is focused on investigations and development of these new trends in SPD processing enabling fabrication of fully dense bulk nanocrystalline metals with a mean grain size of 20-30 nm and homogenous microstructures. We consider physical principles of these routes and present results on the microstructural characterization of several nanocrystalline materials produced as well as on studies of their unique properties.

8:50 AM Invited
TEM Study of the Strain-Induced Low- and High-Angle Boundary Development in ECA-Pressed Commercial Purity Al: *Enrico Evangelista*[1]; Marcello Cabibbo[1]; M. E. Kassner[2]; [1]Polytechnic University Marche; [2]University of California

Cell, subgrain and high-angle boundary evolution under equal channel angular (ECA) pressing was investigated in commercial-purity aluminum using transmission electron microscopy (TEM). Bc route pressing to a strain of about 8 at ambient temperature was investigated. TEM allowed the examination of cells and subgrains with misorientations less than 2-4°. High-angle boundaries (HABs) accounted for about 70% of the boundary population at the highest strain (8 passes). The HABs appear to be substantially formed through the formation of geometrically necessary boundaries (GNBs). The substructural evolution appeared similar to that observed in an earlier study by other investigators on cold rolling of similar purity aluminum. These two processes appear to have similar grain refining potential.

9:10 AM Invited

Microstructure and Texture Development of Al-7034 and 2024 Alloys Processed by ECAP: *Nong Gao*[1]; Marco J. Starink[1]; Shun Cai Wang[1]; Cheng Xu[2]; Terence G. Langdon[2]; [1]University of Southampton; [2]University of Southern California

The evolution of microstructure and texture during equal channel angular pressing (ECAP) of a spray-cast Al-7034 alloy and an Al-2024 alloy were studied through TEM, EBSD and DSC. Microstructural examination showed the grain sizes of both alloys were reduced to the range of 0.3–0.5 µm through ECAP. There is a relatively rapid increase in the fraction of low-angle boundaries during the initial ECAP passes and a subsequent more gradual increase in the fraction of high-angle grain boundaries in subsequent passes. The crystallographic textures and their rotations during ECAP were analysed. The DSC analysis identified the occurrence of several thermal effects involving the formation, coarsening and dissolution of the precipitation phases, and concurrent recrystallization. The heating and ageing response of post-ECAP were studied by microhardness testing of the samples after interrupted heating and ageing treatment to allow a comparison of their respective hardening/softening behaviour between the as-received and ECAP materials.

9:30 AM

3D Atom Probe Investigation of the Nanostructure of a Commercial 6061 Aluminum Alloy Processed by SPD: *Xavier Sauvage*[1]; Gulnaz Nurislamova[1]; Ruslan Valiev[2]; Maxim Murashkin[2]; [1]GPM-CNRS; [2]Institute of Physics of Advanced Materials

Aluminum alloys processed by SPD usually exhibit a significant increase of the mechanical strength and even superplasticity due to the strong grain size reduction. The 6061 commercial aluminum alloy is heat treatable and its remarkably high hardness after aging is due to a fine distribution of nanoscaled precipitates containing Mg and Si. It was shown however that its hardness could be significantly increased thanks to ECAP processing before the aging treatment. The aim of this work is to investigate such nanostructure resulting from SPD to understand these unusual mechanical properties. A solutionized 6061 aluminum alloy was processed by ECAP and aged. Then, the distribution of alloying elements (Mg and Si) was mapped out in 3D at the atomic scale thanks to the 3D Atom Probe technique. In this paper, we would like to discuss the precipitation kinetics induced by SPD and deformation mechanisms leading to high mechanical properties of the alloys.

9:45 AM

Microstructural Analyses of Deformation Induced Crystallization Reactions in Amorphous Al88Y7Fe5 Alloy: *Rainer J. Hebert*[1]; John H. Perepezko[1]; Harald Rösner[2]; Gerhard Wilde[2]; [1]University of Wisconsin-Madison; [2]Forschungszentrum Karlsruhe in der Helmholtz Gemeinschaft

Cold-rolling experiments with amorphous Al88Y7Fe5 ribbons reveal characteristics of crystallization reactions under driven conditions. During initial deformation, Al nanocrystals develop in shear bands. The development of dislocations in deformation induced Al nanocrystals exceeding a size of approximately 11 nm x 5 nm along with a fragmentation of thermally induced dendrites offers a novel approach to tailor the size distribution of the nanocrystals. Autocorrelation images of high-resolution TEM images demonstrate that correlations in the amorphous phase develop not only in the shear bands, but also in the matrix during deformation at near-ambient temperatures. Annealing experiments with deformed and undeformed samples underline the notion of shear transformation zones and demonstrate their influence on thermally induced crystallization. The results highlight novel options for microstructure control in marginal glass formers under driven conditions. The support of the ARO is gratefully acknowledged (JHP, DAAD-19-02-1-0245).

10:00 AM

Torsion Process for Creation of Severe Strain and Ultrafine Grains: *Zenji Horita*[1]; Terence G. Langdon[2]; [1]Kyushu University; [2]University of Southern California

The process of severe plastic deformation (SPD) leads to a significant reduction in grain size in many metallic materials. The SPD may be attained through a torsion process as it is an endless process when a disk is rotated around the center or when a circular rod is rotated about the longitudinal axis. High Pressure Torsion (HPT) is a typical process making use of torsion strain and producing ultrafine grains in the submicrometer or nanometer range. Although the HPT process has been applied to disk samples, recent studies demonstrated that HPT is also applicable to bulk samples. The principle of the torsion process was also used recently in developing the Severe Torsion Straining (STS) process where a rod is moved in the longitudinal direction while introducing torsion strain through rotation. This presentation reviews grain sizes and microstructure features including mechanical properties after these SPD processes.

10:15 AM Break

10:25 AM Invited

Microstructure of Severely Deformed Fe-C Eutectoid Steel and Its Application: *Kazuhiro Hono*[1]; H. W. Zhang[1]; S. Ohsaki[1]; [1]National Institute for Materials Science

Recent studies of cold drawn pearlite steel wires reported fragmentation and substantial decomposition of cementite. Many literatures reported that nanocrystalline microstructure as the typical feature of the white etching layer on pearlite rail tracks. A model experiment by mechanical milling of pearlite demonstrated that the cementite decomposes after long time milling forcing carbon to dissolve in ferrite and to segregate at the grain boundaries. Here, we report the microstructural features of these severely deformed pearlite steels examined by TEM and 3DAP and discuss the mechanism of nanocrystalline structure formation. We found the nanocrystalline ferrite is quite resistant against grain growth, which motivated us to consolidate the power by spark plasma sintering (SPS) to fabricate bulk nanocrystalline Fe-C alloy, which exhibited yield strength of 1,800 MPa, ultimate strength of 3,500 MP with plastic strain of 40% in a compression mode. The microstructural feature of the bulk nanocrystalline Fe-C will be reported.

10:45 AM Invited

Ultrafine-Grained Aluminium Alloys Produced by Accumulative Roll Bonding: *Mathias Göken*[1]; Irena Topic[1]; Johannes May[1]; Heinz-Werner Höppel[1]; [1]University Erlangen-Nürnberg

Accumulative roll bonding (ARB) has been used in recent years to produce ultrafine-grained materials by severe plastic deformation. The ARB process is especially attractive due to its simplicity and technological relevance. Superior mechanical properties, as an increase in strength and in the fracture strain by a factor of two, have been found for technical purity ARB-Al in comparison with cold rolled Al. The surface condition before rolling has a strong influence on the microstructure and on the interlayer bond strength of the material. High roughness and friction show a positive influence on refining the microstructure. The ARB process has been applied on different Al alloys from the 6000 series, as for example AA6016, where also an increase in strength and ductility has been found. The properties of these precipitation hardened alloys will be compared with that of technical purity Al and discussed in terms of microstructure and processing conditions.

11:05 AM Invited

The Effect of Purity Level and Die Cross-Section Geometry on Microstructure Evolution during ECAE of Aluminum: *Ayman A. Salem*[1]; Terence G. Langdon[2]; Terry R. McNelley[3]; Zenji Horita[4]; S. L. Semiatin[1]; [1]Air Force Research Laboratory; [2]University of Southern California; [3]U.S. Naval Postgraduate School; [4]Kyushu University

Microstructure and texture evolution during room temperature ECAE of several lots of aluminum was investigated to establish the effect of purity level and die geometry (i.e., circular vs square) on grain refinement. Aluminum with different purity levels (99.998%Al, 99.99% Al and 99%Al) were subjected to four ECAE passes via route Bc in a 90° square die. Furthermore, the 99.99%Al was deformed in three dies with different cross-sections: a 10-mm diameter circular die, a 50x50-mm square die, and a 6x6-mm square die. The 99%Al showed an ultra-fine microstructure with a grain size of ~1.5-µm and no signs of discontinuously-recrystallized grains. The 99.998%Al, on the other hand, exhibited large discontinuously-recrystallized grains (~20-µm). The 99.99%Al revealed a bimodal microstructure with both ultra-fine grains (~1.5-µm) and moderate-size discontinuously-recrystallized grains (~10-µm). The inhomogeneity of the microstructure was greatest for deformation in the die with circular cross-section and least for material deformed in the square die.

11:25 AM Invited
Microstructure Evolution in Copper during Large - Strain Machining: *Alexander H. King*[1]; Srinivasan Chandrasekar[1]; Dale Compton[1]; Kevin P. Trumble[1]; Travis L. Brown[1]; Lawrence F. Allard[2]; [1]Purdue University; [2]Oak Ridge National Laboratory/High Temperature Materials Laboratory

A study has been made of the microstructure and mechanical properties of machining chips created from pure copper. Varying levels of large shear strain, shear strain rate, and deformation temperature were induced during chip formation by systematically varying the tool geometry and cutting speed. The machining chips were characterized for microstructure and Vickers hardness to map the microstructure evolution over the selected machining-parameter space. Results at low cutting speeds (~1 m/min) show chips composed of ultrafine-grained (UFG) structures with enhanced hardness up to the highest shear strain achieved. At higher cutting speeds, deformation twinning was observed along with elongated UFG structures, and where critical strain values were reached, catastrophic dynamic recrystallization occurred causing a loss in UFG structure and enhanced hardness. These results lead to a processing map for the development of ideal microstructures. We will use this to discuss the prospects for large scale production of UFG copper.

11:45 AM Invited
Influence of Processing Parameters and Annealing on Texture and Microstructure in ECAP'ed Aluminum: Alexander P. Zhilyaev[1]; Keiichiro Oishi[1]; Georgy I. Raab[2]; *Terry R. McNelley*[1]; [1]Naval Postgraduate School; [2]UFA State Aviation Technical University

The influence of ECAP parameters such as relief angle, backpressure and number of passes during equal-channel angular pressing (ECAP) on texture and microstructure in commercially pure aluminum has been studied. Recrystallization behavior during subsequent annealing has also been considered. Material was processed using a 90° die and examined by orientation imaging microscopy (OIM) and transmission electron microscopy (TEM) methods after the initial pressing operation and after four passes by route BC. The microtexture data 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. The OIM data reveal a prominent simple shear texture and deformation bands at a meso scale. Lattice orientations in each band correspond to a texture orientation. High-angle boundaries in the structure correspond to interfaces between the bands. These features may influence recrystallization.

12:05 PM
Structural Peculiarities of UFG Metal after Severe External Influences: *Victor N. Varyukhin*[1]; Borys M. Efros[1]; Vladimir A. Ivchenko[1]; Natalya B. Efros[1]; [1]Donetsk Physics and Technology, Institute of NAS of Ukraine

Nowadays, the problem of producing UFG materials with 10-200 nm grain size is among actively developing scientific problems. In our experiments we took Iridium, Nickel, Tungsten and Copper pretreated with severe plastic deformation and ion implantation. Some interesting results have been obtained by field ion microscopy method. For example, it has been revealed that in Iridium influenced be severe plastic deformation a UFG structure is formed (the grain size of 20-30 nm), but in the bodies of grains there are practically no defects of structure, however, after implantation of argon ions a sub-UFG structure, (sub-grain size of 3-5 nm) is formed, and in the bodies of subgrains there are defects. The sub-UFG structure was also revealed in Nickel and Copper after severe plastic deformation effect (sub-grain size of 3-15 nm), but in the latter case the observed boundary region is broader and the ultra dispersive sub-grain are highly misorientation.

Wechsler Symposium on Radiation Effects, Deformation and Phase Transformations in Metals and Ceramics: Dislocations/Obstacles/Channeling

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Korukonda L. Murty, North Carolina State University; Lou K. Mansur, Oak Ridge National Laboratory; Edward P. Simonen, Pacific Northwest National Laboratory; Ram Bajaj, Bettis Atomic Power Laboratory

Tuesday AM — Room: 208
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Gary S. Was, University of Michigan; Ram Bajaj, Bechtel Bettis Inc

8:30 AM Invited
Deformation and Fracture in Irradiated Metals: *Steven J. Zinkle*[1]; Meimei Li[1]; [1]Oak Ridge National Laboratory

Neutron irradiation at low and intermediate temperatures can cause large increases in the strength of metals. As reviewed by Wechsler and others, heterogeneous deformation in the form of coarse slip bands (dislocation channels) often occurs during mechanical testing of these irradiated metals. This presentation will summarize deformation and fracture mechanisms in irradiated metals. The deformation behavior of irradiated metals can be conveniently represented by radiation-modified Ashby deformation mechanism maps. Dislocation channeling can be considered to be a specialized case of the normal dislocation glide deformation mechanism, whereas irradiation creep represents a deformation mechanism not observed in unirradiated materials. Most of the fracture modes observed in irradiated metals can be directly related to unirradiated fracture mechanisms (transgranular cleavage, ductile intergranular fracture, etc.). One unresolved issue is whether high matrix helium levels can induce new fracture modes in steels irradiated at low temperatures.

8:55 AM
Dislocation Interactions with Stacking Fault Tetrahedron and Frank Loops: *Hyon-Jee Lee*[1]; Lucie Saintoyant[1]; Brian D. Wirth[1]; [1]University of California

Molecular dynamics simulations of the interaction between gliding dislocations and faulted frank loops or stacking fault tetrahedron are presented. An examination of various factors, including dislocation type (edge, screw and mixed), temperature, SFT size, stacking fault energy, dislocation velocity and the interaction geometry lead to the conclusion that SFT are very strong obstacles to dislocation motion. Regardless of dislocation character, the trailing Shockley partial detaches by an Orowan type mechanism, suggesting that shearing of the SFT, and not annihilation or absorption, is the most common result of dislocation interaction. Complete SFT annihilation and absorption into the dislocation core, which is believed to govern the onset of plastic flow localization, may be possible, but only for specific interactions. Dislocation interactions with Frank loops have been observed to produce shear of the loop, or the transformation into a perfect, prismatic loop.

9:15 AM
Microstructural Analysis of Deformation in Neutron-Irradiated Zirconium and Zircalloy-4: *Naoyuki Hashimoto*[1]; T. S. Byun[1]; [1]Oak Ridge National Laboratory

The effects of neutron-irradiation near 80°C on the deformation behavior of hexagonal close packed (hcp) materials, zirconium and zircalloy-4, 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 defect clusters, which resulted in irradiation-induced hardening. Dislocation channel deformation is observed for doses greater than 0.1 dpa, and is coincident with prompt plastic instability at yield. TEM analysis suggests that the loss of work hardening capacity in irradiated zirconium and zircalloy-4 at higher doses is mainly due to dislocation channels that are formed under a high local resolved shear stress, leading to the observed localized deformation.

9:35 AM
Atomic-Scale Mechanisms of Cleared Channels Formation in Neutron-Irradiated Low Stacking Fault Energy fcc Metals: *Yuri Osetsky*[1]; Roger E. Stoller[1]; Steven Zinkle[1]; [1]Oak Ridge National Laboratory

Clusters of self-interstitial atoms (SIAs) and vacancies are formed directly in high-energy displacement cascades in all metallic structural materials. In low stacking fault energy metals they can be faulted and perfect interstitial loops (IL) which are sessile and glissile, respectively, and stacking fault tetrahedra (SFTs). These defects are obstacles for dislocation motion and sources for strengthening, hardening and loss of ductility of irradiated metals. These effects are often accompanied with plastic instability when significant plastic deformation occurs in shear bands which can also be cleared of radiation induced defects, leading to so called cleared channels. In this paper we present results of large-scale atomistic modelling of edge and screw dislocations interaction with IL and SFTs in model Cu crystals. We report a number of different mechanisms and suggest a possible scenario for how shear bands can be cleared of radiation defects. The results are discussed and compared with experimental observations.

9:55 AM
Atomistic Simulations and Dynamic Observations of Dislocation-Defect Interactions: *Brian D. Wirth*[1]; Hyon-Jee Lee[1]; Joshua S. Robach[2]; Ian M. Robertson[3]; C. M. Li[3]; [1]University of California; [2]Northwestern University; [3]University of Illinois, Urbana-Champaign

In the early stages of deformation of irradiated materials, dislocations emitted from grain boundaries and other localized regions of high stress concentration interact with and destroy radiation-induced defects, creating defect-free channels. Although the presence of channels in a wide range of materials is well documented, the atomistic processes responsible for defect annihilation have not been experimentally verified. We present a study of dislocation interactions with defect clusters in copper and gold performed by combining molecular dynamics simulations with dynamic transmission electron microscope studies. Interaction of a dislocation with a stacking-fault tetrahedron can result in shearing of the tetrahedron into two defects, conversion of the tetrahedron to another defect type, as well as unfaulting and annihilation of the tetrahedron. These observations provide insight into irradiation hardening and defect interaction and annihilation mechanisms.

10:15 AM Break

10:35 AM
Deformation Microstructure of Proton-Irradiated Stainless Steels: *Zhijie Jiao*[1]; Gary S. Was[1]; [1]University of Michigan

The deformation microstructure of proton-irradiated stainless steels may play a key role in explaining their irradiation-assisted stress corrosion cracking (IASCC) susceptibility. In the present study, three model alloys (UHP-304, 304+Si, 304+Cr+Ni) with different stacking fault energies (SFEs) were irradiated with 3.2-MeV protons at 360°C to 5.5 dpa and then strained to 12% in 288°C Ar atmosphere. The deformation microstructures of the strained samples were investigated using transmission electron microscopy (TEM). The interaction between dislocations and irradiation-induced defects, characteristics of dislocation channels and dislocation pile-ups and the interaction between dislocation channels and grain boundaries will be reported. The effect of SFE on deformation microstructures, and thus on IASCC susceptibility of stainless steels will be discussed.

10:55 AM
Flow Localization and Fracture in Irradiated FCC Materials: Xianglin Wu[1]; Xiao Pan[1]; *James F. Stubbins*[1]; [1]University of Illinois at Urbana-Champaign

Irradiated material exhibits sharp increases in yield strength and associated reductions in ductility at low to intermediate temperatures, up to 0.4 Tm. Reductions in ductility are troubling since metal alloys are selected for reactor structural applications due to their ability to flow plastically and redistribute stresses. This property also results in high values of fracture toughness. Flow localization is shown to be associate with a critical stress for tensile failure in fcc metals and alloys. The critical stress is the largest stress the material can withstand prior to necking. As the yield strength increases with irradiation exposure, the difference between the material yield strength and the critical stress decreases, leading to an early onset of necking or localized flow. The dependence of the flow localization process on deformation mechanisms, temperature and irradiation exposure is discussed in this paper. Experimental and modeling studies show the related influence on fracture toughness.

11:15 AM
Dose Dependence of True Stress Parameters in Irradiated fcc, bcc, and hcp Metals: *Thak Sang Byun*[1]; [1]Oak Ridge National Laboratory

The metallic materials after irradiation to high doses often experience prompt necking at yield. Such instable deformation without preceding stable deformation makes it difficult to evaluate the radiation effects on the necking and fracture properties. A method to calculate the true stress-true strain behavior for necking deformation is proposed using experimental evidence and theoretical assumptions. The true fracture stress was calculated for a dozen irradiated fcc, bcc, and hcp polycrystalline metals, and the results are discussed focusing on the dose dependence of the true fracture stress and on the relationship with other true stress parameters such as plastic instability stress or strain hardening rate during necking. In most of the test materials the true fracture stress was nearly independent of dose as was the plastic instability stress, while a few bcc metals experience significant embrittlement, with which it failed before yield, and therefore the fracture stress decreased with dose.

2006 Nanomaterials: Materials and Processing for Functional Applications: Nanoscale Magnetics

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: W. Jud Ready, GTRI-EOEML; Seung Hyuk Kang, Agere Systems

Tuesday PM — Room: 214C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Robert D. Shull, National Institute of Standards and Technology; W. Jud Ready, GTRI-EOEML; Seung Hyuk Kang, Agere Systems

2:00 PM Introductory Comments

2:05 PM Invited
Computer Simulation of Domain Structures in Nanoferroics: *Long Qing Chen*[1]; Y. L. Li[1]; S. Choudhary[1]; J. X. Zhang[1]; [1]Pennsylvania State University

This presentation will discuss the phase-field computational approach to ferroic domain structures that involve multiple long-range interactions. It involves the simultaneous solutions to Landau-Lifshits-Gilbert equation for ferromagnetic domains and time-dependent Ginzburg-Landau equations for ferroelectric and ferroelastic domains. The contributions from long-range interactions are obtained by solving the mechanical, electrostatic, and magnetostatic equilibrium equations. Examples to be discussed include ferroelectric domain structures in epitaxial thin films, domain formation and switching in polycrystals of nanoscale grain size, as well as domains and magnetoelectric coupling in nanoscale composites of ferroelectric and ferromagnetic crystals.

2:30 PM
Tailorable Fe_3O_4 Nanocrystals for Use as T_2 Contrast Agents in Magnetic Resonance Imaging: *Alex J. Barker*[1]; Brant Cage[2]; Stephen Russek[2]; Craig Lanning[3]; Robin Shandas[3]; Conrad Stoldt[1]; [1]University of Colorado, Boulder; [2]National Institute of Standards and Technology; [3]Children's Hospital, Denver

Monodisperse Fe_3O_4 nanocrystals of various sizes (10-20 nm) are size controlled synthetically for use as target specific magnetic resonance imaging (MRI) contrast vectors. The crystals are synthesized by time varied thermal decomposition of Fe(III) acetylacetonate ($Fe(acac)_3$)) in the presence of trioctylamine (TOA) and heptanoic acid (HA). The conjugation of 2,3-dimercaptosuccinic acid (DMSA) to the surface of the Fe_3O_4 improves the biostability and aqueous solubility, in addition to supplying a bonding site target molecules. The material and magnetic properties of the Fe_3O_4 ensembles are examined by transmission electron microscopy (TEM), X-Ray diffraction (XRD), superconducting quantum interference device magnetometery (SQUID), and nuclear magnetic resonance (NMR). The particle size, crystal symmetry, magnetic anisotropy, and T_1, T_2 times in de-ionized water are reported. MRI images of 1.5 mL phantoms and aqueous phase T_1, T_2 shortening indicate that the magnetically optimized Fe_3O_4 nanocrystals hold promise for use as size-tailorable contrast agents.

2:50 PM Break

3:00 PM
Magnetic Properties of Mechanically Alloyed and Annealed Fe-40 at. % Al: Q. Zeng[1]; *Ian Baker*[1]; [1]Dartmouth College

This presentation will describe the production and characterization of metastable, nanostructured, disordered b.c.c. Fe-40 at. % Al solid solution from elemental powders using a high-energy ball mill. The effects of milling and subsequent annealing on the formation of disordered nanocrystals, changes in the lattice parameter and grain size, the disorder-to-order transformation, and ferromagnetic-to-paramagnetic transformation were studied by x-ray diffraction, differential scanning calorimetry and magnetic measurements. This work was supported by NIST grant 60NANB2D0120.

3:20 PM
Nanostructured High-Energy Permanent Magnets: Xiangxin Rui[1]; *Jeffrey E. Shield*[1]; [1]University of Nebraska

Two-phase permanent magnets consisting of hard and soft magnetic phases assembled at the nanoscale are the most promising class of high-energy permanent magnets. The magnetic behavior critically depends on the scale of the structure, particularly the grain size of the soft magnetic phase. In this paper, we report on the structure and magnetic properties of two types of cluster-assembled nanocomposite permanent magnets. In the first, ~10 nm Fe clusters are imbedded in an FePt matrix. The fine-scale Fe clusters allows effective exchange coupling, resulting in energy products of 22 MGOe, which are near-record values for isotropic materials. In the second, individual ~10 nm clusters with compositions in the Fe_3Pt/FePt two-phase field have been fabricated. Phase decomposition leads to nanoscale phase separation resulting in complete exchange coupling. Here, energy products close to 20 MGOe have been achieved. These results provide insight into optimum nanostructural features in exchange coupled nanostructured permanent magnets.

3:40 PM
The Effect of Alloying Elements on the Microstructure and Soft Magnetic Properties of Nanocrystalline Fe-Si-B-Cu-Nb Alloys: *Raju V. Ramanujan*[1]; Yanrong Zhang[1]; [1]Nanyang Technological University

Nanostructured magnetic materials are used in novel applications in bioengineering, electronics, data storage and other industries. Soft nanomagnetic materials have been intensively studied following the success of the Fe-Si-B-Cu-Nb nanocrystalline alloys. Copper and niobium are essential alloying elements, they bring about a dramatic change in the microstructure obtained on crystallization of the amorphous alloys. However, the mechanisms by which this change is effected is still unclear. A study using DSC, XRD, CTEM, EDX, VSM and hot stage in situ TEM was conducted, focusing on the synergistic effects between the Cu and Nb atoms. The crystallization mechanisms of amorphous Fe-Si-B alloys was compared to those of Fe-Si-B-Cu, Fe-Si-B-Nb and Fe-Si-B-Cu-Nb alloys. Synergistic composition changes of Cu and Nb at the crystal:matrix interface were found to play a key role in determining the crystal morphology, crystal size and nucleation density. These results and the associated change in magnetic properties will be discussed.

4:00 PM Break

4:10 PM
Polymer-Metal Nanocomposites for Functional Applications: Vladimir Zaporojtchenko[1]; Ulrich Schuermann[1]; Henry Greve[1]; Haile Takele[1]; Christian Pochstein[1]; Abhijit Biswas[1]; Michael Frommberger[2]; Eckhard Quandt[2]; Rainer Podschun[3]; *Franz Faupel*[1]; [1]Kiel University; [2]Center of Advanced European Studies and Research; [3]Kiel University Hospital

Recently, there is much interest in hybrid materials consisting of metal nanoparticles dispersed in a dielectric matrix due to their novel functional properties offering hosts of new applications. Polymers are particularly attractive as matrix. Consequently, various approaches have been reported to incorporate metal nanoparticles into polymers. The present talk is concerned with the preparation of polymer-based nanocomposites by vapor phase co- and tandem-deposition and the resulting functional properties. The techniques involve evaporation and sputtering, respectively, of metallic and organic components and inter alia allow the preparation of composites which contain alloy clusters of well defined composition. Emphasis will be placed on soft-magnetic and optical composites, but antibacterial coatings will also be addressed. In particular, a novel approach to produce magnetic nanorods for potential applications in high-density data storage and other fields will be presented.

4:30 PM
TEM and APT Investigations of Nanocrystalline MRE-Fe-B (MRE = Y, Nd, Dy) Hard Magnets with TiC Additions: *Y. Q. Wu*[1]; W. Tang[1]; M. K. Miller[2]; Iver E. Anderson[1]; R. W. McCallum[1]; K. W. Dennis[1]; M. J. Kramer[1]; [1]Iowa State University; [2]Oak Ridge National Laboratory

Nanocrystalline hard magnets with multi-rare earth (MRE) elements are of interest for their high temperature performance (>200°C). A $(MRE_2Fe_{14}B)_{1-x}+(TiC)_x$ (MRE=Y, Nd, Dy) base alloy with TiC additions (mole fraction, x=0.00-0.03) was investigated using transmission electron microscopy (TEM) and atom probe tomography (APT) to ascertain whether the partitioning of the various RE occurs during solidification, the segregation behavior of elements to the grain boundaries, and the effectiveness of the TiC grain refiner. The TiC additions significantly refine microstructure from an average grain size of >200nm (x=0.01) to ~20nm (x=0.03), to alter magnetic properties. APT investigations reveal the selective behavior of MRE and TiC in the alloys.This work was supported by DOE-EE-FCVT Program, Freedom Car Initiative, through Contract No. W-7405-ENG-82 at Ames Laboratory (USDOE). Research at the SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:50 PM Break

5:00 PM
Characterization of Soft Magnetic Nano-Material Deposited with M3D Technology: *Michael Carter*[1]; Jacob M. Colvin[1]; James W. Sears[1]; [1]South Dakota School of Mines and Technology

Direct Write Technologies are being utilized in antennas, engineered structures, sensors, and tissue engineering. One form of Direct Write Technology is Maskless Mesoscale Material Deposition (M3D). The M3D process is a Direct Write Technology that uses aerosol formation, transport and deposition. Inks for the M3D process utilize nano-particles in suspension for deposition. Soft magnetic material was formulated as an ink suspension, deposited and characterized. This paper will report on the results obtained after depositing the soft magnetic material. The results of the permeability are calculated from magnetic structures created with the deposition. These results are compared to conventional methods of soft magnetic material formation and construction.

5:20 PM
Nano Structured Garnet Films Suitable for Magneto Optic Devices: *Pragati Mukhopadhyay*[1]; [1]Indian Institute of Technology

The rapid development of the technologies of the communication and network has increased the need of high density recording for the storage of the information. Bismuth substituted rare earth iron garnet (Bi: RIG) films are potential materials for magneto-optic devices and efforts are being made to make films by various methods to enhance magneto-optic properties and make low cost viable devices. These types of films are mainly prepared by sputtering, laser ablation, liquid phase epitaxy etc. which can be expensive, need high growth temperature and time. By Sol-gel method nano-structured garnet films suitable for magneto optic devices can be prepared at very low costs and with ease to solve these limitations. We have made bismuth substituted rare earth iron garnet films by sol gel method and compare their preparations and characterizations with other rare earth substituted films grown by us by LPE method showing good magneto optic properties.

5:40 PM
The Role of Inversion Symmetry on the Magnetisation Reversal of Array of Ni Nanobars: *Prabeer Barpanda*[1]; Takeshi Kasama[2]; Rafal Dunin-Borkowski[2]; [1]Rutgers University; [2]University of Cambridge

Ni nanobars are quite promising for MRAM application, as they possess stable and higher coercivity owing to their shape anisotropy. Nanobars possess a special domain structure with an 'inversion symmetry' feature. Any magnetisation process essentially deals with movement and breaking of this symmetry. This high-energy process leads to higher coercivity. The current work investigates the effect of inversion symmetry (in Ni nanobars) on the overall magnetisation behaviour of an array of closely-spaced nanobars using 3-dimensional micromagnetic simulations. We studied arrays of Ni-nanobars (spaced 10 nm apart) of varying length (20~200 nm) and diameter (10~50 nm). The longitudinal magnetisation reversal revealed the formation of symmetric inversion state, which gradually formed a Neel wall at the central cross section of nanobars before switching to the other direction. This was true both for single domain and vortex state bearing Ni nanobars. The results were compared with corresponding electron holographs and analytical calculations.

3-Dimensional Materials Science: X-Ray Methods II/Quantitative Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Jeff P. Simmons, U.S. Air Force; Michael D. Uchic, Air Force Research Laboratory; Dorte Juul Jensen, Riso National Laboratory; David N. Seidman, Northwestern University; Anthony D. Rollett, Carnegie Mellon University

Tuesday PM — Room: 205
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Julie A. Christodoulou, U.S. Navy; Matthew Miller, Cornell University

2:00 PM
3DXRD Investigations of Recrystallization: *Dorte Juul Jensen*[1]; [1]Riso National Laboratory

By 3 Dimensional X-ray Diffraction (3DXRD) it is possible non-destructively to make complete 3D maps of the microstructure and to follow the kinetics in-situ while deforming and/or annealing the sample. 3DXRD results on recrystallization of cold rolled fcc materials are presented. It is shown that nuclei can be registered when they have a size of about 1.4 mm, that nuclei can develop with crystallographic orientations different from the parent phase and that nucleation kinetics vary significantly from nucleus to nucleus. The growth kinetics can be monitored either by fast measurements of only the increase in volume or by slower measurements mapping the complete 3D grain shape evolution. Based on the map-type measurements growth rates are quantified and related to the crystallographic orientations of the growing grains and the surrounding deformed microstructures. Finally an outlook for the planned upgraded 3DXRD is presented.

2:20 PM
Effects of Laser Shock Peening (LSP) on the Microstructure and Residual Stress Distributions in Titanium Alloys: *Yixiang Zhao*[1]; Seetha R. Mannava[1]; Todd J. Rockstroh[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati; [2]GE Aircraft Engines

The present study was undertaken to develop a fundamental understanding of the effects of Laser shock peening parameters on the residual stress distributions and microstructural changes in Ti-6Al-4V alloy. Coupons of the alloy with and without a sacrificial/ablative layer were LSP-treated with varying laser energy density using the GEN IV system at GEAE. In addition Ti-6Al-4V fan blades that had been LSP-treated were also studied. Depth-resolved characterization of the macro residual strains, stresses and degree of cold work in the peening direction and transverse to it was achieved using high-energy synchrotron x-ray diffraction at the Advanced Photon Source. The near-surface and through-the-depth changes in strain, texture and microstructure were also studied using EBSD in an SEM and by TEM, whereas local property changes were examined using micro- and nano-indentation measurements. The results showing the relationship between LSP processing parameters, the microstructure and residual stress distribution in the Ti alloy samples.

2:40 PM
Effects of Laser Shock Peening (LSP) on the Microstructure and Residual Stress Distributions in IN718 Superalloy: *Amrinder Singh Gill*[1]; Vijay K. Vasudevan[1]; S. R. Mannava[1]; [1]University of Cincinnati

LSP enhances service lifetimes of critical metal parts like aircraft engine fans and compressor blades. LSP dramatically improves fatigue strength, life and crack propagation resistance with shock wave-induced generation of deep compressive residual stress and microstructural changes. This study aims to understand effects of LSP parameters on residual stress distributions and microstructural changes in an important aero-engine material, IN718. Coupons of alloy with and without sacrificial layer were LSP-treated with varying energy densities using the GENIV system at GE Aircraft Engines. Depth-resolved characterization of macro residual strains and stresses and degree of cold work in peening direction and transverse to it was achieved using high-energy synchrotron x-ray diffraction at the Advanced Photon Source. Property changes were also studied using EBSD in SEM and TEM. Local property changes were examined using micro and nano-indentation measurements. Results show dominant effects of sacrificial layer and energy density on residual stress distributions and microstructure.

3:00 PM Invited
Topology of 3-D Grain Structures: *Burton R. Patterson*[1]; Alan P. Sprague[1]; [1]University of Alabama

This presentation reviews several aspects of the topology of 3-D grain structures, such as the frequency distributions of the numbers of faces on grains and the numbers of edges on grain faces. Much data has been obtained from separation of grains by liquid metal penetration as well as by serial sectioning. Also reviewed is the path of topological change that grains must traverse during grain growth and the different arrangements of faces experimentally observed and the relative affinities between faces of n and m sides. Frequency distributions and affinities from 2-D structures are compared.

3:25 PM
Microstructology of a Shocked Tantalum Plate: *John F. Bingert*[1]; Benjamin L. Henrie[1]; [1]Los Alamos National Laboratory

The nature of the three-dimensional damage characteristics in a polycrystalline metal are difficult to ascertain from two-dimensional sections. The heterogeneous nature of such a structure leads to uncertainties regarding the true tableau of voids and strain localization. The term 'microstructology' has been used to describe the stereological quantification of a microstructure in terms of its geometry, and its subsequent relationship to microstructural evolution. This approach was applied to understand the correlation between the underlying microstructure and the evolution of damage within a shocked tantalum plate. A three-dimensionally reconstructed microstructure measured from serially sectioned images was used to explore the geometric state of the material. In particular, the relationship between the formation and topology of the cavitation damage and the initial microstructural defect network are investigated at the mesoscale. The evolution of shear localization with respect to the cavitation network is also considered with the aid of the reconstruction.

3:45 PM

Determination of the Lattice Strain Tensor as a Function of Orientation from Three Dimensional Diffraction Data: *Joel Bernier*[1]; Matthew Miller[1]; [1]Cornell University

One of the manifestations of anisotropy in polycrystalline materials is the non-homogenous partitioning of elastic strains over the aggregate. The distributions of these strains have a strong functional dependence on crystallite orientation. In this talk, a versatile and robust method for determining a Lattice Strain Distribution Function (LSDF) from Strain Pole Figure (SPF) data is presented in the context of in situ loading/diffraction experiments. The LSDF is basically the mean lattice strain tensor as a function of crystal orientation. The determination of the LSDF relies on solving an inverse problem using optimization. Inter-granular stresses may be recovered from the LSDF via a constitutive relation, such as anisotropic linear elasticity. With this information in hand, orientations in the aggregate that are susceptible to localization or void growth may be identified. Additionally, knowledge of the evolution of these stresses and strains with deformation provides a robust tool for validation of polycrystal simulations.

4:05 PM Break

4:25 PM

Reconstruction and Characterization of 3D Microstructures: An Unbiased Description of Grain Morphology: *Michael Groeber*[1]; Michael Uchic[2]; Dennis Dimiduk[2]; Yash Bhandari[1]; Somnath Ghosh[1]; [1]Ohio State University; [2]Air Force Research Laboratory

The Dual Beam Focused Ion Beam-Scanning Electron Microscope (DB FIB-SEM) has proved to be a powerful instrument for collecting crystallographic data at a sub-micron level in 3D. The crystallographic data collected by the DB FIB-SEM system can be processed by customized codes in both 2D (Micro-Imager) and 3D (Micro-Imager3D). Micro-Imager is capable of automatically delineating grain boundaries and calculating characterization parameters for the segmented grain structure. Micro-Imager3D can be used to reconstruct the 2D orientation maps obtained during the serial-sectioning experiment into a true 3D microstructure. Once the data is reconstructed a host of statistical descriptions of the 3D structure can be made and compared to their 2D counterparts coupled with conventional stereological methods. These statistical descriptions are anticipated to be especially useful in accurately representing and model grain-level microstructures. This talk will briefly review the serial-sectioning methodology, and will focus primarily on the statistical description and representation of microstructure.

4:45 PM

The 3-Dimensional Characterization and Digitization of Complex Microstructures over Various Length Scales: *Robert Williams*[1]; Santhosh Koduri[1]; Peter C. Collins[1]; Gopal B. Viswanathan[1]; Hamish Fraser[1]; [1]Ohio State University

It is necessary to fully characterize materials for inclusion in accurate neural network and fuzzy logic models relating microstructure-property relationships. Previous methods of characterizing 2-D representations of the microstructures using rigorous stereological procedures have been useful, but new techniques to characterize the microstructure in 3-dimensions are expected to yield more accurate representations of microstructure. Additionally, it is required to characterize complex microstructures at a range of length scales, from meso-scale structures (e.g., grain size) to nano-scale features (e.g., secondary a in Ti-based alloys). Titanium alloys such as Ti-6-2-4-6, an a/ß alloy, offer microstructures rich in a variety of microstructural features, and this alloy has been heat-treated to produce a variety of these features. Three different 3-dimensional characterization techniques, each optimized for a specific length scale, will be shown, and the results will be discussed as they relate to the digitization of the microstructure.

7th Global Innovations Symposium: Trends in Materials R&D for Sensor Manufacturing Technologies: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Global Innovations Committee

Program Organizers: Hamish L. Fraser, Ohio State University; Iver E. Anderson, Iowa State University; John E. Smugeresky, Sandia National Laboratories

Tuesday PM	Room: 204A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chair: John E. Smugeresky, Sandia National Laboratories

2:00 PM

On True Sedimentation in Liquid Phase Sintering: Lei Xu[1]; Shu Zu Lu[1]; Thomas H. Courtney[1]; *Jong K. Lee*[1]; [1]Michigan Technological University

When a binary alloy such as Ni-W is liquid phase sintered, heavy solid W particles sedimentate to the bottom of the container, provided that their volume fraction is less than a critical value. The sintering process evolves typically in two stages, diffusion-driven macrosegregation sedimentation followed by "true" sedimentation. During sedimentation, the overall solid volume fraction decreases concurrently with elimination of liquid concentration gradient. However, in the second stage of true sedimentation, the average solid volume fraction in the mushy zone increases with time, and oddly, no concentration gradient is necessary in the liquid zone. For the mechanism, there are several possibilities such as solid state sintering, preferential coarsening, or packing rearrangement of the solid particles. In this presentation, we will first present the experimental results of three alloy systems, Cu-Fe, Ni-W, and Pb-Sn, and then discuss theoretical analyses including a modelling work for understanding the driving force for true sedimentation.

2:25 PM

Master Decomposition Curve for Binders in PM Processing: Gaurav Aggarwal[1]; Seong Jin Park[1]; *Ivi Smid*[1]; Randall M. German[1]; [1]Pennsylvania State University

Thermal debinding is one of the crucial steps in powder processing. In order to systematically analyze and design the thermal debinding step, the master decomposition curve (MDC) has been formulated and constructed based on intrinsic kinetics of organic pyrolysis. The Kissinger method is used to estimate the activation energy from TGA experiments. Overall thermal decomposition was synthesized from MDCs of individual components of binder systems with experimentally good agreement, which can help process designers to change the composition without additional experiments and can predict the remaining amount of each binder component during the debinding process. The input data are obtained from industrial debinding practice. In addition, the catalytic effect of metal powders has been investigated in terms of different powders, shapes and sizes. When extrapolating to very small particle size, this approach is of particular interest for predicting the behavior of nano-particulates.

2:50 PM

Characterization of Engineering Materials Utilizing Thermoelectric Power Measurements: *Joshua E. Jackson*[1]; Angelique N. Lasseigne[1]; David LeRoy Olson[1]; Brajendra Mishra[1]; Victor I. Kaydanov[1]; [1]Colorado School of Mines

Advanced thermoelectric power sensors have been developed for numerous applications to guarantee material integrity by providing a non-destructive electronic property correlation to material microstructure, phase stability, specific solute addition, lattice strain, and assessment during material processing will be discussed. These properties can be correlated to the thermoelectric power coefficient using classical electron model descriptions of the electronic state of the alloy. How the electron concentration, the effective mass, and the dominating scattering mechanisms allow for non-destructive evaluation of materials will be described. Because thermoelectric power is dependent upon numerous variables, additional non-destructive techniques are necessary to further characterize or classify the material. The use of additional collaborative NDE technologies will be described.

3:15 PM Break

3:35 PM
Overview of the Use of Direct Write Technologies for Use in Sensor Developments and Fabrication: *James W. Sears*[1]; [1]South Dakota School of Mines and Technology

Direct Write Technologies (DWT), like the ones developed under the DARPA MICE program, provide a tool for the novel manufacturing of various sensors (e.g. thermocouples and pressure transducers). DWT are also being used in electronics, engineered structures, and tissue engineering. Many for the precursors materials used as inks and pastes for the Direct Write technologies are comprised of nano-scale particles (i.e., Ag, Au, Pt). Various formulations are used to carry the nano-particles during the Direct Write processing. The Direct Write processes deliver these inks and pastes to a surface where they are then post processed to form functional devices. This presentation will provide an overview of these technologies and provide a examples of some of the sensors that have been fabricated.

4:00 PM Invited
Composite Magnetostrictive Materials for Advanced Automotive Magnetomechanical Sensors: *R. William McCallum*[1]; K. W. Dennis[1]; D. C. Jiles[1]; J. E. Snyder[1]; Y. H. Chen[1]; [1]Ames Laboratory

Co-ferrite and metal-bonded Co-ferrite composites have been investigated by the authors as an alternative to existing materials competing for use in automotive and other torque sensing applications. The materials are inexpensive and non-corroding, and the metal-bonded Co-ferrite composites are mechanically robust and can be attached (e.g. by brazing) to metal parts. They show a steep slope of magnetostriction at low applied fields, $(d\lambda/dH)\sigma$, which contributes to a high sensitivity of magnetic induction to stress, hence giving high signal-to-background noise ratios in sensor applications.

Advanced Materials for Energy Conversion III: A Symposium in Honor of Drs. Gary Sandrock, Louis Schlapbach, and Seijirau Suda: Metal Hydrides II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada; John J. Petrovic, Petrovic and Associates; Renato G. Bautista, University of Nevada; M. Ashraf Imam, Naval Research Laboratory

Tuesday PM	Room: 214B
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: James Wang, Sandia National Laboratory; John J. Petrovic, Petrovic and Associates; M. Ashraf Imam, Naval Research Laboratory

2:00 PM Invited
Defects Formation in $Lani_5$ and Its Related Alloys with Hydrogenation and Dehydrogenation: *Etsuo Akiba*[1]; Kouji Sakaki[1]; Yumiko Nakamura[1]; [1]AIST

With hydrogenation and dehydrogenation, lattice of hydrogen absorbing alloys expands and contracts in 20 to 30% in volume. Defects are expected to be introduced into the lattice to relax the strain energy that is generated by volume expansion and contraction. We have studied defect formation into the lattice of $LaNi_5$ and related alloys using in-situ diffraction and positron life time measurements. In the lattice of $LaNi_5$, a large amount of defects such as dislocations and vacancies was introduced at the first hydrogenation. Once defects were introduced, they were not relaxed at the working temperature of the alloys. Using diffraction method, we measured the size of crystallite before and after hydrogenation. The crystallite size did not changed with hydrogenation in all the alloy studied; $LaNi_5$ and $LaNi_{5-x}M_x$ (M=Al, Sn). Volume expansion and contraction can be accepted by introducing defects in the lattice without reducing the crystallite size.

2:25 PM Invited
Kinetics of Hydrogen Absorption and Desorption in Magnesium: Role of the Structure and of Catalysts: *Antonio Miotello*[1]; [1]Università di Trento

Among the light metals forming hydride phase, magnesium is one of the most interesting for application in hydrogen storage technology because of the very high capacity, close to 7.6 wt. % : unfortunately, the very slow kinetics in hydrogenation and dehydrogenation reactions requires the addition of a proper catalyst even at temperatures as large as 673 K. The catalyst, typically in form of metallic nanoparticles (Nb, PdFe3, Pd) dispersed at the Mg surface, favours the H2 dissociation and the jump of H atoms towards the subsurface layers of the Mg matrix by the formation of the metastable Nb-H phases or by the transfer of H atoms to Mg after the H migration through the Mg-Nb interface. MgD2 film samples with microcrystalline structure containing 5 at. % Nb as metallic additive (prepared by r.f. magnetron sputtering) show an improved D2 desorption kinetics compared to the pure MgD2 film samples, as evidenced by thermal desorption spectroscopy (TDS) analysis: the D2 desorption spectra of the Nb doped deuteride show a maximum of the D desorption rate at 440 K compared to a maximum at 630 K for the pure MgD2 sample. The evaluated values of the effective activation energies for desorption were 141 ± 5 and 51 ± 5 kJ mol-1H for the pure and the Nb- doped hydride, respectively. It is relevant to note that the activation energy for H diffusion in a-Mg is 40 kJ mol-1. To understand the role of catalyst in enhancing kinetics, Nb K-edge Extended X-ray Absorption Fine Structure spectroscopy (EXAFS), X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM) were used to analyse the Nb coordination and phase formation in Nb-doped (5 at. %) h-Mg film samples. Results show that the catalytic effect of the Nb doping in the H2 absorption and desorption kinetics is connected with the formation of Nb nano-clusters dispersed in the host matrix. In particular, while the Nb atoms are dispersed into the Mg matrix upon sputtering deposition, H-containing Nb nanoclusters (-NbH0.89) are present in the film upon H2 absorption, while Nb single metal clusters remain upon the H2 desorption process. From the above results, we may conclude that Nb clusters act as efficient catalysts, through a number of mechanisms which include: i) a reduced stability of the hydride due to elastic stresses produced by the -NbH0.89 nanoclusters distributed in the matrix, ii) the presence of Mg-Nb interfaces where the transfer of H atoms preferentially occurs. This last mechanism is justified on the basis of the activation energy for diffusion, as well as the kinetics order, being ˜1. On the contrary, when clusters are not formed, kinetics is low and of order ˜ 4. The different kinetics orders are discussed in terms of structure and of dissociation mechanisms.

2:50 PM Invited
Storing Hydrogen with Metal Perhydrides: *Jiann-Yang James Hwang*[1]; Shangzhao Shi[1]; Bowen Li[1]; Xiang Sun[1]; [1]Michigan Technological University

Research on hydrogen storage materials has been pursued for many years, and has proved to be a tough task. The key problem is perhaps that most of the candidate materials are much heavier than carbon but their capabilities to bind hydrogen (in atomic ratios) are lower. In addition, the host materials cannot contribute to the energy content. It becomes more and more apparent that the hydrogen storage materials so far developed cannot meet their application goals. In recent years, the U.S. Department of Energy sponsored a number of projects exploring new materials and concepts for hydrogen storage. This article briefs a project being currently pursued at Michigan Technological University. The project is exploring a novel kind of materials that contain hydrogen clusters in their molecules. The novel materials are termed as metal perhydrides, which are anticipated to have sufficient content of hydrogen and are promising for high-efficiency hydrogen storage.

3:10 PM
Suppression of the Martensitic Transformation in Ti-Ni-Cu Alloys with Shape Memory Properties: Specific Impact of Hydrogenation: *Nataliya Skryabina*[1]; Daniel Fruchart[2]; Aleksandr Shelyakov[3]; Dmitrii

Gunderov[4]; [1]Perm State University; [2]Centre National de la Recherche Scientifique; [3]Moscow Physical-Technical Engineering Institute; [4]Institute of Physics of Advanced Materials

Two possibilities were known to suppress the thermo-elastic martensite phase transformation in alloys based on the Ti-Ni-Cu system. The first one route can operate via a refinement of the grain size up to 10 to 15 nm from fast quenching treatments from the melt. The second route results from severe plastic deformation (SPD) or high pressure torsion (HPT) methods developed at room temperature. We have discovered a new possibility to suppress the martensite B2-B19 transformation by hydrogenation of amorphous then nano-crystallised alloys. This results in a two step process of the grain growth promoted by hydrogenation and subsequent heat treatments of the nano-crystallized materials. The nucleation of the nano-grains at a first step of crystallization has made impossible the B2-B19 transformation due to a reduced size of the grains. In this contribution, we compare the merits of the different process as mentioned here above.

3:30 PM
Investigations on Light Metal Hydrides at Padova, the City of San Antonio: *Gianni Principi*[1]; P. Palade[1]; S. Sartori[1]; A. Maddalena[1]; F. Agresti[1]; S. Lo Russo[1]; [1]Università di Padova

The activity of the "Hydrogen Group" of Padova University, addressed to the study of materials for solid state hydrogen storage, is illustrated. Various Mg-based materials have been considered and are being studied: a) MgH2 and 0.5% mol Nb2O5 mixture ball milled under argon atmosphere; b) Mg-Ni-Fe intermetallic compounds prepared by short time ball milling of ribbons obtained by melt spinning and by long time ball milling of a mixture of MgH2, Ni and Fe powders; c) MgH2 ball milled in argon with Ni-Al and Zr-Cr-Fe catalyst alloys. All the samples have been structurally characterized by X-ray diffraction (Rietveld refinement) before and after hydrogen absorption/desorption cycling and tested with a Sievert apparatus as regarding their thermodynamic and kinetic properties. Investigations are also in progress on complex hydrides as alanates and Li amides modified with transition metal based catalysts.

3:50 PM Break

4:05 PM Keynote
Is High Weight H Intake in Pseudobinaries Still an Option?: *Issac Jacob*[1]; [1]Ben Gurion University of Negev

Pseudobinary intermetallic compounds constituted the backbone of hydrogen storage research for tens of years. This is because they present versatile hydrogenation properties, and some of them form reversible hydrides under moderate hydrogen pressures near room temperature. However, a current limit of about 2% hydrogen weight causes a shift of the research interest to other hydrogen absorbers. We overview here the conditions and the factors, determining hydrogen absorption in intermetallic compounds. A special attention is given to recently found differentiation and divergence of elastic properties in intermetallics upon pseudobinary substitution. The possibility to form superior intermetallic compound for hydrogen storage is considered.

4:25 PM
Hydrogen Short-Long Range Dynamics in Nano-MgH2 a Mechanical Spectroscopy Approach: *Ennio Bonetti*[1]; Annalisa Fiorini[1]; Luca Pasquini[1]; Amelia Montone[2]; Marco Vittori Antisari[2]; [1]University of Bologna; [2]ENEA Centro Ricerche Casaccia

The technological applications of MgH2 hydrides for hydrogen storage demand the overcoming of some critical issues: an improvement of the reaction kinetics and a reduction of the desorption temperature. The synthesis of nanocrystalline MgH2 by reactive milling and inert gas condensation and proper catalysts addition, are recently employed strategies. In the investigation of the hydrogen short and long range dynamics, the sorption kinetics and the nanostructure stability, the mechanical spectroscopy offer some specific advantages. In the present research this technique was employed with other experimental approach XRD, TEM, to investigate relaxational and structural damping processes in nanocrystalline MgH2 with different added catalysts. Time temperature variation of the dynamic elasticity moduli and mechanical quality factor are correlated to hydrogen anelastic relaxation processes and concomitant structural transformation of MgH2 to Mg during long range diffusion. Results are discussed with reference to microstructural modification strategies able to assist hydrogen release.

4:45 PM
Characterization of Hydrogen Storage Capabilities of the Two-Phase Region of LaNi5: *Angelique N. Lasseigne*[1]; M. Ashraf Imam[2]; Brajendra Mishra[1]; David LeRoy Olson[1]; [1]Colorado School of Mines; [2]Naval Research Laboratory

Hydrogen storage capabilities of LaNi5 intermetallic compounds are investigated utilizing the combination of thermoelectric power, Beeghly Ester-Halogen digestion, and Leco hydrogen determination. Thermoelectric power has demonstrated a rapid hydrogen assessment capability and can achieve the equivalent of the pressure-composition-temperature (activity) diagram. Effective use of hydrogen storage materials occurs in the (alpha+beta)-phase plateau region of the PCT diagram. A thorough assessment of the content of each phase in this two-phase region to optimize performance of the hydrogen storage materials will be described. Practices using the combination of these three analytical practices for rapid characterization of LaNi5 intermetallic alloys will be discussed.

5:05 PM
Improving a Rechargeable Hydrogen Storage Capacity of BCC Alloy by Eliminating Internal Defects: *Toshiki Kabutomori*[1]; Kazuya Kubo[1]; Hironobu Arashima[1]; Hideaki Itoh[1]; Takanori Suda[2]; Somei Ohnuki[2]; Keizo Onisi[1]; [1]Japan Steel Works, Ltd.; [2]Hokkaido University

Although a Ti-Cr-V hydrogen storage alloy having a BCC structure can absorb about 4wt% hydrogen, a rechargeable hydrogen storage capacity is only about 2.6 wt% because the alloy has a wide hydrogen solid solution. On the other hand, many internal defects are introduced by the volume expansion (> 40 %) at the hydriding of the Ti-Cr-V BCC alloy. Due to hydrogen trapping to the defects, it is expected that hydrogen solid solution increase, resulting to decrease a plateau width in the P-C-T diagram. In this study, we examined an influence of the internal defects produced by hydriding affects the P-C-T characteristic of a Ti24Cr36V40 BCC alloy. After annealing at 1,000°C for 10 min, a Ti-Cr-V BCC alloy shows increase in the plateau width from 2.4 to 3.3wt%. Accordingly, it is thought that a rechargeable hydrogen capacity of the BCC alloy can be largely improved by eliminating the internal defects.

5:25 PM
Low Pressure Hydriding of V-0.5 at%C Hydrides: *Joshua H. Lamb*[1]; Dhanesh Chandra[1]; Michael Coleman[1]; Joseph R. Wermer[1]; Stephen N. Paglieri[1]; [1]University of Nevada

The properties of vanadium (V) hydride formation at low pressures have significance in hydrogen transport membranes. Low pressure hydriding characteristics of V-0.5 at% C alloy using gas phase hydrogen has been performed in this study. There are few prior reports on the stability and low pressure isotherms of (pure) vanadium 1 phase pure V and some with alloying elements (transition elements), but no reports on V-C alloys. The V-0.5%C has many advantages from strengthening of the alloy as well as structural integrity of the membranes. In our study, van't Hoff plots were plotted and enthalpies of the low pressure plateau region of the V-0.5%C and pure V are compared.

Alumina and Bauxite: Bauxite and Bauxite Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Jean Doucet, Alcan Inc; Dag Olsen, Hydro Aluminium Primary Metals; Travis J. Galloway, Century Aluminum Company

Tuesday PM — Room: 7B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Milind V. Chaubal, Sherwin Alumina Company

2:00 PM Introductory Comments

2:10 PM
Cross Country Bauxite Slurry Pipeline Transportation: *Ramesh L. Gandhi*[1]; Yueguang Che[1]; Jay Norwood[1]; [1]Pipeline Systems Incorporated

Over the last four decades, slurry pipelines have proven themselves to be a safe, reliable and cost effective way to transport large tonnages of minerals over long distances. A variety of minerals have been successfully transported over distances ranging from a few kilometers to 400 km. However, no long distance bauxite pipelines have been built to date. Bauxite has developed a reputation for being difficult to pump. This is about to change with the first long distance slurry pipeline. The 250 km long 10 MT/y capacity pipeline is designed by PSI. Several other pipelines are currently being considered for transportation of bauxite. It will be shown that a properly designed hydro-transport system is an economically viable alternative for the long distance transport of bauxite ore. The focus of this paper is on how to integrate a pipeline into an existing facility.

2:35 PM
The Future of Alumina/Bauxite Mining in Guinea: *Koulibaly Siafa*[1]; [1]Ministry of Geology and Mines

Guinea is one of the richest countries in Africa in view of its bauxite mining and energy potential. It has nearly one-third of the world's bauxite reserves and it is the second world producer of this mineral, coming after Australia. The mining sector plays a key role in the Guinean economy. The state will continue to depend highly on this sector's fiscal revenue and foreign exchange for the coming years at least. Guinea is currently facing serious economic problems. It is therefore of prime importance that government should adopt effective policies with regard to management and making viable regulation to govern the alumina/bauxite industry, as well as acquire strong capacity to implement them. This paper will survey the basic parameters such as resources and competitiveness, Guinea's prospective in terms of efficiency of technology, and environmental considerations. The article will emphasize Guinea's bauxite position in the market, increasing production, and opening new alumina plants.

3:00 PM
Occurrence and Characterization of Zn and Mn in Bauxite: *Frank Roman Feret*[1]; Jeannette See[1]; [1]Alcan Inc

Zinc is one of the secondary elemental constituents occurring in Caribbean bauxite and the associated non-bauxitic material. It is generally believed that Zn in bauxite could either occur in gahnite or sphalerite, or substitute for Fe in goethite. Manganese represents an appreciable impurity in Caribbean bauxites and is identified on diffractograms as lithiophorite (Li,Al)MnO2(OH)2. Because Zn was observed to increase with the MnO content the objective of this work was to better understand the mineralogical nature of Zn and Mn compounds. Data representative of 340 bauxite samples of different origin was assembled. It was found that Zn in bauxite cannot substitute for Fe in goethite or hematite. Strong evidence was obtained that Zn occurs in the same compound as Mn. The application of the Rietveld-XRD method to the quantification and characterization of a new mineral called zincophorite Al(ZnxMn1-x)O2(OH)2 is discussed. Caustic solubility of ZnO in bauxite is also assessed.

3:25 PM Break

3:45 PM
Beneficiation of High Quartz Content Bauxite from Los Pijiguaos: *Jean-Marc Rousseaux*[1]; Perdo Flores[2]; Stefan Buntenbach[3]; Hans Verschuur[1]; [1]Alcan; [2]CVG Bauxilum; [3]AKW Apparate und Verfahren

At Los Pijiguaos, the economic bauxite horizon attains an average thickness of 7.6 m. Below this layer, High Quartz Content bauxite can be found. The aim of this study was to investigate the possibility to extract quartz from High Quartz Content bauxite of LP, with a minimum of Al2O3 losses. The beneficiation process studied in this work is carried out in several steps. The first step is an elutriation, operated in a washing drum, aiming at liberating quartz particles. The second step is a classification process, aiming at obtaining different grain sizes according to the quartz content. Part of the study was to investigate if the finest fractions could further be beneficiated using such technologies as cyclones and shaking tables. This paper describes tests carried out and results obtained. Characteristics of beneficiated bauxite and beneficiation recovery are also detailed. Conclusions and perspectives delineate pilot test strategies.

4:10 PM
Thin Layer Chromatography of Bauxite: Instrumental Characterization and Quantification by Scanning Densitometry: *Mohamed Najar*[1]; Ramana Rao Kondapalli[1]; [1]Jawaharlala Nehru Aluminium Research Development and Design Centre

Thin layer chromatography (TLC) was successfully used for the detection and characterisation of various elements present in bauxites. The chromatographic characteristics have been evaluated on silica gel and cellulose layers developed with double distilled water and aqueous sodium chloride solutions. The densitometric evaluations of chromatograms of Al, Fe, Ti and Si in bauxites have shown a percentage recovery of 98 ±2. The method developed was successfully extended to the characterisation of organic impurities (<0.2%) in bauxites by coupling TLC with FTIR spectroscopy. The characteristic functional groups for some organic compounds were identified in the IR spectra. The advantages of these methods over the conventional methods of characterisation were also presented.

Aluminum Reduction Technology: Cell Development and Operations - Part II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Stephen Joseph Lindsay, Alcoa Inc; Tor Bjarne Pedersen, Elkem Aluminium ANS; Travis J. Galloway, Century Aluminum Company

Tuesday PM — Room: 7A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Yousuf Ali Mohammed Alfarsi, Dubal Aluminium Company Ltd

2:00 PM
Paradox in Cell Temperature Measurement Using Type K Thermocouples: *Xiangwen Wang*[1]; Gary P. Tarcy[1]; Mike Slaugenhaupt[1]; Susanne L. Albright[1]; [1]Alcoa Inc

Type K (Chromel-Alumel) thermocouples or temperature probes are widely used in routinely measuring cell temperature because they are readily available and economical while offering the kind of accuracy and precision for smelter operations. However, the popular usage does not mean there are no issues. More often than not, we, as an end user of the thermocouples, are not aware of the existence of several alloys available in the market in making the Type K thermocouples. Accordingly, some confusions or ignorance in selecting proper thermocouples and their proper accessories causes inaccurate temperature measurements or temperature measurement bias. Sometimes, a temperature bias as high as 10C may be observed. This paper discusses our smelter operating experience in dealing with the temperature bias issues in using Type K thermocouples and probes. Selection from various suppliers and proper use of the Type K thermocouples are discussed.

2:25 PM
The Liquidus Enigma Revisited: How it Could Be Solved: *Bjørn P. Moxnes*[1]; Asbjørn Solheim[2]; Trond Støre[1]; Bjørn Erik Aga[1]; Lisbet Støen[2]; [1]Hydro Aluminium; [2]SINTEF

The superheat calculated from bath analyses often show negative values, although measurements made with the superheat-sensor from Heraeus Electro-Nite show positive values. Aiming at coming up with an explanation, the bath temperature and superheat were measured in different cells at the Hydro Aluminium Sunndal plant and at the Research Centre in Årdal. Simultaneously, bath samples were taken for Al4C3 analysis, XRF, XRD and ICP analyses, as well as alumina analysis on LECO and thermal analysis. It turned out that Al4C3 content was very low. By comparing all measured temperatures, it could be concluded that the Heraeus probe in some cases gave 4-5°C too high superheat. The key factor, however, appeared to be the precision of the standard bath analysis. Furthermore, it is likely that the some of the trace elements in the bath lower the liquidus temperature more in an acidic bath than in pure cryolite.

2:50 PM
Rate of Metal Cooling in Aluminum Reduction Cell Removed from Line Current – Method and Model: *Kayron F. Lalonde*[1]; H. Wayne Cotten; Richard M. Beeler; [1]Alcoa Inc

A method was established for obtaining temperatures of molten aluminum as it solidifies in a reduction cell after the cell has been removed from line current (a "dead" cell). The temperatures were used to develop cooling curves, so the time at which the metal pad solidifies can be estimated and demolition of the lining can begin. The effect on the cooling rate of removing all the anodes in the cell at 24 hours was assessed. Mathematical modeling including the heat of fusion of aluminum was developed to explain the effect of anode removal and to provide predictive capability based on several input variables.

3:15 PM
Method for Automated Adjustment of Alumina Feeding Times in Smelting Pots: Constantin Radulescu[1]; Puiu Chirimbu[1]; *Cristian T. Stanescu*[1]; Mihail Atanasiu[1]; Ioan Cojocaru[1]; Gheorghe C. Dobra[1]; [1]ALRO SA

This paper presents a method for automated adjustment of alumina feeding times in smelting pots with a continuous control of alumina concentration in the bath. The method performs a recalculation, each 4 - 8 hours, of the feeding frequency for theoretical, over and underfeeding in order to keep an alumina concentration in the bath in the range of 1.6 - 2.2%. The basis for recalculation is the connection of the main pot parameters with the theoretical feeding frequency. This allows keeping optimal condition for alumina dissolution and counteracts the time variation of the weight of alumina dozes. Moreover this method limits the sludge accumulation on the pot bottom and prevents the occurrence of anode effects resulting from temporary failures of equipment.

3:40 PM Break

3:50 PM
CVG Venalum Potline Supervisory System: *Carlos Abaffy*[1]; Jesús Lárez[1]; Rafael Aiquel[1]; Jesús González[1]; [1]CVG Venalum

This paper describes architecture and features of CVG-Venalum aluminum reduction Cell Supervisory System (SSC) which, besides the standard characteristics of a supervisory system (real-time parameters display, historic trends, operation tracking, etc.), allows effective (easy interpretation), accurate (allows quantitative evaluation) and adaptable (serves multiple needs) data visualization and analysis of potlines. The SSC has an outstanding performance compared with other supervisory systems. It's been installed in 4 CVG-Venalum potlines of 180 cells each, where a desktop PC serves each line with no impact on performance. In addition, the potlines have been integrated in a high-level system for visualize the whole plant. The SSC is part of the V-350 cell technology and it will be used in CVG-Venalum and CVG-Alcasa expansion plans. The system was developed using free software platform: LAMP (Linux-Apache-MySql-Php) and C++. The Human-Computer Interface is mainly web, allowing the user interact easily with the system using a web browser.

4:15 PM
300 KA Pre-Baked Cells Start-Up at the "Sual" JSC: *Viatcheslav Veselkov*[1]; Yury Bogdanov[1]; Boris Ayushin[1]; [1]Sibvami JSC

In May 2005 the Test Site of six 300 KA (design amperage) pre-baked cells was put in operation at the Ural Aluminium smelter. All calculations and Test Cell Project was developed by "SibVAMI" JSC, Irkutsk. The Site consists of the Reduction Pot Room, "Dry" Scrubber Unit, and injecting Silicon Rectifier. Power for the Test Site is supplied from two sources: the existing Pot Line (165 KA) and the injecting SR (135 KA). The cell shows highly stable behavior, MGD-noise in the cell is under 15-20 mV. Test cell bottom firing was carried out using gas-flame unit for 72 hours. The competitive up-to-date electrolyzer was developed. These cells are intended for operation at 330 – 350 KA.

4:40 PM
Investigation of the Failure of a 300kA Prebaked Anode Reduction Cell: *Zhongning Shi*[1]; Bijun Ren[1]; Hongtao Zhang[2]; Quanhong Cao[2]; Tiejun Wen[2]; Hui Li[2]; Zhuxian Qiu[1]; [1]Northeastern University; [2]Yichuan Aluminum Smelter Plant

Because of the failure of side lining and cathode, some 300kA prebaked anode aluminum reduction cells were shut down in Yichuan aluminum smelter plant. Combining with actual observations, the samples of side lining and cathode were analyzed from different locations of the cell, and the failure reasons of the Si3N4-SiC side lining and cathode were investigated. Also, the influencing factors of cathode and side lining failure were summarized as superheat temperature, anode effect coefficient, interval from anode edge to the sidewall, anodic current density and the operating parameters. Some suggests are offered to solve these problems occurring in large scale prebaked anode reduction cell in China.

5:05 PM
Production of Refined Aluminum and High-Purity Aluminum: *Huimin Lu*[1]; Yongheng Wang[1]; Zongren Liu[2]; Xinsheng Zhai[2]; Jie Liu[2]; Tao Hong[2]; [1]University of Science and Technology Beijing; [2]Xinjiang Joinworld Company, Ltd.

Xinjiang Join-world Co. Ltd. is the largest producer in China and an important one in the world for refined aluminum and high-purity aluminum. At present, 99.99~99.999% refined aluminum is produced by 80kA three-layer electrolysis cells (XJ-80) with the Gadeau process electrolyte system and solid refined aluminum cathode, the current efficiency is up to 99%, DC power consumption 14000kWh/t refined aluminum, the XJ-80 cell is also the largest one in aluminum refining industry in the world; 99.999~99.9999% high-purity aluminum is yielded by the combined technique of three-layer electrolysis and segregation method; high-purity aluminum in excess of 99.9999% is produced by the new combined technique of ionic liquid electrolysis and zone refining method. This paper introduces these characteristics of the XJ-80 three-layer electrolysis cell, the combined technique of three-layer electrolysis and segregation method and the new combined technique of ionic liquid electrolysis and zone refining method for producing high-purity aluminum.

5:30 PM End

Amiya Mukherjee Symposium on Processing and Mechanical Response of Engineering Materials: Processing of Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
Program Organizers: Judy Schneider, Mississippi State University; Rajiv S. Mishra, University of Missouri; Yuntian T. Zhu, Los Alamos National Laboratory; Khaled B. Morsi, San Diego State University; Viola L. Acoff, University of Alabama; Eric M. Taleff, University of Texas; Thomas R. Bieler, Michigan State University

Tuesday PM — Room: 217C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Donald R. Lesuer, Lawrence Livermore National Laboratory; George C. Kaschner, Los Alamos National Laboratory

2:00 PM Invited
A Microtexture Investigation of Recrystallization during Friction Stir Processing of As-Cast NiAl Bronze: Keiichiro Oishi[1]; Alexander P. Zhilyaev[1]; *Terry R. McNelley*[1]; [1]Naval Postgraduate School

OIM and TEM methods were used to obtain microtexture data in an NiAl Bronze subjected to FSP. Random textures, grains 1 – 2 μm in size and annealing twins indicated recrystallization in the a phase inside the stir zone; subgrains near the plate surface apparently formed after passage of the tool. Distinct shear textures and texture gradients were apparent in the thermomecanically affected zone (TMAZ) outside and along the periphery of the stir zone. The TMAZ texture gradient was steeper on the advancing side and under the stir zone center than on the retreating side. Grain refinement in the a phase reflects dynamic recrystallization and particle stimulated nucleation at undissolved particles during FSP. Random stir zone textures reflect recrystallization textures for materials experiencing warm deformation in shear during FSP as well as contributions from PSN and the twin chaining.

2:20 PM

Aspects of Thermomechanical Processing Controlling the Austenite Transformation in Si-Mn TRIP Steel: *Jozef Zrnik*[1]; Ondrej Stejskal[2]; Zbysek Novy[1]; Peter Hornak[3]; [1]COMTES FHT; [2]West Bohemian University; [3]Technical Univesity of Kosice

The main emphasis of this study has been placed on thermomechanical processing simulations of TRIP steel performed by using forging. The choice of applied strain and thermal parameters modified austenite conditioning and consequently affected its transformation kinetics and resulted in variety of final structure characteristics in time of two step transformation process. The development of multiphase structure consisted of ferrit, bainite and retained austenite had strong impact on the transformation induced plasticity effect of steel. The complex relationship among the volume fraction of the retained austenite, the morphology and distribution of phases, and mechanical properties of TRIP steel was revealed. The tensile tests were conducted and due to TRIP effect functionality good combination of tensile strength and ductility was found. The results proved that TM processing conducted in intercritical temperature region significantly effected forming and refinement of convenient multiphase structure, providing sufficient ductility at increased strength of bulky forgings.

2:40 PM

Design of High-Strength Steels by Microalloying and Thermomechanical Treatment: *Patricia Romano*[1]; Araz Ardehali Barani[1]; Dirk Ponge[1]; Dierk Raabe[1]; [1]Max Planck Institute for Iron Research

Steels with higher strength, ductility and improved fatigue behaviour are required for light-weight structures in the automotive industry. It is shown that for ordinary steels the combination of microalloying and an optimized thermomechanical treatment results in increased strength and improved ductility. Proper conditioning of the austenite by a sequence of deformation steps refines the austenitic grains and generates a dislocation substructure that is inherited to the martensite structure. In contrast to simply quenched and tempered martensite with no prior deformation, the thermomechanically processed martensite exhibits a more refined structure and is free of grain boundary carbides. Addition of vanadium is beneficial for the stabilization of the austenite defect structures that are produced by deformation.In this study an increase of more than 600MPa in the ultimate tensile strength and an improvement of 40% in the reduction area are reported. A general thermomechanical treatment for high-strength martensitic steels is proposed.

3:00 PM

Development of the Surface Structure of TRIP Steels Prior to Hot-Dip Galvanizing: *Erika Bellhouse*[1]; Anne Mertens[1]; Joseph McDermid[1]; [1]McMaster University

Focusing on improving the galvanizability of TRIP steels, the effect of the alloying elements; manganese, silicon and aluminum on the surface state prior to galvanizing were studied. A C-Mn steel was used to determine the effect of manganese, and TRIP steels with varying amounts of Si and Al were used to determine the effect of the silicon and aluminum. Different dew points and reducing atmospheres were tested during annealing in a galvanizing simulator in order to improve the wettability of liquid zinc on the steel surface. This paper will present the kinetics and development of the surface structure prior to galvanizing as a function of annealing atmosphere, temperature and time.

3:20 PM

Effect of Annealing Atmosphere on Galvanizing Behavior of Dual Phase Steel: Rubaiyat Khondker[1]; *Anne Mertens*[1]; Joseph R. McDermid[1]; [1]McMaster University

Selective surface oxidation of alloying elements such as Mn can cause DP wettability problems by liquid Zn during continuous galvanizing. It is well known that process parameters, such as annealing atmosphere %H2 and dew point, can affect surface and subsurface oxidation. The purpose of the paper is to study the effect of annealing atmosphere to find the desired DP steel surface that could produce better wetting by zinc. In particular, the evolution of the surface phases and structures during the continuous galvanizing annealing cycle were studied. It will be shown that the internal/external oxidation behavior of the alloying elements of DP steel (e.g. Mn, Si, and Mo) at the surface and subsurface demonstrate that segregation can be controlled by changing process parameters (dew point and H2/N2 ratio) and that some segregation of elements is unavoidable but can produce good wetting by liquid galvanizing alloys.

3:40 PM

New Aqueous Metal Feedstock Materials for Injection Molding Process: *Mohammad Behi*[1]; Kamal Shahrabi[1]; Ahmad Shohadaee[1]; [1]Kean University

A new water-based stainless steel 17-4 feedstock material has been developed for the injection molding process. Fine stainless steel 17-4 powder (10-25 microns) is mixed with a binder system at about 85-90°C to make the feedstock. The binder comprises 98-99% de-ionized water and 1-2wt% polysaccharide binder. A conventional injection molding machine is used to mold the feedstock into articles of various shapes at temperatures between 80-90°C and injection pressures of 500-900 psi. The flowability of the feedstock material was evaluated at 500 and 1000 psi injection pressures. Unlike the polymer based metal feedstock, there is no thickness limitation and no debinding process associated with this new material upon sintering. The molded parts are sintered at 1300-1350°C in hydrogen atmosphere. The feedstock material is user friendly and environmentally safe.

4:00 PM Break

4:10 PM Invited

Nano-Subgrain Strengthening in Ball-Milled Iron: *Donald R. Lesuer*[1]; Chol Syn[1]; Oleg Sherby[2]; [1]Lawrence Livermore National Laboratory; [2]Stanford University

Recent studies of ball-milled iron have shown that nano-scale subgrains can form during the early stages of ball milling. In this paper we evaluate the strength and strengthening mechanisms of these ball-milled materials containing nano-subgrains. The results are compared with the strength and strengthening mechanisms resulting from larger-scale subgrains in iron and iron-base alloys produced by traditional mechanical working. The data covers over 2 orders of magnitude in subgrain size (from 30 nm to 6 µm). For all materials studied, at the larger grain sizes the strength varied as λ-1, where λ is the subgrain size. In addition, the ball-milled materials showed significant strengthening contributions from nano-scale oxide particles. The data shows deviation from the λ-1 relation and a breakdown in subgrain strengthening at about 150 nm. The results are interpreted in terms of changes in the dominant strengthening mechanism with subgrain size and oxide particle spacing.

4:30 PM

Deformation Mechanisms of Cryomilled Al Alloys with Bimodal Microstructure: *Bing Q. Han*[1]; David B. Witkin[1]; Riqing Ye[1]; Enrique J. Lavernia[1]; [1]University of California

Cryomilling is one of several synthesis techniques that is capable of producing engineering materials with grain sizes in the 10-500 nm ranges. In the present study, processing of several bimodal nanostructured Al alloys from cryomilled nanostructured powders is reviewed briefly, followed by a discussion of mechanical behavior and the underlying deformation mechanisms. Particular emphasis is placed on strategies aimed at the development of materials with a balance of properties, i.e., strength and ductility. In one such strategy, as the volume fraction of submicron grains is increased, tensile ductility increased and strength decreases. Enhanced tensile ductility is attributed to the occurrence of crack bridging as well as delamination between nanostructured and submicron-grained interfaces during plastic deformation. The finite element simulation on the deformation behavior and failure process of cryomilled bimodal Al alloys via a unit-cell model is also presented. The numerical results are in good agreement with tensile experimental data.

4:50 PM

Effect of Degassing on Mechanical Properties, Density and Porosity in Cryomilled Nanostructured Al-Mg Alloys: *Byungmin Ahn*[1]; Piers Newbery[2]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California; [2]University of California

5083 Al powders were ball-milled in a cryogenic temperature to accomplish nanocrystalline grains, and then degassed to remove residual gaseous phases at different temperatures and time. The degassed powder was consolidated using CIP (Cold Isostatic Pressing) followed by forging to produce bulk nanostructured materials. The mechanical properties, such as yield strength and elongation, of the bulk materials were enhanced relative to 5083 Al with conventional grain size. The mechanical properties of these materials tended to vary with respect to different degassing conditions, which also correlated with the density and porosity of the resulting bulk materials. The relation between the mechanical properties and microstructures also will be discussed in detail. In the present study, the variation of microstructure, porosity and density was investigated using optical microscope, SEM, TEM and density measurements. The quantitative analysis of density and porosity provides insight and a basis for optimizing the degassing conditions and production procedures.

5:10 PM
Characterization of Mechanical Alloying Processed Ti-Si-B Nanocomposite Consolidated by Spark Plasma Sintering: H. B. Lee[1]; I. J. Kwon[1]; *Young-Hwan Han*[2]; [1]Myong-Ji University; [2]University of California, Davis/Sungkyunkwan University, Korea

The microstructure and mechanical properties of TiB2/Si nanocomposites based on the Ti-Si-B system, consolidated by spark plasma sintering of mechanically alloyed activated nanopowders, have been characterized. Mechanical Alloying was carried out in a planetary ball mill for 180 min with 350 rev min^{-1}. The powders were pressed in vacuum at a pressure of 60 MPa, generating a maximum temperature in the graphite mould of 1400°C. Analysis of the synthesized nanocomposites by SEM, XRD and TEM showed them to consist of TiB2 second phase, sub-micron in size, with no third phase. Composites consolidated from powders mechanically alloyed from an initial elemental powder mix of 0.3 mol Si, 0.7 mol Ti, and 2.0 mol B achieved the best relative density (97%) and bending strength (774 MPa); the highest Vickers hardness of 14.7 GPa was achieved for the 0.1-0.9-2.0 mol starting composition.

5:30 PM
Processing Ti-Al-Nb Sheet Materials by Accumulative Roll Bonding and Reaction Annealing from Ti/Al/Nb Elemental Foils: *Rengang Zhang*[1]; Viola L. Acoff[1]; [1]University of Alabama

The Ti-46Al-9Nb (at%)was produced using accumulative roll bonding followed by two-stage annealing from elemental foils. Well-bonded sheet materials were successfully obtained with the initial rolling reduction of 50%. Two-stage annealing of 600°C and 1400°C was employed to promote the formation of intermetallic compounds. The SEM and XRD analysis indicated that there were no room temperature solid-state reactions between Ti/Al/Nb foils, even at high cycles. Besides the annealing time and temperature, rolling strain also has a significant effect on the production of the intermetallic compound. Differential thermal analysis (DTA) was used to characterize the effect of rolling strain on the solid-state reaction and phase formation in the composites during annealing. It is clear that the first annealing stage did promote the complete reaction of the Al layers to form the intermetallic compounds TiAl3 and NbAl3. After the second stage annealing, the desired intermetallic compounds, composition and lamellar structure were achieved.

5:50 PM
High Strength and Ductility of Nanostructured Al-Based Alloy Prepared by High Pressure Technique: *Nikolay Krasilnikov*[1]; [1]Ulyanovsk State University

The structure and mechanical properties of Al-based alloy 2024 after high-pressure torsion (HPT) was investigated. Alloy 2024 with homogeneous structure and grain size about 70 nm was obtained using HPT at 6 GPa pressure and 5 turns of anvils at room temperature. The nanostructured alloy at room temperature demonstrated very high UTS above 1100 MPa, and superplastic behavior at temperature higher then 300°C. The microhardness of nanostructured alloy after superplastic deformation (1.5 GPa) was more than after standard treatment of coarse-grained alloy (1.2 GPa). The influence parameters of HPT and heat treatment on structure and deformation behavior of alloy were studied. Opportunity of achievement in metals and alloys of combination high strength and good ductility opens perspectives of its application in industry, particularly, for mircosystems and for high-strength details with complex geometry obtained due to superplastic forming.

TUESDAY PM

Amiya Mukherjee Symposium on Processing and Mechanical Response of Engineering Materials: Poster Session: Processing and Mechanical Response of Engineering Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
Program Organizers: Judy Schneider, Mississippi State University; Rajiv S. Mishra, University of Missouri; Yuntian T. Zhu, Los Alamos National Laboratory; Khaled B. Morsi, San Diego State University; Viola L. Acoff, University of Alabama; Eric M. Taleff, University of Texas; Thomas R. Bieler, Michigan State University

Tuesday 5:15-7:00 PM Room: 217C
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Comparative Analysis of Microstructural Defects Obtained by Quasi-Isentropic Compression of Copper via Gas-Gun and Laser Driven Loading: *Hussam Nassib Jarmakani*[1]; J. M. McNaney[2]; M. S. Schneider[1]; D. Orlikowski[2]; J. H. Nguyen[2]; B. Kad[1]; M. A. Meyers[1]; [1]University of California, San Diego; [2]Lawrence Livermore National Laboratory

This current effort aims at understanding the differences in microstructural defects in copper obtained by quasi-isentropic loading achieved using two different methods: gas-gun loading and laser-driven loading. Although the pressures reached in both cases are the same, the strain-rates achieved are approximately three orders of magnitude higher in the laser experiments (10^7 1/s as compared to 10^4 1/s for gas-gun experiments). Dislocation activity at lower pressures in both cases are compared, and the transition of the defect substructure into stacking faults and twins is discussed. A constitutive model describing the slip-twinning transition for both cases is also introduced.

Effect of Competing Deformation Mechanisms on the Plastic Anisotropy of Zr Severely Deformed by Equal Channel Angular Extrusion (ECAE) at Room Temperature: *Guney Guven Yapici*[1]; Irene J. Beyerlein[2]; Ibrahim Karaman[1]; Carlos N. Tome[2]; [1]Texas A&M University; [2]Los Alamos National Laboratory

Recent findings have shown that a number of materials exhibit plastic anisotropy after large strain deformation. Huge strains and various routes applied during ECAE alter the microstructure and crystallographic texture significantly leading to considerable flow stress anisotropy. High purity Zr billets with different initial textures are successfully extruded at room temperature. Post-processing compressive response up to 30% strain along three orthogonal directions are reported along with microstructural and textural evolution at intermediate strain steps. This extensive experimental approach will shed light on the governing deformation mechanisms, namely slip and twinning and their interactions. Also, it will help in developing accurate single crystal hardening formulations that take into account the interaction between dislocation substructures and the operative deformation modes. A visco-plastic self consistent model predicting the texture and shape evolution of individual grains is utilized to relate these single crystal hardening models to the anisotropy in the deformation of polycrystalline Zr.

Finite Element Simulation of Selective Superplastic Forming of Friction Stir Processed 7075 Al Alloy: *Yanwen Wang*[1]; Rajiv S. Mishra[1]; [1]University of Missouri-Rolla

For many superplastic formed components, only some regions undergo superplastic deformation. In these cases, in stead of choosing expensive superplastic materials for the entire sheet, conventional materials can be chosen and friction stir processing (FSP) can be performed in the selected regions to make them superplastic. This is called "selective superplastic forming"(US Patent #20020079351). In this study, finite element simulation of superplastic forming of bowl shape component has been conducted. We chose commercial 7075 Al alloy and FSP was used to generate fine grained regions with different grain sizes. The pressure schedule, the overall forming time and the final thickness distribution in the formed component were calculated. These simulations demonstrate the design possibilities with this new concept. The authors gratefully acknowledge the support of National Science Foundation through grant # 0323725.

Fracture Mechanism of Bimodal Structured Nanocrystalline Al-Mg Alloys: *Zonghoon Lee*[1]; Velimir Radmilovic[1]; Enrique J. Lavernia[2]; Steven R. Nutt[3]; [1]Lawrence Berkeley National Laboratory; [2]University of California at Davis; [3]University of Southern California

Bimodal structured bulk nanocrystalline Al-Mg alloys, which were comprised of nanocrystalline grains separated by coarse grains, show balanced mechanical properties of enhanced ultimate strength and reasonable ductility/toughness compared to conventional Al-Mg alloys and other nanocrystalline metals. Our previous investigation of tensile test, TEM and FEM analyses had proposed unusual deformation mechanisms and interactions between ductile coarse grains and nanocrystalline regions. However, the direct evidence of the deformation and fracture has not been fully provided. In this work, embedded cracks and voids in the tensile fractures were revealed using FIB, SEM and analytical TEM, which gave explanations of the relation between the improved mechanical properties and the fracture mechanism. The findings suggested that cracks nucleated in nanocrystalline region during tensile loading were arrested at coarse grains. The incorporation of ductile coarse grains, which effectively blunted advancing cracks and impeded crack growth by bridging crack wakes, resulted in enhancing the toughness.

Mechanical Behavior of Bulk Nanocrystalline Ti Alloys Produced by Cryomilling: Alejandro Zúñiga[1]; *Fusheng Sun*[1]; Paula Rojas[2]; Bing Q. Han[1]; Enrique J. Lavernia[1]; [1]University of California, Davis; [2]Pontificia Universidad Católica de Valparaíso

The influence of the microstructure and chemical composition on the mechanical behavior of Ti alloys consolidated from cryomilled powders is described. The powders were cryomilled in liquid nitrogen and liquid argon, and then consolidated using Spark Plasma Sintering. The microstructure of the consolidated samples consisted of an α matrix with some retained β phase. The chemical analyses showed that the amount of nitrogen in the nitrogen-milled sample was higher than that of the argon-milled sample. The tensile properties of the argon-milled sample were 825 MPa of yield stress and 17% of elongation; whereas the compression properties of the nitrogen-milled sample were 2650 MPa of UTS and 0.11 of strain to failure. The compression behavior of nitrogen-milled samples after annealing at 500, 600 and 700°C was also investigated. The low ductility-high strength observed in the nitrogen-milled sample may be attributed to the large amount of nitrogen incorporated in solid solution.

Mesoscale Effects on the Mechanical Behavior of Inertial Confinement Fusion Ablator Materials: *Eric N. Loomis*[1]; Pedro Peralta[2]; Damian Swift[1]; Shengnian Luo[1]; Tom Tierney[1]; Ken McClellan[1]; [1]Los Alamos National Laboratory; [2]Arizona State University

The mechanical behavior of inertial confinement fusion (ICF) ablator materials is being studied to determine mesoscale shock effects during the implosion of spherical capsules. Specifically, plastic wave and grain boundary effects on macroscopic deformation are being considered. Laser shock and recovery experiments with peak applied pressure between 5 and 15 GPa have been performed on ~200 micron thick (5 mm diameter) planar specimens of a model anisotropic material, nickel aluminide (NiAl). Cross-section transmission electron microscopy (XTEM) was performed on shocked samples to characterize the degradation of the plastic wave along the shock direction. Post-shock characterization has shown that defect production and large lattice rotations are confined to within 30 microns from the ablation surface of single crystal NiAl. Be-Cu alloys have also been tested under shock conditions with in-situ velocity diagnostics to measure variations in shock breakout time at the free surface as a way of quantifying implosion symmetry.

Microstructure and Mechanical Behavior of Liquid Metal Cooling Single Crystal Ni-Based Superalloy, N5: Ganjiang Feng[1]; *Steve Balsone*[1]; Andrew Elliott[1]; Jon Schaeffer[1]; [1]General Electric

The increased firing temperature and the thermal efficiency of industrial gas turbines (IGT) demand sound castings and improved mechanical properties for hot gas path components. In the past ten years, the liquid-metal cooling (LMC) high gradient casting process has emerged as the most promising technology that produces single crystal (SC) and directionally solidified (DS) castings with substantially refined dendrite arm spacing and homogeneous microstructure. In the present study, the microstructures of LMC and conventional radiation cooled single crystal N5 castings were compared. Microstructural features such as dendrite arm spacing, phase segregation, carbides, and gamma prime morphology were analyzed. The effect of the refined microstructure under different heat treatment conditions on low-cycle fatigue and creep properties was also discussed.

Microstructure and Mechanical Properties of Hot and Cold Deformed Ti-6Al-4V: *Mehmet N. Gungor*[1]; Lawrence S. Kramer[1]; Hao Dong[1]; Ibrahim Ucok[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation

The microstructures and mechanical properties of seamless Ti-6Al-4V tubes manufactured by various deformation processes, including hot extrusion, hot rotary piercing and cold flowforming, were studied. Tensile and fatigue tests were conducted on extruded and rotary pierced tubes in the annealed condition, and on flow formed tubes in the stress relieved condition. Microstructure development in the tubes was examined via metallography, texture analysis, scanning electron microscopy and transmission electron microscopy techniques. This paper compares and discusses the mechanical testing and microstructural examination results for tubes produced by different manufacturing methods. This work was prepared by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation (CTC), under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the U. S. Navy Manufacturing Technology Program.

Modeling the Anomalous Flow Behavior of Ni_3Al Single Crystals: *Yoon S. Choi*[1]; Dennis M. Dimiduk[2]; Michael D. Uchic[2]; Triplicane A. Parthasarathy[1]; [1]UES Inc; [2]Air Force Research Laboratory

In the present study we developed a new comprehensive crystallographic constitutive model for Ni_3Al and $Ni_3(Al,X)$ intermetallic single crystals. This model aims at capturing important thermo-mechanical features of Ni_3Al, such as anomalous flow stress and work-hardening rate (WHR) variations with temperature, the strain dependence of these anomalous behaviors, tension-compression asymmetries over a stereographic projection triangle and the Cottrell-Stokes type two-step (at T_1 and T_2) deformation behavior. The framework of the model was structured based on two major contributions to the plastic flow of Ni_3Al, the motion of the macro-kinks (MKs), and the repeated cross-slip exhaustion and athermal defeat of the screw dislocation segments. The contribution of the irreversible obstacle storage was also incorporated in the constitutive formulations as a resistance against the glide of MKs. The model was implemented in the FEM framework, and the simulation results showed a qualitative agreement with experimental observations.

Modeling the Metal Deformation Path during Friction Stir Welding: *Judy Schneider*[1]; Arthur C. Nunes[2]; [1]Mississippi State University; [2]NASA-Marshall Space Flight Center

In friction stir welding (FSW), a rotating threaded pin tool is inserted into a weld seam, literally stirring the edges of the seam together. To determine the optimal pin tool design and processing parameters to produce a defect free weld, a better understanding of the resulting metal deformation flow path or paths is required. In this study, wire markers are used to trace the streamline flow paths of the metal. X-ray radiographs are used to record the post weld segmentation and position of the wire. The post weld position of markers are used to evaluate the effect of the weld parameters on the entrainment of the metal into the different flow paths. This data is being used to validate a kinematic based model to describe the metal deformation path as a function of the weld parameters and pin tool design.

Primary Creep in Equiaxed (Duplex) TiAl Alloys: *Thomas R. Bieler*[1]; [1]Michigan State University

Primary creep in TiAl is of significant importance for heat engine applications. Though lamellar microstructures usually have better creep resistance and toughness than duplex microstructures, some applications need the better ductility afforded by duplex microstructures. Primary creep in duplex alloys is characterized by a two stage primary creep process, consisting of a viscous glide process that exhausts itself, where a diffusional based dislocation creep process is observed. Much of this initial glide process occurs by either mechanical twinning parallel to the existing lamella in lamellar grains or by a shear transformation process by which metastable alpha phase is transformed to gamma phase, in some alloys.

TUESDAY PM

The influence of several alloying elements including C on primary creep properties is summarized.

Synthesis and Mechanical Properties of Ultrahard $AlMgB_{14}/TiB_2$ Composite: *Xuezheng Wei*[1]; Bruce Cook[1]; [1]Iowa State University

$AlMgB_{14}/TiB_2$, the second hardest (hardness around 46GPa) bulk material only after diamond, has recently attracted some attention due to its superior wear resistance compared with cemented carbide and cubic BN. This compound was fabricated by high energy ball milling and subsequent hot pressing. One problem encountered in this process is the existence of agglomerate in mechanical milled powder lowered the density and mechanical properties of hot pressed ceramic compound. In this study, process control agent (PCA) was added during mechanical milling and the effects of PCA on agglomerate, powder yield, particle size, and the density of hot pressed ceramic were investigated. XRD, SEM, and TEM were used for phase identification and microstructure, particle size analysis. It was found that with optimum amount of PCA, agglomerate-free powders were obtained; and the density and mechanical properties including hardness, toughness, and wear resistance of ceramic prepared with agglomerate-free powders were significantly increased.

Wear Behavior of AA1060 Reinforced with Alumina under Different Loads: *Mario Roberto Rosenberger*[1]; Romina Borelli[1]; Alicia Ares[1]; Elena Forlerer[2]; Carlos Schvezov[1]; [1]National University of Misiones; [2]National Commission of Atomic Energy

The wear behavior of samples of AA1060 aluminum matrix reinforced with 15% of alumina particles in a range of loads between 4.9 N and 130.5 N, were determined using a pin-on-ring machine. The tests were performed at a velocity of 2.7 m/s up to achieve the steady-state. The counterface was a carbon steel ring of 280 HB in hardness. Optical and electronic microscopy, X-Ray energy analysis and hardness measurement were performed in order to characterize the worn samples. A mild wear mechanism is present for loads lower than 80 N and at larger loads changes to severe wear mechanism. In the mild wear regime a Mechanically Mixed Layer (MML), with Iron from the counterface and material of the composite, was formed. This MML was responsible of the wear resistance of the composite. At larger load the conditions produced large instabilities which prevent the formation of the protective Mechanically Mixed Layer.

Process-Structure-Property Relationships in LENS® Processed PH13-8Mo Steel: *John E. Smugeresky*[1]; Baolong Zheng[2]; Yizhang Zhou[2]; Enrique J. Lavernia[2]; [1]Sandia National Laboratories; [2]University of California, Davis

The LENS® additive manufacturing processing of PH13-8 Mo has been evaluated for a range of conditions to establish process-structure-property relationships. Since LENS is also a tool for repair and modification, the interfaces between wrought material and laser deposited replacement features were also investigated. The effect of processing parameters on resultant shape and microstructure was determined using statistically designed experiments where melt pool volume was chosen as input variable. Powder feed rate was adjusted to insure a constant melt pool volume during part fabrication. The experiments were assisted with feedback control of the melt pool size. Microstructural homogeneity, mechanical property variation, and dimensional uniformity were determined for 10 mm by 10 mm by 50 mm parts. Tensile testing, Metallographic and electron optical analytical techniques, including fractography and EDS analysis were used to characterize the relationship between processing conditions, microstructure, and properties.

Biological Materials Science: Biological Materials Science

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Andrea M. Hodge, Lawrence Livermore National Laboratory; Chwee Teck Lim, National University of Singapore; Richard Alan LeSar, Los Alamos National Laboratory; Marc Andre Meyers, University of California, San Diego

Tuesday PM — Room: 212A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Andrea M. Hodge, Lawrence Livermore National Laboratory; Robert O. Ritchie, University of California

2:00 PM Invited
Biological Issues in Materials Science and Engineering: Interdisciplinarity and the Biomaterials Paradigm: *Lawrence E. Murr*[1]; [1]University of Texas

Biological systems and processes have had, and continue to have, important implications and applications in materials extraction, processing, and performance. This presentation will illustrate some of the important interdisciplinary, biological issues in materials science and engineering. These will include metal extraction (especially copper) involving bacterial catalysis; galvanic couples and bacterial-assisted corrosion of materials; antibacterial metals and other materials (particularly metal colloids and nanoparticle regimes); metal and material implants and prosthetics and the bio/materials interface; metal and material (particularly nanomaterial) toxicities and related health effects; materials as drug delivery systems (aerosol cages and controlled release configurations); biomimetics and biologically inspired materials developments. These examples will provide compelling evidence for emphasizing biology in materials science and engineering curricula, and the implementation of a biomaterials paradigm to facilitate the emergence of interdisciplinarity involving the biological sciences and the materials sciences and engineering. Supported by SCERP Projects A-04-1 and A-05-1, and a Murchison Endowment.

2:40 PM Keynote
From Concept to Patient - Materials Solutions for Bone Replacement: *William Bonfield*[1]; [1]University of Cambridge

Materials derived fromengineering practice have provided the foundation for a wide spectrum of first generation skeletal implants and prostheses, based on an acceptable biological response in the host tissue. However, it is now well recognised that a controlled and enhanced biological response, such as to produce bone apposition rather than fibrous encapsulation on an implant surface, can result in an extended device lifetime in vivo. As a consequence, there has been a major drive to innovate novel second generation biomaterials for specific clinical applications, with appropriate bio- and mechanical-compatibililty. An early example of this approach was the innovation of hydroxyapatite reinforced polyethylene composite (HAPEX(TM)) as a bone analogue. Modelled on the structure of cortical bone, HAPEX(TM) was tailormade to provide matching deformation characteristics and superior fracturetoughness to cortical bone, together with a favourable bioactive response. This research has culminated in amajor clinical application as a middle ear prostheses.

3:20 PM Invited
Failure of Human Cortical Bone: Aspects of Fracture Mechanics and Crack Propagation: *Robert O. Ritchie*[1]; Ravi K. Nalla[1]; Jamie J. Kruzic[2]; John H. Kinney[3]; [1]University of California; [2]Oregon State University; [3]Lawrence Livermore National Laboratory

The origins of the toughness of human cortical bone are examined by considering the salient micro-mechanisms of failure over a range of characteristic nano to macroscopic length-scales. It is argued that the structure of bone at the hundreds of microns, specifically the osteon structures, is most important in determining the fracture resistance. These mechanisms act to toughen bone by lessening the magnitude of stresses at the tip of any cracks. Although crack deflection along cement lines provides a source of toughening in the transverse orientation, crack bridging by intact regions in the crack wake provides for toughening in the medial-lateral and proximal-distal orientations. We show that biological aging, and certain disease states, which cause a deterioration in "bone quality" and hence raise the fracture risk, can be attributed to a degradation in the potency of crack bridging, a phenomenon that we believe is associated with excessive remodeling.

3:50 PM Break

4:10 PM Invited
The Ever Changing Materials Science and Engineering Curriculum: *Mark C. Hersam*[1]; Morris E. Fine[1]; [1]Northwestern University

In 1950, materials science and engineering did not exist as a university department. Instead, one found that the different classes of materials (e.g., metals, ceramics, polymers, and electronic materials) were taught in different departments. During the following 10 to 15 years, many universities initiated educational programs in materials science and engineering. The curricula were based on principles that apply to all classes of materials rather than separate introductory courses for each. Such core course sequences covered solid state theory, thermodynamics, phase transformations, kinetics, diffraction, microscopy, structure, and properties. The history of this development will be discussed. The current challenge is to introduce biological materials into the curriculum on the same basis. Strategies for meeting this challenge will also be outlined.

4:50 PM Invited
Mechanotransduction in Cells is Conducted by Supuramolecular Complex: *Masahiro Sokabe*[1]; [1]Nagoya University

Mechanotransdution is an important cell function to convert mechanical stimuli into electrical/chemical signals leading to important responses such as hearing, touch, proprioception or regulation of blood pressure. It also plays a crucial role in the regulation of cell-volume, -shape and -motility. In the past decade, we have partially understood the molecular mechanism of mechanotransduction based on the molecular identification of a certain class of mechanosensors such as mechanosensitive (MS) ion channels. The best known example is bacterial MS channels that can be activated simply by cell inflation upon hypotonic shock. However, eukaryotic cells have evolved more complicated mechanosensor systems. We found that some of their MS channels are associated with cytoskeletons and adhesion molecules, by which they obtain increased mechanosensitivity and force-direction sensitivity. Cytoskeleton would be a more efficient force transmitter than the membrane by its larger elastic modulus and adhesion molecules would provide stiffer base to generate stronger forces.

5:20 PM
Microstructure and Properties of Biocompatible TiO2/Ti: *Grant A. Crawford*[1]; K. Das[2]; Nikhilesh Chawla[1]; S. Bose[2]; A. Bandyopadhyay[2]; [1]Arizona State University; [2]Washington State University

Titanium oxide coatings have been shown to exhibit desirable properties as biocompatible coatings. In this talk we report processing and characterization of nanoporous TiO2 grown on commercially pure titanium substrates through anodic oxidation. Characterization of the as-processed coatings was conducted using scanning electron microscopy. Nanoindentation was used to probe mechanical properties such as Young's modulus and hardness. In addition, nanoindentation was used to measure the adhesion strength between the oxide coating and titanium substrate. Nanoscratch testing was employed as an additional method of testing adhesion strength. By changing processing conditions, both coating thickness and porosity were varied, and these variables related to mechanical behavior. Coatings were also immersed in simulated body fluids (SBF) up to 21 days to study apatite growth on coated surfaces. In vitro cell-materials interaction was studied using OPC1 human osteoblast cells to understand the influence of coating microstructure on cell attachment and proliferation.

5:40 PM
Cytotoxic Response and Cell Death Mechanisms for Carbon Nanotubes and Other Nanoparticle Aggregates: The Role of In Vitro Biological Assays in Nanomaterials Evaluations: *Karla F. Soto*[1]; K. M. Garza[1]; L. E Murr[1]; [1]University of Texas

Over the past years, research in nanotechnology has increased significantly. Carbon nanotubes and nanoparticulates have become a key component in this research. As there is a high demand and interest for these materials, there should also be a need to investigate their potential health and environmental aspects. The present study deals with cytotoxicity assays performed on an array of commercially manufactured nanoparticulate materials such as: Ag, TiO2, Fe2O3, Al2O3, ZrO2, Si3N4, carbonaceous nanoparticulate materials: single-walled and multi-walled carbon nanotube aggregates, black carbon; and naturally occurring chrysotile asbestos. The nanomaterials were characterized by TEM; the aggregates ranged from 25 nm to 20um. Cytotoxicological assays of these nanomaterials were performed utilizing a murine alveolar macrophage cell line and human macrophages as well as a human epithelial lung cell line as a comparator. The mechanism of cell death induced in these cell lines will also be discussed.

Bulk Metallic Glasses: Processing and Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Raymond A. Buchanan, University of Tennessee

Tuesday PM — Room: 217B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Daniel B. Miracle, U.S. Air Force; Katharine M. Flores, Ohio State University

2:00 PM Invited
Synthesis of High-Density Refractory Metal/Metallic Glass Nano-Composites: *Daniel Sordelet*[1]; Min Ha Lee[1]; [1]Ames Laboratory

Shear localization of bulk metallic glasses makes them candidates for some specific applications such as kinetic energy penetrators, but densities are in general too low for consideration. One possible solution is to introduce tungsten into a BMG matrix to achieve the required combination of density and deformation behavior. However, the use of body-centered cubic metals to increase the density presents another problem as they are notoriously resistant to shear localization because of their strong strain-rate sensitivity. We have expanded on the recent discovery that certain bcc metals exhibit a decrease in their strain rate sensitivity and show adiabatic shear-localized flow when deformed under uniaxial dynamic compressive loading conditions. Uniformly-layered nanometer-scale tungsten/metallic glass composite particles were fabricated by a milling process and subsequently consolidated by warm extrusion into fully dense rods. Deformation during quasistatic compression tests exhibited highly localized shear flow in these novel tungsten/metallic glass composites.

2:25 PM Invited
The Boson Peak in $Pd_{40}Cu_{40}P_{20}$: *D. J. Safarik*[1]; Ricardo B. Schwarz[1]; M. F. Hundley[1]; F. Trouw[1]; [1]Los Alamos National Laboratory

A low-temperature hump in C_p/T^3 vs. T (commonly known as the "Boson Peak") is ubiquitous to amorphous solids and is attributed to an excess of low-frequency vibrational modes not accounted for by the Debye model. We have measured the low-temperature heat capacity, the elastic constants, and the phonon density of states of both glassy and single-phase crystalline $Pd_{40}Cu_{40}P_{20}$ (the elastic constants and the specific heat were measured in a $Pd_{40}Cu_{40}P_{20}$ single crystal). The Cp/T^3 vs. T hump in crystalline $Pd_{40}Cu_{40}P_{20}$ was of comparable amplitude and peak temperature as for the Boson peak measured in $Pd_{40}Cu_{40}P_{20}$ glass. Inelastic neutron scattering measurements show that both materials have similar phonon density of states spectra, which deviate markedly from the Debye model assumption $[g(\omega) \sim \omega^2]$ at low frequencies. These densities of states are used to compute the specific heat, which we compare to our direct specific heat measurements.

TUESDAY PM

2:50 PM Invited

Nanocrystallizatrion Pathways in Ternary Zr-Based Bulk Metallic Glass: *Xun-Li Wang*[1]; Ling Yang[2]; Alexandru D. Stoica[1]; Jon Almer[3]; Peter K. Liaw[4]; [1]Oak Ridge National Laboratory; [2]University of Cincinnati; [3]Argonne National Laboratory; [4]University of Tennessee

High-energy synchrotron x-ray was used to study the nanocrystallization pathways in a Zr-based ternary bulk metallic glass. The experiment was conducted in-situ by making simultaneous time-resolved measurements of wide-angle diffraction and small angle scattering data under isochronal and isothermal heating conditions. Two successive phase transitions were observed during nanocrystallization, where an intermediate phase of icosahedral symmetry was identified. This result is similar to that observed in multi-component Zr-Ni-Cu-Ti-Al alloys. The implication of the intermediate phase on nanocrystallization behaviors will be discussed. This research was supported 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 Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38. The financial support by the National Science Foundation's International Materials Institutes (IMI) program at the University of Tennessee; with Dr. C. Huber, as the Program Directors; is also gratefully acknowledged.

3:15 PM Invited

Recent Advances in Development of Bulk Metallic Glasses: *O. N. Senkov*[1]; Daniel B. Miracle[2]; [1]UES, Inc.; [2]U.S. Air Force

Over the past several years, a number of new specific criteria for the selection of bulk metallic glass alloy compositions have been introduced. These new criteria include specification of the elastic properties of solute and solvent atoms, the relative and absolute values of atomic size and compositional ranges. The rules are based on topological and thermodynamic models of metallic glass stability and a new structural model for metallic glasses, and have led to discovery of a number of new metallic glasses. An overview of these selection criteria is provided, along with a brief discussion of the structural and thermodynamic models from which they are derived. These new theoretical developments allow accurate prediction of concentrations of a wide range of simple and complex metallic glasses. Properties of several new Ca-based metallic glasses, which have recently been produced based on these developments, are also presented.

3:40 PM Invited

Atom Probe Tomography Characterization of a Gas Atomized Bulk Metallic Glass: *Michael K. Miller*[1]; Shankar Venkataraman[2]; Jurgen H. Eckert[2]; L. Schultz[2]; Daniel J. Sordelet[3]; [1]Oak Ridge National Laboratory; [2]Technical University of Darmstadt; [3]Ames National Laboratory

An atom probe tomography characterization of gas atomized powder particles of a bulk metallic glass has been performed. The needle-shaped specimens required for the local electrode atom probe were fabricated from individual 10-40 micron diameter particles with the use of a dual beam focused ion beam miller. Details of the specimen preparation process and the microstructure of the as-atomized powder and the powder after different isothermal annealing treatments will be presented. Research at the SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:05 PM Break

4:15 PM

Glass-Forming Ability, Fragility, and Fracture Toughness in Various Bulk Metallic Glass Forming Systems: *Guojiang Fan*[1]; W. H. Jiang[1]; D. C. Qiao[1]; Hahn Choo[1]; P. K. Liaw[1]; [1]University of Tennessee

Understanding the glass-forming ability as well as their inherent fragility of bulk metallic glass (BMG) forming liquids has been actively pursued. More recent studies indicate that the fracture toughness of BMG alloys is closely related to their Poisson's ratio, and, thereby, to their kinetic fragility, which measures the steepness of the viscosity change at the glass transition. The relationship among the glass-forming ability, the fragility, and the fracture toughness for various BMG forming systems will be presented. The glass-forming ability of liquids is controlled both by the thermodynamic driving force and kinetic factor. The implications of thermodynamic and kinetic factors controlling the glass-forming ability on the fracture toughness of BMG alloys will be discussed. This work was supported by the National Science Foundation (NSF) International Materials Institutes (IMI) Program (DMR-0231320) with Dr. C. Huber as the Program Directors.

4:35 PM

Processing of Amorphous Zr52.5Cu17.9Ni14.6Al10Ti5 (VIT-105) with High Oxygen Contents Using Microalloying: *James Wall*[1]; Peter K. Liaw[1]; Hahn Choo[1]; [1]University of Tennessee

One of the obstacles that needs to be overcome to facilitate a wide-scale industrial acceptance of bulk metallic glasses as structural materials is their stringent processing requirements. Very small amounts of oxygen and other impurities can destabilize the supercooled liquid and lead to crystallization during cooling from the melt. Recently, a new microalloying scheme, which allows relatively high amounts of oxygen to be retained within the glass forming alloy Zr52.5Cu17.9Ni14.6Al10Ti5 (VIT-105), has been developed. Preliminary processing results are presented along with characteristic x-ray diffraction and differential scanning calorimetry analyses. Furthermore, preliminary results on fracture strengths of the materials with different oxygen/dopant levels are presented.

4:55 PM

Effect of Processing on Stability and Structure of Amorphous/Nanocrystalline Aluminum Alloys: *Timothy W. Wilson*[1]; Hahn Choo[1]; Cang Fan[1]; Alex C. Hannon[2]; Peter K. Liaw[1]; Laszlo J. Kecskes[3]; [1]University of Tennessee; [2]Rutherford Appleton Laboratory; [3]U.S. Army Research Laboratory

Understanding how the role processing affects the structure and in turn, the properties of amorphous and nanocrystalline aluminum alloys is vital for the future consolidation of ribbon and powder samples into bulk specimen. In this study, two types of $Al_{85}Y_7Ni_8$, $Al_{85}Y_7Fe_5Ni_3$, and $Al_{81}Y_7Fe_5Ni_7$ alloys were examined. One set was produced by rapidly solidifying melt-spun ribbons, and another set was prepared by mechanical alloying of elemental powders. The thermal stability of these two sets of materials was examined, and the studies revealed that powder samples show a higher thermal stability than the ribbon samples. In an effort to gain further insight on the structure–properties–processing relationship, the local atomic structures of Al-Y-Fe-Ni and Al-Y-Ni amorphous/nanocrystalline alloys were examined using neutron scattering. From this, atomic pair distribution function (PDF) analyses were performed on these alloys. Results showed that small changes in the composition and processing conditions affect the local atomic environment.

5:15 PM

Effects of Electrodes and Frequency of the Electromagnetic Vibrations on Glass-Forming Ability in Fe-Co-B-Si-Nb Bulk Metallic Glasses: *Takuya Tamura*[1]; Daisuke Kamikihara[1]; Yoshiki Mizutani[1]; Kenji Miwa[1]; [1]National Institute of Advanced Industrial Science and Technology

It is known that cooling rate from the liquid state is an important factor for producing the bulk metallic glasses. However, almost no other factors such as electric and/or magnetic fields were investigated. The present authors reported in Nature Materials 4(2005)289 that a new method for producing Mg-Cu-Y bulk metallic glasses by using electromagnetic vibrations is effective in forming the metallic glass phase. Moreover, the present authors have reported that the glass-forming ability of Fe-Co-B-Si-Nb alloys also enhances with increasing the electromagnetic vibration force. This study aims to investigate effects of electrodes and frequency of the electromagnetic vibrations on glass-forming ability in Fe-Co-B-Si-Nb bulk metallic glasses in order to investigate further. It was found that the enhancement of the glass-forming ability by the electromagnetic vibrations is not affected by electrodes.

Carbon Technology: Anode Baking

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Morten Sorlie, Elkem Aluminium ANS; Todd W. Dixon, Conoco Phillips Venco; Travis J. Galloway, Century Aluminum Company

Tuesday PM Room: 8A
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Trygve Foosnas, Norwegian University of Science and Technology

2:00 PM
Thermal Dilation of Green Anodes during Baking: *Juraj Chmelar*[1]; Trygve Foosnæs[2]; Harald Arnljot Øye[2]; [1]SINTEF ; [2]Norwegian University of Science and Technology

Pilot scale anodes were made using three single source and one blended coke to determine how the green anode properties affect the final baked anode. Testing was performed in an improved vertical dilatometer using samples 50 mm in diameter and 50 mm long to determine the effect of different heating rates. No sample support was used, however the effect of different packing materials was evaluated. A number of samples were prebaked to 450°C to reduce the initial volatile release and the subsequent sample swelling during dilatometric measurements. The sample shrinkage was calculated from the dilatometric data as the difference between the expansion at 550°C and 950°C. Dilatometric data for anodes prepared identically and with the same composition show differences due to varying mechanical properties of the cokes.

2:25 PM
New Process Control System Applied on a Closed Baking Furnace: *Inge Holden*[1]; Frank Heinke[2]; Frank Aune[1]; [1]Hydro Aluminium AS; [2]Innovatherm, Prof. Dr. Leisenberg GmbH+Co.KG

In 2004 the Årdal Carbon plant increased total baked anode production with 30% through retrofit of Furnace #3. Using the latest technology of the Hydro baking furnace design, annual capacity of a two fire-zone furnace was increased from 45.000 to 100.000 tons. The closed top furnace technology has traditionally higher tar content in the off-gas than furnaces of the open type design. At plants with electrostatic precipitators the major part of uncombusted volatiles are collected as tar which has been dealt with either by deposition, recycling or combustion. With this retrofit, flue gas treatment is now based on the technique of regenerative thermal oxidation. The paper demonstrate the improvements achieved with a new process control system and evaluate the most relevant parameters of the baking process such as combustion of volatiles, energy consumption and quality consistency.

2:50 PM
The Equivalent Temperature Method for Measuring the Baking Level of Anodes: *Lorentz Petter Lossius*[1]; Inge Holden[1]; Hogne Linga[1]; [1]Hydro Aluminium AS

The main use is for monitoring anode quality, but the method is also effective for mapping the baking level in entire sections of baking furnaces and for controlling laboratory scale anode preparation. Examples will be given from Hydro Aluminium baking furnaces and pilot anode plants. The method has been established as an ISO method, ISO 17499: "Carbonaceous materials used in the production of aluminium — Determination of the baking level expressed by the equivalent temperature". The background for the ISO method will be described, including the 2003 round robin for determining the precision which resulted in a repeatability of 9°E and reproducibility (between laboratories) of 14°E. The method is based on a calibration linking the equivalent temperature to L-sub-c crystallite height of a reference coke material. The principle including use of sets of calibration reference materials will be described.

3:15 PM
New Requirements and Solutions for the Fume Treatment at Paste Mixing and Anode Baking Plants: *Matthias Ernst Hagen*[1]; [1]LTB

The new IPPC regulation as well as local legislation forces manufacturers of anodes to improve their existing fume treatment center. The most critical emissions are Benzene and other PAH. Generally a regenerative thermal oxidiser (RTO) would be the best applicable solution. But the first systems installed, had huge problems with heavy tar and soot. The new RTO- based technology should avoid problems due to condensibles like heavy tar and soot and use the calorific value of the tar. For this task a new test plant was installed at a closed type baking furnace. The results of this test confirmed the basic design of the RTO but also caused a totally new design of the inlet valves. Furthermore a ceramic prefilter is required to avoid condensations at the inlet of the RTO. The result of the test and a plant in operation at a mixer will be shown.

3:40 PM Break

3:55 PM
Integrated Technology for Baking Furnace and Fume Treatment Plant: *Wolfgang K. Leisenberg*[1]; [1]Innovatherm GmbH

In the state of art anode plants baking furnace and fume treatment are regarded as isolated stand alone plants. But fume treatment starts in the baking furnace. The treatment begins as preheated air in the cooling area of the baking furnace and ends at the stack. We regard this process as an integrated one; we will find hidden potentials for both plants. Special situations as moving procedure or pitch burn can be anticipated in the FTP as feed forward information, while flue gas parameters can help to improve the baking process as feed back information. The paper also deals with the energy situation of the process including the use of free heat content of the flue gases. Last but not least a concept of the optimisation of the flue gas cleaning is presented, using the furnace as precipitator of VOC and condensable tar emissions.

4:20 PM
Flue Condition Index – A New Challenge to Increase Flue Lifetime, Operational Safety and Fuel Efficiency in Open Pit Anode Baking Furnaces: *Detlef Maiwald*[1]; Wolfgang Leisenberg[1]; [1]Innovatherm GmbH

The condition of the flue walls in open pit anode baking furnaces is an important factor in terms of production efficiency. The flue walls change their physical properties in terms of flow resistance, leakages and even mechanical stability due to the continuous heating/cooling cycle. Therefore the flue walls have to be observed regularly by maintenance staff and exchanged in average after a lifetime of 150 fire cycles. With the introduction of a flue condition index, each flue in a furnace is evaluated continuously. An on-line mathematical model detects the actual condition of each flue by correlation of the relevant process data available in the firing system. As a consequence the firing properties like the maximum fuel input or the draft can be adapted or limited to the actual condition of the flue. This prevents critical situations, avoids hot spots which increases operational safety, flue wall lifetime and fuel efficiency.

Cast Shop Technology: Shape Casting and Foundry Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Rene Kieft, Corus Group; Gerd Ulrich Gruen, Hydro Aluminium AS; Travis J. Galloway, Century Aluminum Company

Tuesday PM Room: 7C
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Mahi Sahoo, National Research Council of Canada

2:00 PM
Factors Influencing the Mechanical Properties of B206 Alloy Castings: *James F. Major*[1]; Geoffrey K. Sigworth[2]; [1]Alcan International Ltd; [2]GKS Engineering Services

The AA 206 family of Aluminum casting alloys includes some of the strongest, toughest, aluminum foundry alloys in current use. Primarily limited to aerospace and military applications, because of their tendency towards hot shortness, these alloys are also seeing increased interest as candidates for automotive suspension components. This paper will outline the results of studies carried out under the auspices of USCAR aimed at more fully characterizing and understanding the mechanical performance of the alloys as a function of composition, casting process, and temper. The results of a statistically designed experiment covering the allowable range of alloy elements and impurities within the AA 206 system will be interpreted in terms of the quality factor for both the T4 and T7 tempers.

2:25 PM
Characterization and Application of Novel 300-Series Die Casting Alloys: *Adam Kopper*[1]; Raymond Donahue[1]; [1]Mercury Marine

High pressure die casting is widely utilized in aluminum casting because of its economic benefits derived from high productivity, inexpensive alloys, and near-net shape part geometry. One important feature of the die casting process not yet fully exploited is the fine grain structure; a result of rapid cooling rates typical of the process. This is largely due to the negative impact of iron content and porosity. Iron offers solder resistance; but iron-containing precipitates are of a morphology which behaves as a stress riser resulting in brittle castings. Two new alloys are introduced which, through specific alloy composition, eliminate the need for iron and manganese in die casting alloys. Results show marked improvements in impact strength and elongation over standard die casting alloys.

2:50 PM
Effects of Sr and B Interactions in Hypoeutectic Al-Si Foundry Alloys: Liming Lu[1]; *Arne K. Dahle*[2]; [1]CSIRO; [2]University of Queensland

Strontium is the most widely used and a very effective element for modifying the morphology of eutectic silicon, while boron is commonly present in the commercial grain refiners used for Al-Si alloys. Recent work on the combined additions of Sr and Al-B master alloys and of Sr and Al5Ti1B master alloy has suggested that negative interactions occur between Sr and B added through the grain refiners. However, the effects and mechanisms for such negative interactions are not fully understood. This paper documents the experimental work and results aimed at determining the effects of Sr and B interactions on the solidification of hypoeutectic Al-Si foundry alloys. The mechanisms responsible for such negative interactions are further discussed.

3:15 PM Break

3:35 PM
Influence of Ca on Properties of Hypoeutectic Silumins Type AlSi7Mg: *Tomasz Stuczynski*[1]; Marzena Lech-Grega[1]; Zbigniew Zamkotowicz[1]; Boguslaw Augustyn[1]; [1]Institute of Non-Ferrous Metals

Paper presents results of investigations defining the role of Ca in range 0-500 ppm on properties and structure of hypoeutectic silumins AlSi7Mg0,4. The following properties were characterized: tensile strength, elongation, castability, gas ability, corrosion resistance for material with variable contents of Fe=0,2-0,6%. Paper also presents the influence of Ca as modifier of eutectic (a+Si) and intermetallic phases.

4:00 PM
Behaviour of the Solid-Liquid Interface at the Moment of Quenching during the Solidification of Aluminium Alloys: *Demian Ruvalcaba*[1]; D. Eskin[1]; L. Katgerman[2]; [1]Netherlands Institute for Metals Research; [2]Delft University of Technology

The fraction of solid phase is a key parameter studied in order to understand how the microstructure evolves during solidification of alloys. The quenching technique is widely employed to understand microstructural changes during solidification by freezing the microstructure. However, it has been found that the results obtained by the quenching technique overestimates the solid fraction when comparing to solidification models (e.g. lever rule and Scheil approximation). Therefore, it is important to understand how the microstructure develops during quenching in order to overcome in some way the overestimation of solid fraction when employing the quenching technique. In the present study aluminium alloys (i.e. Al- 3 wt.% Si and Al- 7 wt.% Cu) were solidified an quenched at different temperatures within the solid-liquid region. The microstructure is analyzed by optical microscopy and Electron Probe Microanalysis (EPMA). Overestimation of solid fraction was found in samples quenched at high temperature, while those quenched close to the eutectic reaction did not show overestimation of solid fraction compared to the lever rule and Scheil approximation. The existence of instabilities was found in those samples that show overestimation of solid fraction. The formation of instabilities away from the solid-liquid interface may be due to the formation and detachment of an instability at the very beginning of quenching. A discussion based on line-scan measurements and optical analysis is made for the existence of overestimation of solid fraction due to the formation of instabilities. The present research provides an insight in understanding the overestimation of solid fraction in order to overcome the problem when reconstructing the real microstructure that was present before quenching.

Characterization of Minerals, Metals and Materials: Structural Engineering Materials II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Jiann-Yang James Hwang, Michigan Technological University; Arun M. Gokhale, Georgia Institute of Technology; Tzong T. Chen, Natural Resources Canada

Tuesday PM — Room: 206A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Arun M. Gokhale, Georgia Institute of Technology; Junji Shibata, Kansai University

2:00 PM
Reconstruction, Visualization, and Characterization of Three-Dimensional Microstructure of High-Pressure Die-Cast AE44 Magnesium Alloy: *Jacqueline Milhans*[1]; Soon Gi Lee[2]; Arun Gokhale[2]; [1]Carnegie Mellon University; [2]Georgia Institute of Technology

High-pressure die-cast Mg-alloys are of significant interest in the automotive industry due potentials of weight savings and fuel efficiency. Visualization, characterization, and representation of three-dimensional (3D) microstructures are of significant interest for understanding and modeling processing-microstructure-properties relationships. In this contribution, an efficient and unbiased montage serial sectioning technique is applied for reconstruction of large-volume high-resolution (~ 1 mm) 3D microstructure of high-pressure die-cast AE44 Magnesium alloy. The 3D reconstruction reveals the morphology and geometry of the eutectic constituent as well as of Mg-rich dendrites. The surface rendering as well as volume rendering techniques is used for three-dimensional microstructure visualization.

2:25 PM
Magneto-Thermo-Mechanical Characterization of NiMnGa Single Crystals to Reveal Guidelines to Increase Actuation Stress by Magnetic Field Induced Phase Transformation and Variant Reorientation: *Haluk Ersin Karaca*[1]; Burak Basaran[1]; Ibrahim Karaman[1]; Yuriy Chumlyakov[2]; Hans Maier[3]; [1]Texas A&M University; [2]Siberian Physical-Technical Institute; [3]University of Paderborn

Magnetic shape memory alloys (MSMAs) have the ability to combine large strain output of conventional shape memory alloys with high frequency response of magnetostrictive materials. However, their operation range under stress is limited to a few megapascals In this work, an extensive experimental program was undertaken on NiMnGa single crystals using a unique magneto-thermo-mechanical (MTM) testing system in quest for identifying physical and microstructural parameters critical in increasing the magnetic actuation stress. The present paper will discuss guidelines to increase the actuation stress considering the coupled effects of magnetocrystalline anisotropy energy, detwinning stress, operating temperature, phase transformation temperatures and Curie temperature. Few specific experimental results will be presented in which one to two orders of magnitude increase was achieved in the magnetic actuation stress. This giant increase will be shown to be a consequence of field induced phase transformation which has been observed for the first time in these alloys.

2:50 PM

Comparison of Dislocation and Other Deformation Microstructures in As-Grown, Compression Tested, and Ballistically Penetrated W Single-Crystal Rods: *Micah T. Baquera*[1]; Lawrence E. Murr[1]; Carlos Pizaña[1]; Thomas L. Tamoria[2]; H. C. Chen[2]; S. J. Cytron[3]; [1]University of Texas; [2]General Atomics; [3]U.S. Army TACOM-ARDEC

A comparison of microstructures associated with W single-crystal ([001], [011], [111]) rods prior to deformation (as grown), and after quasi-static compression (at a strain rate of ~1/s) by transmission electron microscopy (TEM) shows a more than 10-fold difference in dislocation density. Dislocation substructures were not significantly different in the deformed samples. In contrast, [001] W rods penetrated into steel targets, at impact velocities of ~1.3 km/s, exhibited heavy dislocation densities and dynamic recrystallization as very distinct microstructural regimes. Twinning on {112} also occurred as a precursor to these microstructures near the projectile head. These results suggest that deformation accommodating the extreme strains and strain rates associated with the penetration of a single-crystal rod into a metal target is not adequately represented by conventional deformation as envisioned within the realm of room temperature strain-strain-rate deformation. (U.S. Army TACOM-Picatinny Prime Contract W15QKN-04-M-0267, Project No. 1A4CFJERIANG).

3:15 PM

Engineering Analysis of Subway Rolling Stocks for Safety Evaluation: *Jeongguk Kim*[1]; Jung-Won Seo[1]; Sung-Tae Kwon[1]; [1]Korea Railroad Research Institute

The safety assessment of subway rolling stocks was performed with various types of engineering analysis techniques for the understanding of the current wear status. The car body and bogies were characterized by the employment of nondestructive evaluation techniques, corrosion testing, and three-dimensional measurements. In braking system, degradation and performance tests were conducted, and functional and degradation tests for electrical system in order to identify the actual performance of the system. Moreover, stress and structural analyses using commercial finite element method software provided important information on stress distribution and load transfer mechanisms, and the FEM results were compared with running safety testing results. In this investigation, various advanced engineering analysis techniques for the safety analysis of subway rolling stocks have been introduced and the analysis results have been used to provide the critical information for the criteria of safety assessment.

3:40 PM Break

3:50 PM

Characterization and Modeling of 3-Dimensional Damage Progression of Laser-Deposited Iron-Based Materials Using X-Ray Tomography: *Paul T. Wang*[1]; Haitham El Kadiri[1]; Gabiel Pontimiche[1]; Mark F. Horstemeyer[1]; [1]Mississippi State University

LENS™ is one of the direct digital manufacturing techniques for fabricating high strength, near net shape metallic components. To understand and be able to virtually predict the service performance of LENS™ components, i.e., ductility, fatigue, and fracture strength, three-dimensional CT becomes a vital characterization tool for measuring initial and as-deformed states of material. In this case, dual X-ray tubes representing fine and finer resolutions of measurement were used to trace the evolution of damage state and assist the development of damage models. At the macro-scale, an internal state variable constitutive theory representing plasticity and hardening behavior is adopted for its ability of tracing deformation history; at the micro-scale, a 3-dimensional void-cell model is introduced where void growth and coalescence are validated by the dual-tube X-ray tomography. Results of 3-dimensional reconstruction of void images and its connection to modeling are discussed.

4:15 PM

Characterization of Aged U-Nb Alloys by X-Ray Diffraction: *Heather M. Volz*[1]; Robert E. Hackenberg[1]; Robert D. Field[1]; Andrew C. Lawson[1]; W. Larry Hults[1]; Ann Kelly[1]; David F. Teter[1]; [1]Los Alamos National Laboratory

Uranium alloys exhibit many different stable and metastable phases, including martensite. It is desirable to understand any long-term changes in the structure of these materials in order to better predict behavior over time, but changes may be subtle and elude standard metallographic techniques. To this end, several U-Nb alloys have been quenched and artificially aged for various times at temperatures up to 300C. Powder diffraction patterns were collected on a laboratory X-ray diffractometer along with a ceria standard, and analyzed using full pattern Rietveld analysis with GSAS. As surface condition is a critical issue in obtaining meaningful diffraction patterns in uranium alloys, sample preparation issues related to these materials and their effects on structural refinement and precision lattice parameters will be discussed. Aging-induced changes (or lack thereof) in lattice parameters, unit cell volume, atomic positions in the crystal structure, and phases will be also presented and interpreted.

4:40 PM

EDXRF Analysis of Alloys and Corrosion Products of Metallic Pre-Hispanic Pieces of the Museum of Ethnology and Archaeology of University of Sao Paulo: *Augusto Camara Neiva*[1]; Jérémie Nicolãe Dron[1]; Hercilio Gomes de Melo[1]; Silvia Cunha Lima[2]; [1]Escola Politecnica da Universidade de Sao Paulo; [2]Museu de Arqueologia e Etnologia da Universidade de Sao Paulo

An energy-dispersive x-ray fluorescence spectrometer was assembled with the aim of analysing alloys and corrosion products of artistic, ethnological and archaeological pieces. The spectrometer is semiportable and consists basically of an 60kV/2mA x-ray tube with W anode and a Si-drift x-ray detector with Peltier cooling, with no need of liquid nitrogen. It was used for the analysis of the alloys and occasional corrosion products of several metallic pre-Hispanic pieces of the Museum of Ethnology and Archaeology of the University of Sao Paulo. Special care was taken to account for the contribution of spurious radiation from both the Pb primary collimator and the Zr detector collimator. The main components of most alloys were Au, Ag and Cu, but Pb, Ca, Fe and other elements were also identified. The characterization of the alloys and corrosion products is important for the subsequent conservation of the collection.

5:05 PM

Corrosion Processes and Mineral Formation on the Surface of Bronze Monuments in Urban Environments: *Olga Frank-Kamenetskaya*[1]; Evgenii Treivus[1]; [1]Saint Petersburg State University

The experimental results of study of corrosion processes on Saint-Petersburg bronze monuments surface, received in situ and by means of simulating experiments, have been discussed. Influence of the time factor on corrosion film (patina) mineral composition has been analyzed. Direct dependence between patina mineral composition and environmental conditions has been proved. The sequence of patina phases crystallization has been established. Effect of the chemical composition and heterogeneity of artistic bronze and also monument surface structure on the rate of corrosion process has been regarded. The time variations of mass of the crystallized in the hydrochloric acid vapor phases have been investigated. The kinetic laws of their development at the initial stage of formation, indicating simple (non chain) reactions, have been revealed.

TUESDAY PM

Computational Thermodynamics and Phase Transformations: Alloy Models and Thin Films

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Dane Morgan, University of Wisconsin; Corbett Battaile, Sandia National Laboratories

Tuesday PM — Room: 210A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Vidvuds Ozolins, University of California; Michael I. Baskes, Los Alamos National Laboratory

2:00 PM Invited
Calculated Properties of Pu-Ga Alloys Using the Modified Embedded Atom Method: *Michael I. Baskes*[1]; Shenyang Hu[1]; Marius Stan[1]; [1]Los Alamos National Laboratory

The Pu-Ga system is perhaps the most complicated binary alloy system in nature. Not only does this system have important technological importance, but also scientifically it is extremely challenging. Previously we have used the Modified Embedded Atom Method (MEAM) to describe the behavior of both Pu and Ga. This method, though semi-empirical, is able to capture most of the important unusual behavior of both of these elements. In this presentation we show the results of recent calculations using MEAM for various alloys (phases and composition) in this complex system. Results presented will include simple bulk thermal and mechanical properties such as specific heat, thermal expansion, and elastic constants for the solid phases. Two-phase equilibrium will be discussed with respect to melting and the predicted phase diagram. Predictions will be compared with experiment when available.

2:30 PM Invited
Theoretical and Experimental Studies of Metastable Phases in Devitrification of the Glass Forming System Zr2(Pd,Cu): *James R. Morris*[1]; Yiying Ye[2]; Matthew J. Kramer[2]; Min Xu[2]; [1]Oak Ridge National Laboratory; [2]Ames Laboratory

$Zr2Cu_xPd_{1-x}$ is a glass forming system that shows a composition-dependent devitrification pathway. We have used ab initio calculations and x-ray diffraction to examine the competition between the phases, as a function of composition. Calculations reveal that Cu and Pd mix nearly ideally in the ground state structure. However, in the closest competing metastable phase, Cu and Pd preferentially mix. Supporting this calculation, this metastable phase is clearly observed in the experiments only when for equal Cu and Pd concentrations. For Pd-containing compounds, a metastable quasicrystalline phase is the first observed devitrification product. Contrary to existing suggestions, our calculations demonstrate that this phase is not related to the $NiTi_2$ structure, which is destabilized by Pd additions. 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, LLC, and Contract W-7405-ENG-82 with Iowa State University.

3:00 PM
A Modified Embedded Atom Method Interatomic Potential for the Fe-C System: *Byeong-Joo Lee*[1]; Young-Min Kim[1]; [1]POSTECH

For an elaborate control of microstructures and materials properties, it is desired to understand the materials behavior from more fundamental level, e.g. the atomic level. A semi-empirical interatomic potential for the Fe-C binary system has been developed based on the Modified Embedded Atom Method formalism. The potential describes various fundamental physical properties of pure carbon and iron well, as well as known physical properties of carbon as an interstitial solute element in bcc- and fcc-Fe (dilute heat of solution of carbon, vacancy-carbon binding energy and its configuration, location of interstitial carbon atoms and migration energy of carbon atoms). The applicability of the potential to atomistic approaches to investigate the interactions between carbon interstitial solute atoms and other defects such as vacancies, dislocations and grain boundaries, etc., will be presented. Some atomistic simulation results on the effects of carbon on various deformation and mechanical behavior of iron will also be presented.

3:20 PM
Building Empirical Interatomic Potentials to Describe C Interstitials in Fe: *Diana Farkas*[1]; Margarita Ruda[2]; [1]Virginia Tech; [2]Centro Atomico Bariloche

In this work, thermodynamic and lattice expansion data for C in Fe were used to develop an empirical potential that can describe small amounts of C interstitials in Fe. The potentials were tested for the prediction of martensitic structures and successfully predict the stability of the martensitic phase. They were also compared with first principle calculations for the effect of interstitial C in the cohesive energy of grain boundaries, where the correct trend is predicted. The results for lattice relaxation and energetics for the structure of the interstitial sites in the bulk are also compared with first principles results and the limitations of the technique are discussed.

3:40 PM
Molecular Dynamic Simulations of Thermodynamic Properties and Stability of Precipitates in Al-Cu Alloys: *Shenyang Hu*[1]; Michael Baskes[1]; Marius Stan[1]; Longqing Chen[2]; [1]Los Alamos National Laboratory; [2]Pennsylvania State University

Precipitate strengthening is one of the main mechanisms responsible for the improved mechanical properties of alloys. The thermodynamic stability of precipitates and mobility of precipitate/matrix interfaces control the rates of nucleation, growth and coarsening, and hence the precipitate volume fraction, morphology, size distribution, and the mechanical properties. The plate-like theta-prime precipitate is one of the main strengthening precipitates in Al-Cu alloys. In this work, molecular dynamics (MD) simulations are employed to calculate thermodynamic properties of various phases: alpha solid solution, theta prime, and theta phases. The free energy and entropy differences between a reference state and an Einstein solid were calculated using the method of adiabatic switching in a MD formalism. Analytic free energy functions were constructed as a function of temperature and Cu composition. In addition, Interface energies, interface structure and critical nuclei were obtained, and compared with those from first principles calculations and experimental measurements.

4:00 PM Break

4:10 PM Invited
Strain Contributions to Phase Transitions in Epitaxial Thin Films: Y. L. Li[1]; *Long Qing Chen*[1]; [1]Pennsylvania State University

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 a film thickness is small, i.e. below the critical thickness for nucleation of interfacial dislocations. There are increasing evidences that the thermodynamics of phase transitions in such epitaxial films can be profoundly affected by the strain imposed by a substrate. For example, it has been experimentally determined that ferroelectric phase transition temperatures in epitaxial thin films can be hundreds of degrees higher than the corresponding bulk. It will be shown that the observed huge shifts in phase transition temperatures can be rationalized using continuum thermodynamics taking into account the strain effect. It is suggested that significant changes in phase transition temperatures should be expected for any phase transition whose order parameter is coupled to strains.

4:40 PM Invited
Ab Initio Modeling of Pattern Formation in Bulk-Immiscible Heteroepitaxial Alloy Films: *Vidvuds Ozolins*[1]; Tejodher Muppidi[1]; [1]University of California

Formation of two-dimensional (2D) nanoscale patterns (stripes and disks) has been observed in several surface alloy systems composed of bulk-immiscible species. These 2D structures occur due to competition between short-range chemical bonds favoring phase separation and long-range elastic forces favoring intermixing. We present an accurate theoretical framework that incorporates interatomic interactions obtained from first-principles electron-structure methods. The central role in this formalism belongs to a Fourier transform of long-ranged pair interactions, which include both alloying and elastic strain effects. This formulation enables rapid screening for new alloy/substrate systems with strong energetic tendencies to form long-period ordered patterns. Applications to several metallic alloy systems on Ru(0001) and Mo(110) substrates and rare-earth disilicide nanowires on Si(100) will be shown.

5:10 PM
A New Model of Stress Generation during Thin Film Growth: *Edmund B. Webb*[1]; Steven C. Seel[1]; J. J. Hoyt[1]; Jonathan A. Zimmerman[1]; [1]Sandia National Laboratories

Residual stress in a material influences microstructure evolution. This is particularly true for thin films, which support higher stresses than bulk material due to restricted geometry. During Volmer-Weber thin film growth, discrete islands grow until their separation distance becomes small. Islands then elastically deform to close the gap between them, trading surface energy for strain energy. Island coalescence is believed to generate stress during thin film growth; however, details are only qualitatively understood - especially for nanometer islands since experiments are challenged to probe this scale. We present results from an analytical coalescence model that predicts, contrary to prior models, significantly reduced stress as a function of island size. Our model improves agreement with experiment by evoking a reasonablemechanism for island coalescence. Results are supported by atomistic and continuum models; however, the former reveal inelastic behavior for very small islands that cannot be predicted from analytical or continuum approaches.

Deformation and Fracture from Nano to Macro: A Symposium Honoring W. W. Gerberich's 70th Birthday: Length Scales

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: David F. Bahr, Washington State University; James Lucas, Michigan State University; Neville R. Moody, Sandia National Laboratories

Tuesday PM — Room: 214D
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: James Perry Lucas, Michigan State University; Maarten P. de Boer, Sandia National Laboratories

2:00 PM Invited
Cyclic Contact Deformation of Nanocrystalline Ni: *Ruth Schwaiger*[1]; Joerg Knyrim[1]; Subra Suresh[2]; [1]Forschungszentrum Karlsruhe, IMF2; [2]Massachusetts Institute of Technology

Nanocrystalline materials show potential for high performance applications due to their improved mechanical properties, such as higher strength and hardness, and enhanced resistance to wear. Understanding the damage behavior and the microstructural changes due to repeated contact loading is critical to assess the usefulness of nanocrystalline materials. Cyclic contact deformation was studied by repeatedly indenting the surface of Ni specimens of different grain size and by repeatedly sliding a tip over the surface. A systematic series of experiments under well-controlled conditions were performed using instrumented indenters. We studied the microstructural changes and changes in the mechanical response to the cyclic contact. We observed prominent changes of the grain structure during these experiments. This microstructural instability is most apparent in materials with a very fine initial grain size and represents a threat to the use of fine-grained materials in many applications. Effects of microstructural and experimental length scales will be described.

2:20 PM
Indentation Size Effect in Metallic Material: From Pop-In to Macroscopic Size Independent Hardness: Karsten Durst[1]; Björn Backes[1]; *Mathias Göken*[1]; [1]University Erlangen-Nürnberg

In this paper, size effects observed during indentations with Berkovich and cube corner indenters are discussed in terms of geometrically necessary dislocations (GND). Considering the plastically deformed volume underneath the indenter, a modified Nix/Gao model of the ISE is used for describing the depth dependence of hardness. For the formation of a permanent hardness impression, the necessary dislocation density must be nucleated. The transition from elastic to plastic deformation generally occurs in a pop-in event. After the pop-in, the hardness of the material is described by the Taylor relation for dislocation hardening, where the geometrically necessary dislocations are the dominant factor. At higher depth, the density of the statistically stored dislocations, at a certain representative strain, controls the deformation resistance. An excellent agreement is found between the modeled depth dependence of hardness and indentation experiments on Al, W, Cu, Ni at indentation depths from the pop-in to 3000 nm.

2:35 PM
Characterization of the Dislocation Substructure beneath Nanoindentations in hcp α and bcc β Phases in Ti-V Based Alloys: *Gopal B. Viswanathan*[1]; Eunha Lee[1]; Dennis Maher[1]; Srikumar Banerjee[1]; Hamish L. Fraser[1]; [1]Ohio State University

The hardness of α and β phases in the alloys Ti-6Al-4V and Ti-22V are assessed using nanoindentation technique. Results indicate that the hardness varied with the depth of indentation and the orientation of grains in both alloys. Dislocation substructures are characterized by TEM analysis of the thin-foil membranes cut through specific indents with a focused-ion-beam instrument. Regarding the orientation dependence of hardness in the α phase, the nature of statistically stored dislocations and that of geometrically-necessary dislocations is identified. The occurrence of former dislocations are predicted from Schmid's Law, while noting the presence of minor densities of other dislocations required presumably because of the shape change imposed by the nanoindenter. The geometrically-necessary dislocations are identified as appropriate combinations of slip dislocations such that an overall-displacement parallel to the direction of the indentation results. Detailed dislocation-substructure analysis in the β phase is currently underway. These results will be presented and discussed.

2:50 PM
Correlating Changes in Dislocation Substructures to Plasticity Size-Effects in Ni, Ni_3Al, and Ti-6242: *Dave M. Norfleet*[1]; Steven Polasik[1]; Dennis M. Dimiduk[2]; Michael D. Uchic[2]; Michael J. Mills[1]; [1]Ohio State University; [2]Air Force Research Laboratory

Recently much attention has been given to mechanical size effects at the micron size scale. However, the specific details of how dislocation micro-mechanisms are affected by sample volume are still unresolved. Using current micromechanical fabrication and testing methods, micron-sized compression samples of Ni, Ni_3Al, and single colony Ti-6Al-2Sn-4Zr-2Mo-0.1Si were tested at room temperature. The pure Ni and Ni_3Al alloys displayed strong size effects (a marked increase in strength) as the sample volumes were reduced. In comparison, the Ti-6242 material exhibited opposite behavior, displaying a reduction in strength as the sample volume approached micron size scales. Subsequent FIB-based TEM foils were prepared along active slip bands and along the gauge length to study the dislocation dynamics and deformation homogeneity. Dislocation analysis was performed on each of these materials to gain an understanding as to the micro-mechanisms that control plasticity at this size scale.

3:05 PM
Slip Behavior in Single Crystal Ni-Base Superalloys: *Fereshteh Ebrahimi*[1]; Eboni Westbrooke[1]; [1]University of Florida

It has been shown that single crystal Ni-base superalloys do not comply with the Schmid's law, i.e. the critical resolved shear stress depends on the loading orientation. Furthermore, the slip bands do not follow the predicted slip traces and their appearance depends on the loading orientation. In this study, the room temperature plastic deformation of two Ni-base superalloys in HIP'ed and un-HIP'ed conditions was investigated. Optical, scanning and transmission electron microscopy techniques were employed to investigate the evolution of deformation bands as functions of strain and loading orientation. Detailed analysis of the results revealed that geometrical arrangements of precipitates, pores (or eutectic pools) and dendrites affect the plastic deformation at nano-, micro-, and macoscales, respectively. It is shown that the development of local tri-axial stresses owing to these inhomogenities influence the path of deformation bands as well as the level of remote stress needed for yielding.

3:20 PM
Dislocation Nucleation and Source Activation through Yield Point Phenomena during Nanoindentation: *Ali Zbib*[1]; David F. Bahr[1]; [1]Washington State University

Experimental studies on single crystal tungsten were performed using indentation to examine yield point phenomena with respect to dislocation nucleation and source activation. Macroindentation was used to create a spatially distinct array of dislocations in the single crystal. Nanoindentations which exhibited a yield point indicative of the onset of plasticity were then performed at various positions, corresponding to different dislocation densities identified using dislocation etch pitting techniques. The maximum shear stresses underneath the indenter at which yielding occurs was then compared to dislocation densities, and found to relate inversely with dislocation density. The maximum shear stress reached 1/11 of the shear modulus at a dislocation density of 1.81 per square micron and 1/9 the shear modulus at a dislocation density of 0.0035 per square micron. The effects of tip radius on the stress field under the tip, and the volume in which a pre-existing dislocation is activated, will be discussed.

3:35 PM Break

3:55 PM Invited
Deformation and Patterning in the Micro- and Nano- Scale: *Elias C. Aifantis*[1]; Daniel Walgraef[2]; [1]Michigan Technological University; [2]Free University of Brussels

Various spatiotemporal patterning phenomena of strain and dislocation populations are discussed. A self organization approach is employed to describe the relevant instabilities in small volumes, surfaces and interfaces. The competition of strain, interaction and surface energies are considered in both linear and nonlinear regimes. The results of the analysis are compared with experimental observations.

4:15 PM
Analytic Treatment of Metallic Multilayer Strength at All Length Scales: *Lei Fang*[1]; Lawrence H. Friedman[1]; [1]Pennsylvania State University

Metallic multilayers can be used as ultra-high strength coatings. They exhibit a very pronounced size-effect where the mechanical strength depends on the layer thickness. Traditionally, the Hall-Petch relation from dislocation pileup theory is used to describe the size effect However, more rigorous application of dislocation pileup theory as applied to multilayers predicts significant deviation from the Hall-Petch relation due to elastic inhomogeneity, discreteness of dislocations and dislocation source operation. The necessary modifications to the Hall-Petch Relation are presented. An analytic formula accounting for these effects can only be obtained in a piecewise fashion. The variation of strength with layer thickness must be broken down into four length-scale regimes, and a simple analytic formula is obtained for each regime. This formulation allows one to bridge the length scales and predict multilayer strength from microscopic parameters (interface strength and dislocation source characteristics) and fundamental material parameters (elastic moduli and crystal structure).

4:30 PM
Thickness Effects on the Plasticity of Gold Films: *Megan J. Cordill*[1]; Neville R. Moody[2]; David P. Adams[2]; David F. Bahr[3]; Alex A. Volinsky[4]; William W. Gerberich[1]; [1]University of Minnesota; [2]Sandia National Laboratories; [3]Washington State University; [4]University of South Florida

Gold films are used in advanced applications where limited film thickness can affect the film's mechanical properties. It has been shown that as film thickness decreases the energy required for film delamination also decreases. This decrease is most likely due to the amount of plasticity available for deformation. To examine these effects, nanoindentation was employed to simulate a sharp crack on the film's surface. The indenter tip will cause deformation by dislocation nucleation. The time dependent elastic recovery of the indents will be monitored using scanning probe microscopy techniques. Gold films (40 nm and 200 nm) were indented using three tips: a blunt berkovich, a sharp berkovich, and an ultra sharp cube corner. Each tip demonstrates different behavior as a function of film thickness and initial indentation depth. Experimental results are compared to existing dislocation shielding models. This work funded by the United States Department of Energy through contract DE-AC04-94AL85000.

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Relating Gradient Plasticity with Dislocation Mechanics through the Use of Nanoindentation: *Katerina E. Aifantis*[1]; Jeff T. H. DeHosson[1]; John R. Willis[2]; [1]University of Groningen; [2]University of Cambridge

During the past twenty years strain-gradient plasticity has been used extensively to capture deformation features at the micron/nanometre scale, such as the development and persistence of dislocation patterns, and the occurrence of size effects that classical plasticity could not describe. A key material parameter that comes into play in all gradient theories is the internal length. From a mathematical point of view it is required for dimensional consistency; nevertheless it has not yielded to precise physical interpretation. In the present talk by relating a theoretical gradient plasticity formulation to experimental results obtained through nanoindentation the internal length is investigated and interpreted within a dislocation mechanics framework. In the experiments on bi-crystals of bcc metals, the deformed volume is limited to a submicron length scale by the indenter on the one side and the grain-boundary on the other side, resulting in a significant size effect on the strain bursts observed.

5:00 PM
Deformation Behavior of Matal-Matrix Nanocomposites Reinforced by Carbon Nanotubes: *Donghyun Bae*[1]; [1]Yonsei University

Deformation behavior of nanocrystalline (nc) aluminium and its alloys (the grain size < 100nm) reinforced by carbon nanotubes(CNTs) at room temperature has been systematically investigated. Nc powders and nanocomposite powders with randomly embedded CNTs are produced via mechanical milling. And the rods (diameter: 6mm, length: 300mm) are fabricated by hot-extrusion. Nanocomposites show yield strength more than 10 times higher than that of the starting sample due to the effects of nanograins, the presence of nano-particles and solute atoms distributed in the nc matrix, and the incorporation of uniaxially aligned CNTs. Furthermore, nanocomposites exhibit high elongation around 8% under uniaxial tension mainly due to the resistance of neck growth. High resolution transmission electron microscopy analyses reveal that twins and partial dislocations are frequently observed in the matrix and the interface between CNTs and the matrix is strongly bonded. The detailed deformation mechanisms of such nanocomposites will be presented.

Fatigue and Fracture of Traditional and Advanced Materials: A Symposium in Honor of Art McEvily's 80th Birthday: Fatigue and Fracture V

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Leon L. Shaw, University of Connecticut; James M. Larsen, U.S. Air Force; Peter K. Liaw, University of Tennessee; Masahiro Endo, Fukuoka University

Tuesday PM — Room: 216
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: David L. Mcdowell, Georgia Institute of Technology; Johannes Weertman, Northwestern University

2:00 PM Invited
Corrosion Fatigue Crack Initiation and Propagation Process of 12 PCT Chromium Stainless Steel: *Ryuichiro Ebara*[1]; [1]Kagawa University

Corrosion fatigue failure of steam turbine blades has been a big concern in the world for more than these thirty years. In this paper characteristics of corrosion fatigue failures of steam turbine blades learned from failure analysis are summarized. Then influencing variables on corrosion fatigue crack initiation and propagation process of 12 pct chromium stainless steel are briefly reviewed. The emphasis is focused upon initiation and propagation of corrosion pit in corrosion fatigue crack initiation pro-

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cess. A recent investigation of early stage of corrosion pit initiation process by use of electrochemical noise method is demonstrated. Finally future problems should be solved in corrosion fatigue crack initiation and propagation process of 12 pct chromium stainless steel.

2:25 PM
Creep-Fatigue-Environment Interactions in INCONEL 617: *Terry Craig Totemeier*[1]; Hongbo Tian[1]; [1]Idaho National Laboratory

Wrought nickel-base alloys are being considered for use in very-high temperature gas-cooled nuclear reactors as piping, heat exchangers, and reactor internals, with operating temperatures in the range of 850-950°C. Creep-fatigue damage will play an important role in determining component lifetimes. The results of initial work on clarifying the separate and synergistic roles of creep, fatigue, and environment in INCONEL alloy 617 are presented. Creep-fatigue tests have been performed at 1000°C and 800°C; the effect of tensile hold time on cycles to crack initiation and failure was determined for total strain ranges of 1.0 and 0.3% at both temperatures. Tests were performed in air and a purified He inert gas environment.

2:50 PM
Fatigue Behavior of a C-2000 Superalloy with a Nanostructured Surface Layer: *J. W. Tian*[1]; K. Dai[2]; D. Fielden[1]; D. L. Klarstrom[3]; J. C. Villegas[2]; L. L. Shaw[2]; P. K. Liaw[1]; [1]University of Tennessee; [2]University of Connecticut; [3]Haynes International, Inc.

A series of C-2000 superalloy samples were treated with different times and/or ball sizes using a newly developed surface nano-crystallization and hardening (SNH) process. After the treatment, a nanostructured surface layer with thicknesses of several tens of microns was found, and four-point-bending fatigue tests were conducted. The results show that a nanolayer with moderate thickness can effectively improve the fatigue limit, while with the further increase of the thickness of the nanolayer, the fatigue behavior was severely deteriorated. Therefore, nanostructured surface layers do not necessarily improve the fatigue properties of this material. A possible mechanism involved with the competitive relations among the residual stresses, nanograin strengthening, and surface defects is proposed to illustrate the effect of the nanostructured surface layer on the fatigue behavior. The present work is supported by the National Science Foundation (NSF) with the grant number of DMR-0207729.

3:15 PM
High-Cycle Fatigue of a Nickel-Base Single-Crystal Superalloy: *Jianzhang Yi*[1]; Chris J. Torbet[1]; Tresa M. Pollock[1]; J. Wayne Jones[1]; [1]University of Michigan

The fatigue behavior of a second-generation nickel-base superalloy with a platinum aluminide coating was investigated using an ultrasonic fatigue testing system, operating at a frequency of approximately 20kHz. Single crystals were stressed along the <001> orientation with a stress ratio of 0.2 at a temperature of 982°C (1800°F) up to 10^9 cycles. For the testing conditions investigated, multiple types of initiation sites were observed: the Pt/Al coating layer, solidification porosity and Ta-rich carbides within inter-dendritic region. Cracks initiating at or near specimen surfaces propagated macroscopically along the (001) plane with the crack tip influenced by oxidation, creep and/or cyclic deformation processes. Conversely, cracks initiating from interior carbides or porosity tended to propagate along (111) planes.

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3:55 PM Invited
Effects of Microstructure on Dwell Fatigue Crack Growth in Ti-6242: *Winston O. Soboyejo*[1]; [1]Princeton University

This paper present s the results of a combined experimental and mechanistic modeling approach to the study of dwell fatigue in Ti-6242. Crack shape evolution, depth and surface crack growth rates are established using beachmarking, acoustic emission and scanning electron microscopy (SEM) techniques. The differences between the dwell crack growth rates and pure fatigue crack growth rates in the short regime are attributed to possible creep effects that give rise to a mean stress estimation of fatigue life in the three microstructures. The implication of the predictions are discussed for the modeling of dwell fatigue.

4:20 PM
The Effect of Porosity on the HCF of a Model Carbon-Containing Ni-Base Superalloy: *Elyssa R. Cutler*[1]; Gerhard E. Fuchs[1]; Adam W. Haskins[1]; [1]University of Florida

A carbon-containing single crystal Ni-base superalloy was tested in high cycle fatigue (HCF) in a hot isostatically pressed (HIPed) and unHIPed condition. Eighty samples were tested at five carbon levels and four conditions. The HIPing reduced porosity significantly and greatly improved creep properties. However, the fatigue lives of the HIPed alloys were not improved and in most cases, decreased. Failure in the unHIPed samples was initiated by internal defects such as porosity and carbides, while most HIPed samples failed due to persistent slip bands. To separate the effects of carbide morphology and gamma prime size, a third and fourth set were given a 1000°C/1000hr thermal exposure, with the fourth set being re-HIPed and heat treated. As expected, the third set showed decreased fatigue lives due to overaging. The fourth set had improved fatigue lives, but not nearthe order of magnitude expected.

4:45 PM
Influence of Cyclic Loading on the Residual Stress Profile in High Strength Steel Wires: *Jesús Toribio*[1]; Miguel Lorenzo[1]; Diego Vergara[1]; [1]University of Salamanca

This papers deals with the influence of cyclic (fatigue) loading on the residual stress profile in high strength steel wires. To this end, wires with several residual stress profiles (of tensile and compressive nature) were analysed, and different sinusoidal loads with diverse values of maximum loading level and number of cycles were applied on the wires. The evolution of the residual stress profile was numerically evaluated throughout the loading sequence, in particular at the instants of maximum and minimum load during the fatigue cycle. Results of the simulations showed no significant variation with the fatigue loading sequence of the key parameters of the residual stress profile, neither its original shape nor the boundary values at the external surface of the wire. Thus fatigue loading does not affect the residual stress profile in high-strength steel wires (introduced into the material during the manufacturing process by wire drawing through different dies).

Fatigue and Fracture of Traditional and Advanced Materials: A Symposium in Honor of Art McEvily's 80th Birthday: Fatigue and Fracture VI

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Leon L. Shaw, University of Connecticut; James M. Larsen, U.S. Air Force; Peter K. Liaw, University of Tennessee; Masahiro Endo, Fukuoka University

Tuesday PM — Room: 215
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Richard P. Gangloff, University of Virginia; James C. Williams, Ohio State University

2:00 PM Invited
Effect of Material Microstructure on Fatigue Behavior of AZ31 Magnesium Alloy: *Sotomi Ishihara*[1]; Zhenyu Nan[1]; Takahito Goshima[1]; [1]University of Toyama

Fatigue experiments were carried out using an extruded magnesium alloy, AZ31. In the alloy, a lamellar structure which is parallel to the extruded direction exists. Two kinds of specimens with axial directions, parallel (EP) or tangential (ET) to the lamellar structure, were used. By comparing the S-N curves and crack generation and propagation characteristics of both specimens, effects of the lamellar-structure of the AZ31 magnesium alloy on the fatigue characteristics was studied. In the specimen EP, fatigue life was almost occupied with a fatigue propagation period. Crack retardations or arrests due to the lamella structure were observed. Therefore, a sharp bend in the S-N curve was observed. In the specimen ET, many cracks were generated and propagated as compared to the specimen, EP. The rate of fatigue crack growth in the specimen, ET was faster than in the specimen, EP and led reduction in fatigue lives of the former.

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Effect of Porosity and Frequency on Very Long Life Fatigue of Cast Aluminum Alloys: *Xiaoxia Zhu*[1]; Jianzhang Yi[1]; John E. Allison[2]; J. Wayne Jones[1]; [1]University of Michigan; [2]Ford Motor Company

The fatigue behavior of a cast Al-Si-Cu alloy was investigated for lifetimes up to 10^8 cycles using ultrasonic fatigue instrumentation at 20 kHz and at lifetimes up to 10^7 cycles using conventional fatigue testing at 30 Hz. Fatigue cracks predominantly initiate from microshrinkage pores at or near the specimen surface. The growth rates of fatigue cracks was measured at 20 kHz and at 30 Hz. Fatigue lifetime was accurately predicted, even in the gigacycle regime, by a model that assumed lifetime to be dominated by crack growth from porosity, and neglecting crack initiation. Porosity size and spatial distribution has been quantified and correlated with fatigue life and the fatigue limit. A comparison of the crack growth behavior at ultrasonic and conventional frequencies and the implications for the use of ultrasonic fatigue in examining the behavior of very long life fatigue in cast aluminum alloys will be described.

2:50 PM
Fatigue Crack Growth in Al-Si-Mg Cast Alloys: Microstructural and Processing Considerations: *Diana A. Lados*[1]; Diran Apelian[1]; Peggy E. Jones[2]; Fred J. Major[3]; [1]Worcester Polytechnic Institute; [2]General Motors Corporation; [3]Alcan International Ltd.

The fatigue crack growth behavior of long and small cracks was investigated for hypoeutectic and eutectic Al-Si-Mg cast alloys. Microstructure related mechanisms were used to explain the near-threshold behavior and the crack growth response in Regions II and III for alloys with different Si composition/morphology, grain size level, and matrix strength. The effect of SDAS was qualitatively assessed using the developed knowledge. The thresholds of long cracks reflect combined effects of global residual stress induced and microstructure/roughness induced closure. The threshold behavior of small cracks is explained through closure unrelated mechanisms, specifically through the barrier effects of characteristic microstructural features specific to each alloy. In Regions II and III the observed changes in the fracture surface appearance were associated to changes in crack growth mechanisms at the microstructure scale. The extent of the plastic zone ahead of the crack tip was successfully used to explain the changes in growth mechanisms.

3:15 PM Invited
Analysis of the Effect of the Cold-Working of Rivet Holes on the Fatigue Life of an Aluminum Alloy: *Paulo T. de Castro*[1]; Paulo F. P. de Matos[1]; Pedro M. G. P. Moreira[1]; [1]Universidade do Porto

The understanding of fatigue crack propagation requires the link between the micro and macro structural problem. Results of quantitative microfractography are presented to quantify the crack growth rate in a detail of an aircraft structure. The structural detail under analysis is represented by open-hole specimens in Al-alloy 2024-T3, with and without residual stresses due to hole expansion. The residual stress field created by the cold expansion was experimentally assessed by using the X-ray technique and predicted by FEA. Fatigue tests at several stress levels were supplemented by fatigue striation spacing measurements along the longitudinal crack surface of each specimen. The rates of fatigue crack propagation were deduced from a determination of striation spacing measured by scanning electron microscopy. The approach proposed by McEvily et al. was used to analyze the results. A novel feature of the present analysis was the use of ΔK dependent, plasticity-induced crack closure.

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3:55 PM
Analysis of Fatigue Resistance Improvements via Surface Nanocrystallization and Hardening Process: Kun Dai[1]; Jiawan Tian[2]; Juan C. Villegas[1]; *Leon L. Shaw*[1]; Peter K. Liaw[2]; Dwaine Leroy Klarstrom[3]; [1]University of Connecticut; [2]University of Tennessee; [3]Haynes International Inc

Numerical simulations have been performed to analyze the work hardening and residual compressive stresses induced by the surface nanocrystallization and hardening (SNH) process. The contributions of work hardening and nano-grains at the surface region are separated from that of residual compressive stresses. The study suggests that work hardening and nanoscale grains at the surface layer play a significant role in enhancing the fatigue resistance of a nickel-based alloy achieved via the SNH treatment. In comparison, less contribution is provided by residual compressive stresses.

4:20 PM
Effects of Shot-Peening and Re-Shot-Peening on Four-Point Bend Fatigue Behavior of Ti-6Al-4V: *Xiuping Jiang*[1]; Jinxia Li[1]; Chi-Sing Man[1]; Michael J. Shepard[2]; Tongguang Zhai[1]; [1]University of Kentucky; [2]U.S. Air Force

The effects of shot peening and re-shot peening treatment on four-point bend fatigue strength of Ti-6Al-4V were investigated at room temperature and 150°C. The maximum compressive residual stress produced by shot peening was measured by XRD to be around 800 MPa, which improved the fatigue strength from about 65%sy to 71%sy. A step-test method was applied to determine the fatigue strength of the shot-peened and re-peened specimens at different conditions. Re-peening recovered the residual stress, which was relaxed due to bending fatigue at 150°C, and significantly enhanced the fatigue strength both at room temperature and 150ºC. Intergranular fracture was also observed in the crack initiation region of shot-peened and re-peened samples.

4:45 PM
Cyclic Plastic Deformation of GTA Welds in Titanium Alloys: *Shing-Hoa Wang*[1]; Ming-De Wei[1]; [1]National Taiwan Ocean University

The effects of strain amplitude and strain rate on the low cycle fatigue are investigated in three kinds of aerospace applied Ti alloys and their welds. Sheets of commercially pure titanium (CP-Ti), alpha + beta titanium alloy (Ti-6Al-4V, Ti-64) and beta titanium alloy (Ti-15V-3Al-3Cr-3Sn, Ti-153) in 0.3 mm thick were melted using the TIG welding process. The microstructures of the fusion zones for CP-Ti and Ti-64 consisted of needle-like martensite with maximum hardness. LCF tests of the received titanium alloys and welds were performed. The fatigue strength increased with the strain rate. The strength of Ti-153 β phase titanium alloy demonstrated more sensitivity to strain rate changes than the CP-Ti and Ti-64 alloys. The fatigue strength of Ti-153 welds was slightly superior to that for the received metal, even though it failed in the fusion zone. The fatigue strengths of CP-Ti and Ti-64 welds were inferior to that for the received metal.

General Abstracts: Materials Processing and Manufacturing Division: Surface Modification and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee, TMS: Global Innovations Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Thomas R. Bieler, Michigan State University; Ralph E. Napolitano, Iowa State University; Fernand D. Marquis, South Dakota School of Mines and Technology

Tuesday PM — Room: 211
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: D. Graham McCartney, University of Nottingham

2:00 PM
Interaction of Nanofluids with Heat Transfer Surface: *Satyanarayana Kuchibhatla*[1]; Denitsa Milanova[1]; Swanand D. Patil[1]; Keith E. Rea[1]; Sameer A. Deshpande[1]; Sudipta Seal[1]; Ranganathan Kumar[1]; [1]University of Central Florida

Nanofluids are a new category of heat transfer fluids, where nano particles are dispersed in a conventional heat transfer fluid, imparting very

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high heat transfer rates. One major aspect that is less focused is the interaction of these nano dispersions with heat transfer surfaces that interact with nanofluids. Pool boiling experiments were carried in Ceria, silica and alumina nano fluids with NiCr wire. The NiCr wires, before and after the experiments, were characterized with Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) and Energy Dispersive Spectroscopy (EDS) measurements have clearly shown that the oxide formation is relatively low in the presence of ceria, alumina when compared to the conventional fluid and also the nanofluid with silica. Various possible reasons for the differences in interaction of the other nanoparticles with the heat transfer surface and their role in the over all nanofluid performance shall be discussed critically.

2:20 PM
Investigating Functionally Graded Materials to Improve the High Temperature Operating Characteristics of Industrial Tools and Dies and Processing Equipment: *Sudip Bhattacharya*[1]; Jerrod A. Roalstad[1]; Aaron C. Costello[1]; Stanley M. Howard[1]; James W. Sears[1]; [1]South Dakota School of Mines and Technology

The goal of this ongoing investigation is to obtain a five times improvement in the service life of industrial tools and dies by laser depositing functionally graded materials on their working surfaces. Ni and Co based superalloys like Inconel® and Stellite® and conventional alloys like NiTung60, DM21 and CCW+ have been deposited onto H13 surfaces and actual tools and dies. The initial work on hot forging tools clad with NiTung60 has shown slight improvement in service life while those clad with CCW+ have almost doubled. Alloys similar to AeroMet 100 and Co-WC are also being evaluated. However, it is also felt that cladding refractory metal alloys onto tool surfaces may be necessary to achieve the stated program goals. Therefore in a parallel effort, pairs of metals such as; H-13/Cr, H-13/Ni, Ni/Cr, Cr/V, Cr/Nb, V/Ta, Nb/Ta and Nb/W are being investigated.

2:40 PM
Load and Friction Issues in Sliding Wall ECAE Tooling: *Robert E. Barber*[1]; Karl Ted Hartwig[1]; [1]Shear Form, Inc.

Load influencing factors and friction issues in sliding wall equal channel angular extrusion tooling are discussed. Analysis of the load curves produced while extruding several different materials and billet sizes shows that billet upsetting, starting material texture, lubrication efficiency and billet surface to volume ratio all influence extrusion force and load efficiency. The benefits of a sliding-wall tool design are reduction of the coefficient of friction between all sliding surfaces, the possibility of variable backpressure, and punch face stress reduction. These benefits translate into the ability to extrude longer billets at lower loads. Friction forces in sliding wall tooling can be sufficiently low so as to allow discrimination of the main factors that influence punch load including material flow stress and texture, lubrication efficiency and billet geometry.

3:00 PM
Integrity of Spark Eroded Surfaces: *Laxminarayana Pappula*[1]; Ramesh Nagabhushana Nunna[1]; Sudhakar Kasoju[1]; [1]Osmania University

Electrodischarge machining involves material erosion by high frequency electrical sparks. The tool and work piece form a pair of electrodes separated by a small gap through which a dielectric liquid circulates to facilitate sparking, quenching of eroded metal in liquid form and flushing away the resultant debris. The associated thermal cycle is quite severe which results in considerable metallurgical changes involving high inducement of tensile residual stresses, microhardness and interelectrode material diffusion. This study presents the differing nature and severity of these effects with different process parameters, particularly spark energies, type of dielectrics and type of workmaterial. Spark energy depends on the current and pulse durations. The dielectrics selected are kerosene and distilled water whereas the work materials employed are stainless steel and aluminium. Not only they have significant influence by themselves but also in association with each other exhibiting both direct and interaction effects.

3:20 PM
Thermal Stability of Various Alloys Clad on H-13 Tool Steel by Laser Powder Deposition: *Jerrod A. Roalstad*[1]; Sudip Bhattacharya[1]; Stanley M. Howard[1]; James W. Sears[1]; [1]South Dakota School of Mines and Technology

Thermal stability investigations were performed under research to improve high temperature wear resistance of industrial tools and dies. The commercial alloys CCW+®, DM21®, and NiTun60®were deposited onto H13 tool steel using a 3-KW Nd:YAG laser. The samples were heat treated at 1200ºC for 1, 10, and 100 hours. Optical microscopy was performed to identify if any phase transformation had taken place during the heat treatment. DM21® samples revealed precipitate growth within the clad. SEM analysis showed the composition of the precipitates observed within the clad to contain Titanium, Tantalum, and Tungsten. Compositions of the precipitates were compared with published phase diagrams to identify the possible phases. Dictra® computational software was used to theoretically determine the extent of the growth of the phases present. Good bonding was observed for all deposited materials.

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4:00 PM
Microstructure and Properties of Thermally Sprayed Al-Sn Based Alloys for Automotive Journal Bearings: *D. Graham McCartney*[1]; Tiziana Marrocco[1]; Samuel J. Harris[1]; [1]University of Nottingham

Al-Sn automotive journal bearings traditionally comprise a multilayer structure manufactured by casting the Al-Sn alloy and roll-bonding to steel. Recently, high velocity oxy-fuel (HVOF) thermal spraying has been employed as a novel manufacturing route to produce coatings, approx 250 µm thick, on steel substrates which are subsequently annealed to achieve the desired microstructure. The present research extends work on ternary Al-Sn-Cu alloys to quaternary systems with specific additions for potentially enhanced properties. Al-20wt.%Sn-1wt.%Cu-2wt.%Ni and Al-20wt.%Sn-1wt.%Cu-7wt.%Si alloys were studied and we will describe the microstructure formation and wear behaviour of the deposits. Starting powders and coatings were investigated by scanning electron microscopy and X-ray diffraction. Deposit microhardness was measured in both as-sprayed and heat treated conditions, and the wear behaviour in hot engine oil determined using an industry standard test. The results will be interpreted in terms of non-equilibrium microstructure formation and the influence of key microstructural features on mechanical behaviour.

4:20 PM
Improvement of Corrosion Resistance of Plain Carbon Steel Bars by Friction Induced Surface Coating with Aluminum: *Pravash Chandra Maity*[1]; B. RaviKumar[1]; [1]National Institute of Foundry and Forge Technology

A novel technique of surface modification of plain carbon steel by Rotational Pressure Friction (RPF) has been developed to improve its corrosion resistance. The technique involved movement of a pressurized cylindrical alloying tool (Al) over rotating round steel substrate. Rpm of steel specimen, composition of the coating alloy, feed of coating alloy over rotating specimen and no. of passes over 25 mm dia., 75 mm long specimens. 1 Mpa pressure was used in all the experiments. Selected uniformly coated samples were tested for corrosion resistance using potentiodynamic polarization technique (ASTM G5) with 3.5 % NaCl solution as a corrosive medium. Coated steel specimens showed substantial improvement in corrosion resistance from 5.37 mpy for uncoated steel to as low as 0.125 to 0.30 mpy for coated steel specimens.

General Abstracts: Structural Materials Division: Processing and Properties of Light Metals

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizers: Rollie E. Dutton, U.S. Air Force; Ellen K. Cerreta, Los Alamos National Laboratory; Dennis M. Dimiduk, U.S. Air Force

Tuesday PM — Room: 218
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Rollie E. Dutton, U.S. Air Force

2:00 PM
A Group of New Ti-Based Composite Alloys: *Faqiang Guo*[1]; Joe Poon[1]; Gary Shiflet[1]; [1]University of Virginia

New Ti-based composite alloys with amorphous phases plus a/b Ti(Zr) solid solution have been discovered, which show superior mechanical properties. Both compression and tension tests of the prepared Ti alloys exhibit a 1.6 % of elastic strain limit. A plastic deformation of around 10% was obtained from compression test while a plastic elongation of ~ 4% was found in the tension test experiment. The Ti composite alloys yield at about 1200 MPa, showing steady work-hardening during deformation, and finally fracture at a strength of about 1700 MPa. These Ti composite alloys have already been ready to be used as structural materials in terms of mechanical properties.

2:25 PM
Dynamics of Twinning in Beta-Titanium Alloys: *Paul G. Oberson*[1]; Sreeramamurthy Ankem[1]; [1]University of Maryland

Recently it has been shown that in the beta-Ti-14.8 wt.%V alloy, twins of the type {332}<113> form and grow slowly at a stress level of 95% yield stress at room temperature. In this investigation a crystallographic model has been developed for the slow-growth of twins. It is shown that the octahedral interstitial sites at the twin-matrix interface, where oxygen resides, are not conserved. The measured activation energy for twin-growth is compared with the activation energy for the diffusion of oxygen in this material and the correlation was very good. The ramification of these findings and the details of the investigation will be presented. This work is being supported by the National Science Foundation under grant number DMR-0513751.

2:50 PM
Friction and Electronic Properties of Al-Based Complex Phases: *Jean-Marie Dubois*[1]; [1]CNRS Institut Jean Lamour

A throughout study of many Al-based intermetallics, including compounds with complex (or giant) unit cells and quasicrystals, was performed using a pin-on-disk tribology set-up housed in a vacuum chamber. The riding counterpart was a hard-steel ball, loaded with a 2N force. Our data show a direct relationship between friction observed in such conditions and the filling of the electronic bands at the Fermi energy, with a small, if not negligible, contribution of the plowing component to friction. The case will be exemplified, especially using data obtained with a decagonal, anisotropic quasicrystal. Furthermore, after proper calibration of the experiment, an estimate of the upper limit of the surface energy of the samples may be derived. These measurements are assessed in the light of partial electronic densities of states, with the view that chemical bonding at the pin-sample interface dominates friction in vacuum on such Al-based compounds.

3:15 PM
Hydrogen Diffusion in Ti-45 Nb and ATI-425: Brian James Sarrail[1]; Shake Babakhanyan[1]; Charles Schrupp[1]; Richard Clark[1]; Omar Es-Said[1]; *John Orgen*[1]; [1]Loyola Marymount University

The objective of this project was to determine the conditions needed to "contaminate" two alloys, Ti-45 Nb and ATI-425, with known and uniform levels of hydrogen. Alloying Ti-based alloys with hydrogen is used to modify the microstructure and improve mechanical properties. The design of Ti-based alloys can be made more efficient through the use of hydrogen as a temporary alloying element since both manufacturing temperatures and stresses can be decreased. The uniform target hydrogen concentration levels are 50, 100, 200, 300, 400 and 500 ppm. Samples of both alloys were annealed in a hydrogen furnace at 12% hydrogen with a 5 psi chamber pressure for 1, 2, and 4 hours at 450°C and 600°C. The concentration profiles were then determined.

3:40 PM Break

3:55 PM
Mechanical Properties of Ti-6Al-4V Castings for Ground Vehicle Applications: *Ibrahim Ucok*[1]; Mustafa Guclu[1]; Hao Dong[1]; Joseph R. Pickens[1]; [1]Concurrent Technologies Corporation

There is a strong interest in reducing weight of combat vehicles because of an increased need for air transportability and reduced logistical support. Titanium alloys are candidates for ground vehicle applications because they combine high specific strength and excellent corrosion resistance. In the present study, gun pod and elevation arm castings for the Stryker - Mobile Gun System (MGS) were made by investment and rammed graphite casting methods using alloys Ti-6Al-4V and Ti-4Al-2.5V-1.5Fe at different oxygen levels. Redesigning the gun pod to exploit titanium and replace steel reduced weight by 28%. Tensile and fatigue properties, as well as the microstructures of the castings, were evaluated. 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. Approved for public release; distribution is unlimited.

4:20 PM
Property Investigation of Laser Cladded TiAl6V4: *Johannes Vlcek*[1]; Sven Orban[2]; [1]European Aeronautic Defense and Space Company; [2]IWS Dresden

Laser cladding is an interesting process technology for structural repair or overhaul of engine components. In order to evaluate the technology for structural repair or direct component manufacturing the properties under dynamic loading and fracture toughness values are of interest.Laser cladded TiAl6V4 material was produced at the Fraunhofer IWS Dresden with a 3 KW Nd:YAG laser and the IWS coaxial cladding head type COAX8. The material was tested in the as sintered and mill annealed condition after a 2 hour vacuum anneal at 850°C. Ultimate tensile strength of maximum 1100 MPa with an elongation greater than 11% was measured. The fatigue resistance can be assumed around 475 MPa, but needs further investigation. A fracture toughness value of 63 MPa m^0,5 was measured. The investigation focused on micro-structural features, the influence of vacuum annealing and fracture behaviour depending on the previous and specimen orientation.

4:45 PM
Quenching Effects on Ti-325 Alloy: Brian James Sarrail[1]; Charles Schrupp[1]; Shake Babakhanyan[1]; Richard Clark[1]; Omar Es-Said[1]; *John Orgen*[1]; [1]Loyola Marymount University

Ti-325 is a near alpha alloy with 3 wt% aluminum as an alpha stabilizer and 2.5 wt% vanadium as a beta stabilizer. This alloy is most widely used in its seamless tubing form. This study determined the effects of two different cooling procedures on the room temperature mechanical properties of the alloy. Samples were machined from a seamless tube and quenched either in water or furnace-cooled at temperatures ranging from 1170°F to 1230°F. The yield strength and ultimate strength were measured as a function of quenching medium. In addition, S-N curves were developed for this alloy.

5:10 PM
The Effect of RRA Treatment on Corrosion and Corrosion Wear Performance of 7175 Aluminum Alloy: *Ozgur Celik*[1]; Ilke Dagli[1]; Murat Baydogan[1]; Eyup Sabri Kayali[1]; Huseyin Cimenoglu[1]; [1]Istanbul Technical University

This study aims to compare the corrosion and wear responses of a 7175 alloy after conventional aging (T6 temper) and retrogression and reaging (RRA) treatments. Corrosion tests were conducted in HCl and H2O2 solutions, according to ISO 11846 and ASTM G 110 standards, respectively. Wear test were made under dry sliding and corrosion (in HCl and H2O2 solutions) wear testing conditions. Among the examined retrogression temperatures, 260°C yielded the superior corrosion resistance, when compared to T6 temper. RRA treated alloy (retrogressed at 260°C) exhibited higher dry sliding wear resistance than T6 tempered alloy. In HCl and H2O2 solutions wear resistance of the alloys dramatically decreased without leading any considerable difference in the corrosion wear resistance of T6 temper and RRA treatment. Although HCl is more aggresive than H2O2 solution, the progress of corrosion wear is more severe in H2O2 solution.

Hume Rothery Symposium: Multi-Component Alloy Thermodynamics: Alloy Thermodynamics II: Experiment and Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Y. Austin Chang, University of Wisconsin; Rainer Schmid-Fetzer, Clausthal University of Technology; Patrice E. A. Turchi, Lawrence Livermore National Laboratory

Tuesday PM	Room: 202A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Adolf Mikula, University of Vienna

2:00 PM Invited
Experimental Thermodynamic Methods to Determine Partial Properties: Examples: *Herbert Ipser*[1]; Klaus Richter[1]; [1]University of Wien

Among the various experimental methods to determine partial thermodynamic properties, there are two main groups: those depending on the measurement of electromotive forces (emf), and those that determine the vapor pressure of one or several components, in both cases as a function of composition and/or temperature. Whereas the value of emf or vapor pressure at a given temperature yields the partial Gibbs energy (thermodynamic activity) itself, its temperature dependence can be used to derive the partial enthalpy and entropy. Two of these methods will be presented here with various examples. The first are emf measurements with solid oxygen conducting electrolytes that were used to determine partial thermodynamic properties of indium in Pt_3In and in ternary Ag-In-Pd alloys. The second are isopiestic vapor pressure measurements that served to determine partial thermodynamic properties of zinc in solid Pt-Zn alloys and partial thermodynamic properties of antimony in ternary Fe-Ni-Sb and In-Ni-Sb alloys.

2:30 PM Invited
Integrating First-Principles Calculations and Thermodynamic Modelling: *Zi-Kui Liu*[1]; Stefano Curtarolo[2]; Aleksey Kolmogorov[2]; Raymundo Arroyave[1]; Dongwon Shin[1]; [1]Pennsylvania State University; [2]Duke University

Results from first-principle calculations are becoming more and more important in thermodynamics modeling of multi-component systems. Enthalpies of formation of stable compounds are routinely predicted by first-principles calculations. However, calculations are more difficult for phases with homogeneity ranges of compositions, which are typically represented by sublattice models. In this presentation, we will discuss following challenging issues: 1. Lattice stability, i.e. the energy difference between two structures of an element; 2. Stability issues related to the end-members in sublattice models; 3. Application of special quasirandom structures for solution phases; 4. Contribution of vibrational entropy on structure stability.

3:00 PM Invited
Potential of Knudsen Effusion Mass Spectrometry for Multi-Component Alloy Thermodynamics: *Klaus Hilpert*[1]; [1]Research Centre Julich

Knudsen effusion mass spectrometry (KEMS) is the most versatile method for the analysis of high temperature equilibrium vapors. It offers the following potential: •Vaporisation studies up to temperatures above 2500 K; •Identification of the gaseous species; •Determination of their partial pressures in the range 10 to 10-5 Pa; •Evaluation of thermodynamic data. This potential can be efficiently used in the determination of thermodynamic data of the condensed phase and the gas phase. For the condensed phase thermodynamic activities and Gibbs energies of formation can be determined with high accuracy and precision as primary data. Moreover, partial and integral enthalpies of formation can be obtained. For the gas phase numerous intermetallic molecules can be identified and their Gibbs energies and enthalpies of formation determined. The fundamentals of modern KEMS will be given and recent results on alloy thermodynamics and gas phase chemistry will be reported.

3:30 PM Break

3:50 PM Invited
Thermodynamics of Roses: *Marius Stan*[1]; [1]Los Alamos National Laboratory

In one of his talks, Alan Oates presented a calculated ternary equilibrium phase diagram that looked much like a rose. He explained that he did that to demonstrate the versatility of the software and to show how, in principle, one can get any diagram they want. This presentation reviews results, benefits, and challenges of making thermodynamics a less descriptive and more predictive science. It builds upon the work of Hume-Rothery and Alan Oates and emphasizes the importance of turning electronic structure calculations into trusted sources of information for thermodynamic models and simulations. The proposed multi-scale and multi-physics approach is illustrated with calculations of equilibrium phase diagrams.

4:20 PM Invited
Thermodynamics of Pd Alloy-H Systems: *Ted B. Flanagan*[1]; Suifang Luo[1]; [1]University of Vermont

The thermodynamics of Pd binary alloy-H systems have been investigated by many workers using mainly equilibrium H pressure measurements which give the relative H chemical potentials. From the T-dependence of these potentials, the partial enthalpies and entropies can be obtained as a function of H content. Alternatively, these quantities can be obtained by simultaneous measurements of enthalpies from calorimetry and equilibrium pressures as function of H contents. The latter gives the partial entropies from the enthalpies and relative H chemical potentials. In this research such calorimetry has been carried out at a moderate temperature where many of the alloys form hydride phases. Some binary Pd alloys which have been investigated are: Pd-Ag(-Au, -Cu, -Sn, -Fe, -Mn).

4:50 PM Invited
Thermodynamic Interaction between Pt, Ni and Al in β-NiAl, γ'-Ni3Al and β-Ni(Al) in the Ni-Al-Pt-O System: *Evan H. Copland*[1]; [1]NASA Glenn Research Center

The addition of Pt to Ni-Al alloys is generally seen to improve oxidation behavior but little is understood about the reasons for this improvement. Determining how Pt affects the thermodynamic activity of Al and Ni in β-NiAl, γ'-Ni3Al and γ-Ni(Al) is fundamental to understanding this behavior. As part an investigation into the solution behavior of the Ni-Al-Pt-O system the activities of Ni, Al, Al2O, O and Al2O3 have been measured directly by the vapor pressure technique with a multiple effusion-cell vapor source coupled to mass spectrometer (multi-cell KEMS). Measurements were made over a wide range of compositions and focused on the {β-NiAl + Al2O3(s)}, {β-NiAl + γ'-Ni3Al + Al2O3(s)} and {γ'-Ni3Al + γ-Ni(Al) + Al2O3(s)} phase fields with increasing Pt content. These results are discussed together with a series of measurements made in the Au, Al-O and Ni-Al-O systems that are used as reference states for activity measurements.

Lead Free Solder Implementation: Reliability, Alloy Development, and New Technology: Microstructure Evolution

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Nikhilesh Chawla, Arizona State University; Srinivas Chada, Medtronic; Sung K. Kang, IBM Corporation; Kwang-Lung Lin, National Cheng Kung University; James Lucas, Michigan State University; Laura J. Turbini, University of Toronto

Tuesday PM — Room: 214A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jin Yu, MSE Korea Advanced Institute of Science and Technology; Iver E. Anderson, Iowa State University

2:00 PM Invited
Near-Single Crystal Character of Sn-3.5Ag Eutectic Solder Joint Specimens: *Thomas R. Bieler*[1]; Telang Adwait[2]; Sven Vogel[3]; [1]Michigan State University; [2]Intel Corporation; [3]Los Alamos National Laboratory

Tin-based solder joints are frequently near single or multi-crystalline rather than polycrystalline, as determined by comparing orientations measured using OIM on opposite sides of two solder joints and by volumetric measurements of crystallographic texture using neutrons (HIPPO beam line). Comparisons among more than 20 joints indicated no preferred solidification orientation, though there is a tendency for tin <110] vector to be roughly aligned with the heat flow (crystal growth) direction, but any rotation about this axis leads to a wide range of observed preferred orientations. This implies that the properties of all lead free solder joints may be different, implying that one of the two joints will deform preferentially in service.

2:25 PM
Effect of Reflow and Thermal Aging on Microstructure Evolution and Microhardness of Sn-3.7Ag-xBi Solder Alloys: *Min He*[1]; Viola L. Acoff[1]; [1]University of Alabama

This work investigates effect of reflow and thermal aging process on microstructure evolution and microhardness of five types of Sn-Ag based lead-free solder alloys: Sn-3.7Ag, Sn-3.7Ag-1Bi, Sn-3.7Ag-2Bi, Sn-3.7Ag-3Bi, and Sn-3.7Ag-4Bi. Microhardness and microstructure of the solders under different reflow time at 250°C and different thermal aging durations at 150°C have been studied. The effect of Bi is discussed based on the experimental results. It was found that the microhardness increases with increasing Bi addition to Sn-3.7Ag solder regardless of reflow or thermal aging process. SEM images show the formation of Ag3Sn particles, Sn-rich phase and precipitation of Bi-rich phase in different solders. The increase of microhardness with Bi addition is due to the solution strengthening and precipitation strengthening provided by Bi in the solder. The trend of decrease in microhardness with the extension of reflow time and thermal aging duration for the same solder alloy was observed.

2:45 PM
Flux Effect on Wetting Behavior of SnZnAl Lead-Free Solder Balls: *Cho-Liang Chung*[1]; Haoyin Tsai[2]; Alex Lu[2]; De-Shin Liu[3]; S. L. Fu[3]; [1]I-Shou University; [2]ChipMOS Techologies Inc.; [3]National Chung Cheng University

Effects of two different fluxes (A6 and B6) on wetting performance of Sn-37Pb, Sn-3.5Ag-0.5Cu and Sn-7.0Zn-100ppmAl lead-free solder balls were investigated during melting process. Solder ball wetting behavior in-real time via an optical microscope coupled with a video recorder during the reflow process was studied. The lead-free solder balls started to melt and wet below normal melting point by using A6. Wetting performance of the lead-free solder ball was dramatically enhanced by using A6. The wettability test indicated that the height of the solder ball after the reflow process with flux A6 was significantly lower than that with B6. It was also observed that strong fluxing capability caused these phenomena.

3:05 PM
Interaction of Pb Free Solder Alloys and Package Pad Finish on Drop/Impact Reliability of CSP Packages: *Ahmer R. Syed*[1]; Tae Seong Kim[1]; Jae Hyeon Shin[1]; Sang Hyun Ryu[1]; Min Yoo[1]; [1]Amkor Technology

Both Sn4.0Ag0.5Cu and Sn3.0Ag0.5Cu solder do not perform as good as SnPb solder under drop/impact loading experienced by CSP packages in handheld electronic applications. This study focuses on finding alternate Pb free solder alloys which can perform similar or better than SnPb solder under board level drop testing. Eight (8) SnAgCu based alloys were considered for this evaluation using both NiAu and Cu OSP surface finish on package ball pads. The alloys included various versions on ternary SnAgCu as well as quatenary alloys with the addition of Co, Ni, and Sb in basic SnAgCu system. Intermetallic analysis and package and board level tests were performed for each solder alloy-pad finish combination. The results show that changes in SnAgCu composition and additional elements in SnAgCu based solder can significantly improve the drop performance, primarily because of differences in IMC formation and the strength of solder alloys.

3:25 PM
A Nano-Structured Approach in Lead-Free Electronic Solders: *Andre Lee*[1]; K. N. Subramanian[1]; [1]Michigan State University

The nano-structured materials technology based on polyhedral oligomeric silsesquioxanes (POSS), with appropriate organic groups, can produce suitable means to promote bonding between nano-reinforcements and the metallic matrix. The microstructures of lead-free solder reinforced with surface-active POSS tri-silanols were evaluated using SEM technique. Steady-state deformation of solder joints made of eutectic Sn-Ag solder containing varying amounts of of POSS of different chemical moieties were evaluated at different temperatures. Mechanical properties such as shear stress versus simple shear-strain relationships, peak shear stress as a function of rate of simple shear-strain and testing temperature for such nano-composite solders are reported. The service reliability of joints made with these newly formulated nano-composite solders was evaluated using a realistic thermomechanical fatigue test profile. Evolution of microstructures and residual mechanical property after different extents of TMF cycles were evaluated and compared with joints made of un-modified eutectic Sn-Ag solder.

3:45 PM Break

4:00 PM
Synchrotron Radiation X-Ray Microdiffraction Study on Orientation Distribution of Cu_6Sn_5 Intermetallic Compound Scallops in Solder Joint: *Jong-Ook Suh*[1]; Albert T. Wu[1]; King-Ning Tu[1]; Andriy M. Gusak[2]; [1]University of California, Los Angeles; [2]Cherkasy National University

Formation and growth of intermetallic compound layer in solder joint is one of the most fundamental issues in solder joint reactions. While intermetallic compound formation provides adhesion in the joint between the solder and the metal, growth of the intermetallic compound layer by solder wetting reaction is also responsible for spalling when the metal is a thin film. Final thickness of the intermetallic compound layer affects mechanical strength and thermal fatigue reliability of the solder joint, especially for solder joints with very a fine pitch. The intermetallic compound growth is a unique interfacial parasitic reaction because each intermetallic compound scallop undergoes non-conservative ripening during the growth. In this talk, we present experimental results of morphological transition and size distribution of intermetallic compound scallops, followed by orientation distribution of the scallops characterized by synchrotron radiation x-ray microdiffraction. Comparison to theoretical model of flux-driven ripening will be made.

4:20 PM
Ternary Additions in the Ag-Sn System: Determination of Phase Equilibria: Evgueni Dobrev[1]; Gueorgui P. Vassilev[1]; *Jean-Claude Tedenac*[1]; [1]Laboratoire de Physique de la Matière Condensée

Isothermal sections of the Ag-Sn-In were studied by X-ray diffraction, DSC, SEM, microhardness and metallographic studies. The calculation of phase equilibria by thermocalc sofware in the ternary have been made in order to determine the region were composition of lead free solder alloys can be defined.

4:40 PM
The Effect of Ag Content on the Formation of Ag_3Sn Platelet in Sn-Ag-Cu Lead Free Solder: *Huann-Wu Chiang*[1]; Kendi Chang[1]; Jeffrey C. B. Lee[2]; [1]I-Shou University; [2]Advanced Semiconductor Engineering, Inc.

The formation of Ag_3Sn platelete at the Sn-Ag-Cu lead-free solder joints for various Ag content solder balls will be investigated in WLCSP (wafer level chip scale package). After appropriate SMT (surface mount technology) reflow process on PCB (printed circuit board), samples will be subjected to 150°C HTS (high temperature storage) 1000 hours aging or -40 to 125°C 1000 cycles thermal cycling test(TCT). Sequentially, the cross-section analysis is scrutinized by SEM/EDX (scanning electron microscope/energy dispersive spectrometer) and to observe metallurgical evolution of the amount of the Ag_3Sn platelets in the interface and solder buck itself. Pull and shear tests will also be performed on samples. The relationship among the solder ball Ag content, the amount of Ag_3Sn platelet and the joint strength will then be analyzed and discussed.

5:00 PM
The Structural Analysis of the Immersed-Au between a Sn-Zn Based Lead Free Solder and a Substrate: *Nobuhiro Ishikawa*[1]; [1]National Institute for Materials Science

The microstructure of the interface between a Sn-Zn based Pb-free solder and a substrate by packaging of ball grid assembly (BGA) process has been studied. The several disadvantages, for example low wettability and low corrosion resistance and so on, put off the spread of the Sn-Zn based alloys. Immersion of Au on the surface of the substrate is sometimes necessary to promote the adhesion properties of soldering especially in case of using Sn-Zn based alloys. But Au forms brittle intermetallic compounds with Zn. The effects of the thickness of immersed Au have been investigated in this study. Angle lapping method was employed to analyze the Au-layer between solder and substrate three dimensionally. The cracks at the Au-layer became smaller with the thinning of the immersed Au. But they spread all directions even having thinner layer of Au and this result was found by using angle lapping method.

5:20 PM
Thermodynamic Models for the Bi-Ga-In-Sn-Zn Lead-Free System: *Raymundo Arroyave*[1]; Joel Williams[2]; Thomas W. Eagar[2]; [1]Pennsylvania State University; [2]Massachusetts Institute of Technology

In the past decade, considerable effort has been made in the development of lead-free soldering techniques due to environmental concerns. In general, the process of alloy development is costly and time consuming; however, the development process can be greatly simplified by the use of computational thermodynamics. Issues such as topology of the liquidus surface, solidification sequence, driving force for precipitation of intermetallic compounds and so forth can be addressed with this technique. In this work, we present a thermodynamic database for the Bi-Ga-In-Sn-Zn system, which is a modification of a previous model for the Bi-In-Sn-Zn quaternary system. The thermodynamic models for the ternary systems formed by the components of this quaternary system and Ga are obtained through the use of the available experimental data. Some examples on the application of this database on the characterization of alloys in this quinary system are also presented.

5:40 PM
Sedimentation of Intermetallics in Liquid Lead-Free Solders: *Jenn-Ming Song*[1]; Yu-Lin Shen[1]; Zong-Mou Wu[1]; [1]National Dong Hwa University

Since Sn-Ag-Cu and Sn-Zn alloys, which are regarded as promising lead-free solders, exhibit a lower density than the Sn-Pb solder, the sedimentation of intermetallic dispersoids may occur during multi-reflow or wave soldering processes due to gravity effect. This is usually viewed as having a harmful influence on soldering quality. This study examined microstructural evolution of intermetallics induced by alloying in isothermal liquid solders. The test materials include Sn-Ag-Cu alloys doped with small amounts of transition metals, Co, Fe or Ni, and Sn-Zn alloys with Ag or Cu. Experimental results show that the behavior of intermetallics was closely related to the temperature of the melt. Except for Cu-Zn compounds in some Sn-Zn-Cu solders, most of the heterogeneous intermetallics tended to coarsen and settle at the bottom of the molten solder when isothermally heated at 250°C. This can be ascribed to the difference in buoyancy and crystallizing temperature of the intermetallic compounds.

Magnesium Technology 2006: Microstructure and Properties II

Sponsored by: International Magnesium Association, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Alan A. Luo, General Motors Corporation; Neale R. Neelameggham, US Magnesium LLC; Randy S. Beals, DaimlerChrysler Corporation

Tuesday PM	Room: 6B
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Bob R. Powell, General Motors Corporation; Xiaoqin Zeng, Shanghai Jiao Tong University

2:00 PM
Effect of 2nd Phases on the Strength and Formability of Twin-Roll Strip Cast Mg-Zn-X Alloys: Sung S. Park[1]; Jung G. Lee[1]; *Geun T. Bae*[1]; Nack J. Kim[1]; [1]Pohang University of Science and Technology

In spite of increasing demand for light-weight vehicle, application of Mg alloy sheets in transportation systems is rather limited since few Mg alloy sheet products fulfill the strength and formability requirements of automobile industry. Recent development of twin-roll strip casting technology has shown that it can efficiently produce low cost, high performance wrought Mg alloy sheets. One of the main advantages of twin-roll strip casting is that novel microstructure can be developed due to its relatively fast solidification rate. Therefore, microstructure of recently developed twin-roll strip cast Mg alloys is usually characterized by the presence of fine second phase particles. In the present study, effect of second phase particles on the strength and formability of twin-roll strip cast Mg-Zn-X alloys has been investigated. Types of second phase particles include precipitates, primary dispersoids as well as quasicrystalle particles. Effects of alloying elements and thermo-mechanical treatment will also be discussed.

2:20 PM
Effect of Grain Size and Solute Distribution for Enhancing Strength-Ductility Balance in Wrought Magnesium Alloys: *Toshiji Mukai*[1]; Hidetoshi Somekawa[1]; Kazuhiro Hono[1]; Tetsuya Shojo[2]; Takumi Hijii[2]; [1]National Institute for Materials Science; [2]Toyota Motor Corporation

Magnesium alloys are the lightest commercial alloys and have great potential for weight reduction of automobiles to save the natural resources and to reduce the exhaust. As well as the development of the cast alloys for the power train components, production of the wrought alloys at low cost is attractive owing to the thinning of structural components. It has been demonstrated that wrought processing, i.e., extrusion or rolling, effectively enhances the strength by means of the grain refinement of materials. The effect of solute has not been, however, fully understood yet to enhance the mechanical properties in the fine-grained alloys. In this study, kinds of solute, i.e., Al, Zn, Y, etc., are investigated for the microstructure modification and enhancing the strength-ductility balance. Concentration of solute atoms inspected with high resolution TEM and EDS correlates with the grain refinement and mechanical properties in tension.

2:40 PM
Effect of Grain Size and Texture on Fracture Toughness in Magnesium and Magnesium Alloy: *Hidetoshi Somekawa*[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

For structural applications, it is important to examine the mechanical properties of magnesium materials whether satisfied with both reliability and safety or not. One of the distinction methods is the investigation of fracture toughness. However, it has been reported that the fracture toughness in magnesium materials are lower than that in commercial aluminum materials. Therefore, it needs to develop the improvement method of fracture toughness for the future applications. Recently, the grain refinement has been used to improve the mechanical properties such as strength and elongation-to-failure in magnesium materials. In addition, it has been reported that the controlling of texture is another effective method. However, these effects on facture toughness in magnesium materials have not been investigated yet. In this study, the effect of grain size and texture were discussed to the enhancement of fracture toughness in commercial pure magnesium and magnesium alloy, AZ31, which produced by sever plastic deformation.

3:00 PM
Fatigue Behavior of High-Pressure Die-Cast Magnesium Alloys: *Oren Bar-Yosef*[1]; Nir Nagar[1]; Nir Moscovitch[1]; Boris Bronfin[1]; German Gertsberg[1]; [1]Dead Sea Magnesium

Diecasting magnesium alloys offer excellent opportunities for achieving high performance, low weight, cost efficient, and fully recyclable solutions to complex engineering design challenges. Magnesium alloys that are being widely used in safety related parts and other applications are mainly subjected to complicated repetitive or fluctuating dynamic stresses at service conditions. Thus, the fatigue behavior that to large degree depends on the component soundness and particularly surface conditions is becoming one of crucial factors that may pave the way for expanding magnesium alloy applications in the automotive industry. The present paper addresses fatigue behavior of several HPDC magnesium alloys dependent on percentage of porosity and surface conditions that were varied via shot peening treatment. The effect of the above parameters on the fatigue life is discussed in terms of Goodman diagram approach.

3:20 PM
Fracture Characteristics of Die Cast Magnesium Alloy AM60B Determined from High-Resolution X-Ray Tomography under Tensile Loading: *Jon Weiler*[1]; J. T. Wood[1]; R. J. Klassen[1]; E. Maire[2]; J. Adrien[2]; R. Berkmortel[3]; G. Wang[3]; [1]University of Western Ontario; [2]INSA de Lyon; [3]Meridian Technologies

In this work, fracture characteristics of a die casting magnesium alloy AM60B sample under tensile loading were studied. A high-resolution X-ray tomography analysis was performed at four different loading levels on a 1 mm x 1mm x 11.5 mm sample cut from a die casting corrosion plate. The differences in distribution, volume, shape and fraction of pores at each loading level are analyzed. It was found that pore growth and coalescence resulted in the fracture of the AM60B sample. The data was applied to a previously reported "critical strain model" to predict the mechanical properties of the magnesium die-casting. This study indicates that mechanical properties of magnesium die castings could be potentially predicted through the unstrained X-ray tomography data.

3:40 PM
Mechanical Behavior of Wrought Mg-Zn-Y(-Mm) Alloys: *Ju Yeon Lee*[1]; Tae Eung Kim[1]; Hyun Kyu Lim[1]; Jun Hyun Han[2]; Won Tae Kim[3]; Do Hyang Kim[1]; [1]Yonsei University; [2]Korea Institute of Science and Technology; [3]Ceongju University

The room temperature mechanical behavior of Mg-Zn-Y(-Mm) alloys has been investigated. Earlier, it has been reported that the Mg-Zn-Y alloys (Mg96Zn3.4Y0.6) reinforced by icosahedral-phase particles show better mechanical properties than AZ31 alloy mainly due to strengthening effect by I-phase particles and stable I-phase/matrix interface at elevated temperature. In order to understand the mechanical behavior in more detail, the grain size of the rolled plate has been controlled in two ways: by controlling the reduction ratio during rolling and by adding alloying elements such as misch-metal (Mm). The grain size in Mg-Zn-Y alloys decreases with addition of Mm due to increased volume fraction of secondary solidification phase which accelerates the dynamic recrystallization during rolling. The mechanical properties of Mg-Zn-Y(-Mm) alloys have been investigated, and compared with those of commercial AZ31 alloys. In particular, the elongation of Mg-Zn-Y(-Mm) alloys is better than that of AZ31 alloy due to the stable I-phase/matrix interface.

4:00 PM Break

4:20 PM
Study of Rolling, Heat Treatment Characteristics and Mechanical Properties of Superlight Mg-Li-Zn Alloys: Hong Bin Li[1]; Guang Chun Yao[1]; Yi Han Liu[1]; Hai Bin Ji[1]; [1]Northeastern University

Cold-rolling workability, and mechanical properties of Mg-5~22wt%Li-2wt%Zn wrought alloys were studied. The limit of reduction for cold rolling of the ß phase alloys at 16 and 22wt%Li exceeds 90% at room temperature. High strength and good elongation can be obtained by quenching at 300°. The tensile strength of both as-rolled specimens increase with decrease in lithium content. Additionally, age-hardening at room temperature occurs in the specimens of (α+ß)phase(9 wt% Li)and ß phase (16,22 wt% Li) alloys quenched after isothermal holding at 300° for 1h. A remarkable decrease in elongation is encountered for the ß phase alloys having the maximum hardness. The appearance of side-bands in the results of X-ray diffraction patterns suggests that the hardening may be attributed to spinodal decomposition.

4:40 PM
The Tensile and Creep Behavior of Mg-Zn Alloys with and without Y and Zr as Ternary Elements: *Carl J. Boehlert*[1]; [1]Michigan State University

The microstructure, tensile, and tensile-creep behavior (100-200°C) of Mg-Zn alloys ranging from 0-5.6wt%Zn were investigated. The greatest tensile-creep resistance was exhibited by Mg-3.6Zn. In addition the effect of Y and Zr as ternary elements was evaluated. The effect of 1.5-2.9wt%Y was minimal on the creep and tensile strength. The greatest tensile strength and elongation was exhibited by Mg-5.6Zn-0.6Zr, which also exhibited the finest grain size and the poorest creep resistance. The measured creep exponents and activation energies suggested that the creep mechanisms were dependent on stress. For applied stresses greater than 40MPa, the creep exponents were between 4-8. For applied stresses less than 40MPa, the creep exponent was 2.2. The activation energies were dependent on temperature where the Q values between 100-150°C (65kJ/mol) were half those between 150-200°C for the same applied stress value (30MPa). Deformation observations indicated that grain boundaries were susceptible to cracking in both tension and tension-creep.

5:00 PM
Improvement in Creep Resistance of Mg-Sn Based Alloys by Microstructural Modification: *Dae H. Kang*[1]; Sung S. Park[1]; Yoon S. Oh[1]; In-Ho Jung[2]; Nack J. Kim[1]; [1]Pohang University of Science and Technology; [2]Research Institute of Industrial Science and Technology

Mg alloys are the lightest commercially available structural alloys developed so far and have great potential for high performance automotive applications. However, current commercial Mg alloys cannot be used for elevated temperature applications such as powertrain due to their poor creep resistance. In the present research, Mg-Sn based alloys which have thermally stable second phase particles along grain boundaries and within matrix have been studied. Effects of alloying elements and cooling rates on the microstructure of the alloys were investigated. Tensile and creep properties were analyzed with emphasis on the role of second phase particles. It shows that the creep resistance of Mg-Sn based alloys can significantly be improved by the incorporation of thermally stable second phase particles within matrix. To understand the effect of second phase particles within matrix, load relaxation behavior was also analyzed in the context of internal variable theory.

Magnesium Technology 2006: Wrought Alloys and Forming Processes I

Sponsored by: International Magnesium Association, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Alan A. Luo, General Motors Corporation; Neale R. Neelameggham, US Magnesium LLC; Randy S. Beals, DaimlerChrysler Corporation

Tuesday PM	Room: 6A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Amit Ghosh, University of Michigan; Sean R. Agnew, University of Virginia

2:00 PM
A Study on the Static Recrystallization of Cold Rolled Magnesium Alloy AZ80: *Jayant Jain*[1]; Warren J. Poole[1]; Chad W. Sinclair[1]; [1]University of British Columbia

Recently, there has been substantial interest in refining the grain structure of as-cast magnesium alloys using deformation processing. The majority of this work has focused on deformation at elevated temperatures where dynamic recrystallization is the dominant softening mechanism. On the other hand, relatively little literature is available on static recrystallization. In this work, the magnesium alloy AZ80 was chosen due to the possibility of examining situations where grain refinement can be combined with precipitation hardening to produce interesting microstructures and mechanical properties. AZ80 in a supersaturated solid solution was rolled 10-30% at room temperature and then annealed in the temperature range of 200-400°C. The evolution of microstructure was systematically studied using optical microscopy and EBSD. It was observed that static recrystallization of the samples occurred in the temperature range of 325-400°C and that the possibility of concurrent precipitation may exist.

2:20 PM

Deformation Anisotropy of High Purity Magnesium and AZ31B Magnesium Alloy: *Veronica Livescu*[1]; Carl M. Cady[1]; Ellen K. Cerreta[1]; Benjamin L. Henrie[1]; George T. Gray[1]; [1]Los Alamos National Laboratory

The deformation in compression of pure magnesium and AZ31B alloy, both with a strong basal pole texture, has been investigated as a function of temperature, strain rate, and specimen orientation. The mechanical response of both metals is highly dependent upon the orientation of loading direction with respect to the basal pole. Specimens compressed along the basal pole direction have a high sensitivity to strain rate and temperature and display a concave down work hardening behavior. Those loading perpendicularly to the basal pole have a yield stress that is relatively insensitive to strain rate and temperature and a work hardening behavior that is parabolic and then linearly upwards. Post mortem characterization was conducted on a subset of specimens to determine the microstructural and textural evolution during deformation and these results are correlated with the observed work hardening behavior.

2:40 PM

Deformation Characteristics of AZ31 Alloy at the Elevated Temperature: *Yong Nam Kwon*[1]; Y. S. Lee[1]; S. W. Kim[1]; J. H. Lee[1]; [1]Korea Institute of Machinery and Materials

Since magnesium alloy has a limited formability at room temperature, forming should be carried out at the elevated temperature. Forming bewteen 473~573K is known to be successful due to active dynamic recrystallization. If the initial grain size is small, superplasticity could be expected over 623K. Deformation assisted grain growth becomes evident at superplastic temperature of AZ31 alloy. It is quite natural to assume that deformation mechanisms could evolve with temperature, which means that deformation proceeds with dislocation and grain boundary sliding depending on temperature. However, the initial (0001) fiber texture remained strong after deformation irrespective of temperature. In the present study, the deformation characteristics of AZ31 is studied to understand several features of elevated temperature deformation behavior. For this purpose, a series of tensile and relaxation tests were performed along with microstructural observations including texture measurement. Grain growth and compatibilty probelms after grain boundary sliding are discussed.

3:00 PM

Deformation Mechanisms and Ductile Failure of Magnesium AZ31: *Matthew Barnett*[1]; Zohreh Keshavarz[1]; X. Ma[1]; [1]Deakin University

A series of tensile tests were carried out at different temperatures and strain rates using commercial AZ31 over a range of grain sizes. Experiments were performed in a conventional load frame and these tests were complemented with a number of tensile measurements carried out in-situ in a Scanning Electron Microscope. Careful measurements of twin populations were made. These enabled the relationship between deformation conditions and the frequency of c-axis compression twins to be determined. The uniform and total elongations are rationalized in terms of the twinning frequencies. A simple model is constructed on the assumption that compression twins are “soft” with respect to the matrix. This model is used to identify the importance of suppressing twinning for improved ductility.

3:20 PM

Ductility and Low Temperature Superplasticity of Ultrafine Grain Mg Alloys: *Amit K. Ghosh*[1]; Qi Yang[1]; Xiang Li[1]; [1]University of Michigan

Current interest to develop Mg alloys formable at low to warm temperatures has led to exploration of grain refinement of several alloys by severe deformation process. Alloys containing Al and Zn as solid solution elements are investigated in wrought and thixomolded conditions. The paper will discuss the evolution of microstructure during processing and mechanical behavior after the processing steps. Impressive gains in strength and strain rate sensitivity of flow stress are achieved through fine grain processing. The effect of crystallographic anisotropy is significantly reduced in grain sizes between 3 micron and 0.3 micron. This paper will discuss results on the variations in strain hardening rate and available tensile elongations when mechanical twinning is present vs. when it is absent. Some results on biaxial formability will be reported. (Project supported by NSF-DMR Project Award 0314218).

3:40 PM

Hardening Evolution of AZ31B-O Mg Sheet: Xiaoyuan Lou[1]; Min Li[1]; Richard K. Boger[1]; *Robert H. Wagoner*[1]; [1]Ohio State University

The monotonic and cyclic mechanical behavior of O-temper AZ31B Mg sheet was measured in large-strain tension/compression and simple shear. Metallography, acoustic emission (AE), and texture measurements revealed twinning during in-plane compression and untwining upon subsequent tension, producing asymmetric yield and hardening evolution. Plastic straining occurs preferentially by basal slip, which provides two independent slip systems with low critical resolved shear stress. The macroscopic flow stress is determined by the required activation of higher-stress mechanisms to accommodate arbitrary deformation: predominantly non-basal slip for initial tension, twinning for initial compression, and untwinning for tension following compression. The nucleation stress for twinning is larger than that for untwinning. Increased accumulated hardening increases the twin nucleation stress, but has little effect on untwinning. Multiple-cycle deformation tends to saturate after a few cycles of +/- 0.02 strain, but does not saturate for +/-0.035 strain cycles by the time fracture intervenes at 10 cycles.

4:00 PM Break

4:15 PM

Study on Microstructures Evolution, Tensile Mechanical Properties and Forming Techniques of AZ31 Alloy Sheets: *Yan Yunqi*[1]; [1]Suzhou Institute for Nonferrous Metal Research

Better properties of magnesium alloys make it as a natural choice for use as 3C materials. However, the magnesium alloy thin sheets used in this field were difficult to be obtained and punched, whereas the homogeneous microstructures without any textures, the better ductility were needed. AZ31 alloy was investigated using rolling and punching techniques in this paper. Various rolling experiments were carried out to make fine-grained Mg sheets. Microstructures were observed using optical and scanning electron microscope with EBSD device. Tensile tests were conducted at the temperatures ranged from 150 to 300°C for as-rolled AZ31 alloy. The analysis revealed that there is an excellent warm forming temperature for as-rolled AZ31 alloy. A warm deep punching tool setup using heating elements was designed and manufactured to produce the cell phone. The certain type cell phone was produced using this tool and method in the temperature range 250-350°.

4:35 PM

A Study of Damping Properties of Mg-Zr Alloys after Rolling and Annealing: *Chuming Liu*[1]; Jia Zhang[1]; Haitao Zhou[1]; [1]Central South University

The effect of hot rolling and annealing on the damping behavior of Mg-Zr alloys was investigated. It was found that rolling lead to a significant grain refinement, and hence improved their tensile properties. In cast condition damping was strain dependent. However, the damping of the hot rolled alloys weakly depended on strain, which indicates that the damping capacity decreases after hot rolling. In contrast, annealing for the rolled alloy made the damping capacity increasing, but it didn’t reach the as-cast level. The varieties of damping behavior can be explained by dislocation model of Granato and Lücke.

5:00 PM

Direct Chill Casting and Plastic Deformation of Magnesium Alloys: *Gady Isaac Rosen*[1]; Menachem S. Bamberger[2]; Christoph Honsel[3]; [1]Alubin Ltd; [2]Technion; [3]RWP GmbH

To be able to produce sound, repeatable and reliable plastic deformation in magnesium alloys, high quality billets are required. Usually extrusion or hydroforming at elevated temperature is used. This is since they have hexagonal close packed structure, which shows limited ductility at room temperature. In order to be able to deform the low ductility magnesium alloys, premium quality billets and well-defined parameters and conditions are crucial. Therefore, in-depth understanding of the influence of direct chill casting conditions on the production quality and safety, and on micro-structure, properties and workability are of high importance. Efficiency of the production can be improved by utilizing advanced process-simulation in the production floor. The proposed research will be focused on these issues. Extensive simulation will be used in order to model the underlying physics of casting so that process improvement variables can be identified and controlled, resulting in significant benefits. The aim of modeling, the filling and solidification of a casting is to: Provide temperature profiles during, and at the end of filling for a more accurate solidification analysis. Predict the pattern of solidification, indicating where shrinkage cavities and other solidification defects, such as hot tearing. And, predict solidification time and the effect of cooling system on the solidification.

5:20 PM

Effects of Microstructural Changes on Tensile Properties of AZ31 Magnesium Alloy up to Localized Necking: *Ravi Verma*[1]; Louis G. Hector[1]; Manuel Marya[2]; [1]General Motors R&D Center; [2]Colorado School of Mines

An AZ31 magnesium alloy was investigated for effects of microstructural modifications on room temperature mechanical properties. Three distinct microstructures were generated through heat-treatments: (1) single-phase, fine grains; (2) single-phase, coarse grains with twins; (3) fine grains decorated with $Mg_{17}(Al,Zn)_{12}$ grain-boundary precipitates. Small coupons fabricated from each microstructure were then tested to failure in a miniature tensile stage. True stress – true strain curves were computed with the digital image correlation (DIC) technique and strain accumulation in the necking region of each coupon was quantified with two-dimensional strain maps. The initial yield points, ultimate tensile strengths and maximum elongations prior to failure were taken as measures of room temperature tensile properties of each microstructure. These values are examined within the context of key features of each microstructure in order to infer the mechanism of plastic deformation in tension and whether or not the potential for improved room temperature formability exists.

Materials Design Approaches and Experiences II: Light Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee

Program Organizers: Michael G. Fahrmann, Special Metals Corporation; Yunzhi Wang, Ohio State University; Ji-Cheng Zhao, General Electric Company; Zi-Kui Liu, Pennsylvania State University; Timothy P. Gabb, NASA Glenn Research Center

Tuesday PM — Room: 202B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Ingo Steinbach, RWTH-Aachen Access EV; Qigui Wang, General Motors Corporation

2:00 PM

Effect of Moderate Pressures (to ~5GPa) on Phase Equilibria in Some Al-Based Alloys: *Joanne L. Murray*[1]; [1]Alcoa Technical Center

Moderate pressure affects solidification, melting, and solid solubility of Al alloys to an extent great enough that the effect must sometimes be taken into account when optimizing alloy composition and processing. Moreover, pressure can play a role in development of qualitatively new alloys. So far little has been done, to model the effect of pressure on the phase equilibria of Al-base alloys. Minamino et al.* have made a start at introducing thermal expansion, modulus and molar volumes as parameters of the Gibbs energies. In the present study, a methodology is developed for assessing the physical property data needed to model the pressure dependence of phase diagrams. The systems Al-Ag, Al-Cu, Al-Ge, Al-Li, Al-Mg, Al-Si, Al-Zn will be modeled. *Y. Minamino, T. Yamane, T. Sato, Journal of Japan Institute of Light Metals. Vol. 38, no. 12, pp. 800-806 (1988).

2:25 PM Invited

The Latest Development of Computational Tools for Virtual Casting of Aluminum Components: *Qigui Wang*[1]; [1]General Motors Corporation

The increasing use of cast aluminum components in critical structures has required improved quality, with more reliable and quantifiable performance. Aluminum shape casting processing is very complex and often involves many competing mechanisms, multi-physics phenomena, and potentially large uncertainties. The only effective way to optimize the processes and achieve the desirable mechanical properties is through the development and exploitation of robust and accurate computational models. Numerous modeling and simulation techniques have been developed and applied in practice for aluminum casting and subsequent processing that enable both casting designers and process engineers to better design and develop sound shape cast components with minimum lead time and cost. This talk will review the latest development of computational tools for aluminum shape casting processing from alloy design to mechanical properties. Some suggestions for future development will be presented.

2:50 PM Invited

Prediction of Porosity Defects and Mechanical Properties of a High Pressure Die Cast A380 Aluminum Alloy Component: *Mei Li*[1]; Jacob W. Zindel[1]; Larry A. Godlewski[1]; John E. Allison[1]; [1]Ford Motor Company

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. Porosity is a commonly found defect in high pressure die cast components which affects both the ductility and fatigue properties. This talk describes the development of an algorithm which can predict porosity defects in HPDC A380 component and the correlation of defects to fatigue property in the component. The prediction can be used to design and optimize casting component and process.

3:15 PM

Study on Preparation of Ceramic Particles Reinforced Aluminum Foam: *Wang Yong*[1]; Yao Guang-Chun[1]; Li Bing[1]; [1]Northeastern University

In this work, fly ash was selected as reinforcement in melt-foaming process. The most component are quartz and mullite was observed by XRD in the fly ash, therefore it's a kind of hardness ceramic particles. Fly ash particles act as viscosity increaser and increase stabilization of bubble wall in the aluminium foam. Put fly ash into melting aluminium, suitable stir and add in frothing agent, after foaming and cooling, then obtain particles reinforced aluminium foam. Through compression strength test, compare aluminium foam which fly ash particles as viscosity increaser with Ca as viscosity increaser. Experimental results indicate: fly ash has not only viscosity increase function, but also particles reinforced function, because of Al2O3 and mullite in the fly ash, in which the Al2O3 particle is quartz and melting aluminium have happened in-situ reaction.

3:35 PM Break

3:55 PM Invited

CalPhad and Phasefield Modeling for the Development of New Mg-Base Alloys: *Ingo Steinbach*[1]; Janin Eiken[1]; Bernd Boettger[1]; Munekazu Ohno[2]; Rainer Schmid-Fetzer[2]; Gerald Klaus[1]; Andreas Buehrig-Polaczek[1]; [1]RWTH-Aachen; [2]TU Clausthal

The solidification behaviour of Mg-base alloys is investigated by CalPhad and Phase Field modelling. The investigation starts from a critical reassessment of the Mg-Al system with special care on micro alloying elements. Equilibrium and Scheil calculations serve to identify candidates for new alloy compositions. Subsequently Phase Field calculations for the equiaxed solidification of the Mg-phase are performed taking into account nucleation on primary precipitates, growth with hexagonal surface anisotropy reflecting the Mg lattice structure and eutectic termination of solidification. The Phase Field simulations reveal the influence of alloy composition and casting conditions on the phase morphology. Phase distribution and morphology together define quality criteria for the as cast structure, dependent on alloy composition and process conditions. The presentation discusses the prospect and limitation of the numerical approach in comparison with experimental results.

4:20 PM
The Mg-Zn-Zr System: From First Principles to Grain Refining - An Integrated Approach to Materials Design: *Raymundo Arroyave*[1]; Dongwon Shin[1]; Axel van de Walle[2]; Zi-Kui Liu[1]; [1]Pennsylvania State University; [2]Northwestern University

In this work we show how, by integrating first-principles techniques with the CALPHAD method, it is possible to address practical problems in materials manufacturing. The thermodynamic models of the Zn-Zr and Mg-Zr binaries are obtained through the CALPHAD approach with the aid of first-principles methods including state of the art calculations of the finite temperature thermodynamic properties of intermetallic phases in which vibrational degrees of freedom are taken into account, as well as the energetics of special quasirandom structures, utilized to mimic the behavior of random solid solution phases. The two binary models obtained in this work are combined with the existing description for the Mg-Zn system to predict the thermodynamic behavior of the Mg-Zn-Zr ternary. This thermodynamic description is then used to address important issues regarding the grain refining behavior and solidification sequences observed in cast Mg-Zn-Zr alloys.

4:40 PM
Balanced Alloy Design for 6000 Series Al-Mg-Si Alloys: Malcolm J. Couper[1]; *Barbara Rinderer*[1]; Mark Cooksey[2]; [1]Comalco Aluminium Ltd; [2]CSIRO Minerals

In 6000 series Al-Mg-Si alloys the optimum strength, with minimum alloy content, can be achieved using a "balanced" alloy composition with respect to MgxSi precipitates, with x close to 1. Al-Mg-Si alloys with a wide range of Mg and Si contents were VDC cast as billet, extruded and the tensile properties in T1, T4, T5 and T6 heat treatment condition evaluated. Balanced alloys can be designed with strengths that increase with increasing total alloy content, for example SF6060 and SF6063. The performance of 6061 alloys with additions of Cu, have also been investigated.

Materials in Clean Power Systems: Applications, Corrosion, and Protection: Interconnection and Sealing in Fuel Cells I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizers: Zhenguo Gary Yang, Pacific Northwest National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Michael P. Brady, Oak Ridge National Laboratory

Tuesday PM — Room: 212B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Frederick S. Pettit, University of Pittsburgh; Lorenz Singheiser, Forschungszentrum Jülich

2:00 PM Keynote
Interconnect Performance in SOFC Environments: *Peggy Y. Hou*[1]; Hideto Kurokawa[1]; Craig Jacobson[1]; Steven Visco[1]; Lutgard De Jonghe[1]; [1]Lawrence Berkeley National Laboratory

For medium temperature solid oxide fuel cells (SOFCs) operating at 600-800°C, ferritic steels can be successful interconnect or support structure materials due to their low cost, compatible thermal expansion with the electrode and electrolyte ceramics and the ability to develop a relatively slow growing and conductive Cr_2O_3 surface layer under SOFC operating conditions. This paper gives an overview of the current status of the development of such interconnect materials and discusses important issues concerning their usage. These include the effect of alloying elements and coatings on the growth and evaporation rates of these thermally grown Cr_2O_3 scales, the boundary conditions on scale adherence and the effect of applied current on Cr_2O_3 growth in typical oxidizing and reducing SOFC environments. Experimental data performed under short term exposures will be presented along with models predicting long term behaviors.

2:45 PM Invited
Metallurgical and Geometrical Parameters Affecting the Oxidation of Ferritic Steels as Interconnector Materials in Solid Oxide Fuel Cells: *W. J. Quadakkers*[1]; [1]Forschungszentrum Jülich

High-Cr ferritic steels are presently being considered as SOFC interconnect materials because they fulfil most of the requirements for SOFC application. In the present paper the oxidation behaviour a number of high-Cr, ferritic steels in the temperature range 700 to 900°C in dry air, wet air and H2/H2O-mixtures will be described. Thereby the effect of main alloying elements (e.g. Cr) as well as minor alloying additions (Mn, Si, Al, Ti, La) on the oxidation properties will be treated. Considering the envisaged possible application of SOFC's (stationary as well as mobile), special emphasis is given to the effect of interconnect geometry (mainly component thickness) on the oxidation behaviour. It will be shown, that oxidation data derived from experiments using thick components (of a few millimetres in thickness) cannot straightforwardly be used to describe the oxidation properties of thin walled (a few tenths of a millimetre) components considered for SOFC-APU application.

3:15 PM
Continuous Improvements in the Commercial Development of Alloy 22APU for Interconnect Applications in Solid Oxide Fuel Cells (SOFC): *Larry Paul*[1]; Ralj Hojda[1]; [1]ThyssenKrupp VDM USA Inc.

Several years ago a new material was introduced for use as an interconnect material for solid oxide fuel cell (SOFC) application. The development of this material was the joint effort of Forschungszentrum Jülich and ThyssenKrupp VDM. Crofer 22APU has unique properties that make it an ideal choice in application as an interconnect material. The key properties are the coefficient of thermal expansion (CTE), which closely matches the ceramic materials used in the SOFC stack, the good electrical conductivity of the protective oxide scale that is formed during operation of the fuel cell stack and the outstanding oxidation resistance at operating temperatures. Several lessons were learned during the development, scale-up, and production of the Crofer APU material. The end result is a commercially available material that meets the technical requirements for interconnects in SOFC application.

3:40 PM
Influence of Minor Alloy Elements on the Performance of Crofer 22APU: *Paul D. Jablonski*[1]; David E. Alman[1]; [1]U.S. Department of Energy

Crofer 22APU, a ferritic stainless steel which contains 22 weight percent chrome, was developed at Jülich specifically for application as an interconnect material in solid oxide fuel cells (SOFC). Thyssen-Krupp has taken on the task of manufacturing this alloy. Two commercial heats of Crofer 22APU were evaluated for use in the SOFC temperature range of 700 to 800C. It was observed that the heat which contained higher levels of the aluminum and silicon, presumably either from alloy makeup or furnace practices, was less resistant to oxidation. Furthermore, these minor differences appeared to also impact the performance of a surface treatment we have developed for enhanced oxidation resistance. We will discuss the impact of these minor impurities in terms of long term component stability.

4:05 PM Break

4:20 PM Invited

SnO2:F Coated Stainless Steels for PEM Fuel Cell Bipolar Plates: *John A. Turner*[1]; Heli Wang[1]; [1]National Renewable Energy Laboratory

The corrosion resistance and the interfacial contact resistance are the major challenges for stainless steels operating as bipolar plates in a polymer electrolyte membrane fuel cell (PEMFC). Modification of the surface of the steels so as to have a lower contact resistance and better corrosion resistance is necessary. Conducting oxides, used as current collectors for solar cells, can carry currents up to 1A/cm2 and may offer protection against corrosion and additionally give high interfacial conductivity. These oxides can be applied at high-speeds during production and are of interest for fuel cell coatings. Films of SnO:F are of particular interest due to their known high stability in a variety of aqueous solutions. In this report we will describe our work with SnO2:F coatings on stainless steels.

4:50 PM

Oxidation Behavior of Fe-Cr-Al Alloy: Metallic Interconnects for Solid Oxide Fuel Cell Applications: *Venkateswarulu Kamavaram*[1]; Ramana G. Reddy[1]; [1]University of Alabama

Solid oxide fuel cells (SOFCs) are developed to meet the energy requirements of the society. Due to high operating temperatures (800-1000°C), the material selection for SOFC is an important aspect of its design and development. Among the four essential components of SOFC; anode, cathode, electrolyte, and interconnect, materials selection for interconnects is a critical factor. Iron based alloy (Fe; Cr: 22%; Al: 5%; Zr: 0.1% and Y: 0.1%) was investigated as metallic interconnect material. Oxidation behavior of Fe-Cr-Al alloy in the temperature range 650-850°C under air and oxygen atmospheres was investigated. Both short-term (50 hrs) and long-term (1000 hrs) oxidation of the FeCrAl alloy was studied using thermogravimetric analysis (TGA). Effect of thermal cycling on the stability of FeCrAl alloy also was investigated. Short-term oxidation kinetics of the alloy followed parabolic rate law in the temperature range 600-850°C with an Arrhenius activation energy of 196 kJ/mol.

5:15 PM

Effect of Ceramic Coating on Chemical Stability of a Composite Seal for Solid Oxide Fuel Cells: Srivatsan Narasimhan[1]; Kristoffer Ridgeway[1]; *Xinyu Huang*[1]; [1]University of Connecticut

The interconnects in planar solid oxide fuel cells (SOFC) require stable hermetic sealing to their adjacent components to maintain efficiency and longevity. The seal material, typically glass, has to be chemically compatible with the Fr-Cr based ferritic stainless steel commonly used as interconnect for intermediate temperature SOFCs. A novel multilayered composite seal structure that consists of layers of intermetallic bondcoat, a ceramic topcoat, and a filler glass, has been investigated by the authors as an alternative to traditional glass seals, where glasses are in direct contact with Fr-Cr interconnects. One advantage to use a ceramic layer in the composite seal is to mitigate the undesirable chemically interaction. In this study, high temperature aging tests of SOFC seal samples with and without a ceramic inter-layer were conducted. After aging, the degrees of interfacial chemical interactions were characterized by SEM/EDX and the results for the two types of samples were compared.

Materials Processing Fundamentals: Smelting and Refining

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Fundamentals Committee, TMS: Process Modeling Analysis and Control Committee
Program Organizers: Princewill N. Anyalebechi, Grand Valley State University; Adam C. Powell, Massachusetts Institute of Technology

Tuesday PM	Room: 203A
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Princewill N. Anyalebechi, Grand Valley State University

2:30 PM

Mechanisms of Oxygen Partial Lead Softening: Daryl Vineberg[1]; *Sadegh Firoozi*[2]; Ralph Harris[2]; [1]Ontario Ministry of the Environment; [2]McGill University

Simultaneous kinetic studies and visual observations of the oxidation of static and/or agitated baths of lead containing arsenic, antimony and tin as impurities were conducted. Their purpose was to further the understanding of the mechanisms of partial lead softening with pure oxygen as practiced at Teck Cominco Ltd. The visual observations of the melt/gas interface between 500 and 600°C saw the formation of a solid oxide layer on pure lead and lead-tin alloys and formation of liquid oxides during the oxidation of lead-arsenic and lead-antimony alloys. Thermogravimetric measurements showed that the rate of oxygen uptake was strongly influenced by the form of oxides being generated at the melt/gas interface. Tin was most effective in retarding the rate of oxidation. Arsenic increased the rate of oxygen uptake throughout the temperature range and antimony showed a moderate increase in the rate of oxygen uptake at 600°C as compared to arsenic.

2:55 PM

Prevention of Resulfurization in Magnesium Desulfurization Process of Molten Iron: *Jian Yang*[1]; Mamoru Kuwabara[1]; Keiji Okumura[2]; Zhongzhu Liu[1]; Masamichi Sano[1]; [1]Nagoya University; [2]Nagoya Institute of Technology

There are two kinds of mechanisms of resulfurization in magnesium desulfurization process of molten iron. One is decomposition of the desulfurization product of MgS under the inert atmosphere. The other is oxidation of MgS under the oxidative atmosphere. In the desulfurization process with magnesium vapor produced in-situ by aluminothermic reduction of MgO, lowering operating temperature and oxygen partial pressure in the atmosphere, and adding more pellets containing magnesium oxide and aluminum could effectively prevent the resulfurization. The resufurization took place more markedly by using Al2O3 crucible than by using MgO or C crucible. Adding CaO onto the melt surface was an effective method for preventing the resulfurization due to transformation of the desulfurization product of MgS into the more stable compound of CaS. Since addition of the activated charcoal powders greatly decreased the transfer rate of oxygen in the atmosphere to the melt surface, it also reduced the resulfurization remarkably.

3:20 PM

Carbothermal Reduction Study of Kaolin and Two Different Silica Sources: *Sutham Niyomwas*[1]; [1]Prince of Songkla University

Synthesis of Al2O3-nSiCw composite was obtained in situ by carbothermal reduction of a mixture of kaolin and two different silica sources. The carbothermal reduction was carried out in a horizontal tube furnace under flow of argon gas. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of the reacting species. The synthesized products were mixtures of alumina and silicon carbide in the form of whiskers. The effects of adding two different silica sources of rice husk ash and silica powder to the mixture of kaolin and activated carbon were investigated. XRD and SEM analyses indicate complete reaction of precursors to yield Al2O3-nSiC as product powders, with the SiC having whisker morphology.

3:45 PM
Increase in Concentration of a Zn-Containing Volatile Complex by UV Irradiation of a Target for ZnO Films Synthesis: *Emma Rubenovna Arakelova*[1]; V. G. Parvanyan[1]; F. A. Grigoryan[1]; G. G. Asatryan[1]; G. G. Petrosyan[1]; [1]Statye Engineering University of Armenia

An increase in concentration of a Zn-containing volatile complex for synthesis of ZnO films was investigated when processing their target by ultraviolet radiation emitted by mercury lamp. The experiments were carried out using a flow-type quartz reactor. ZnO tableted target was preliminarily irradiated during 2 to 7 hours. H2O2 (98 %) vapor at 25 Pa passed through the reactor with ZnO target at 373 K. As substrate material, five subsequent quartz cylinders were used at 423 K. The experiment lasts 4 hours. H2O2 decomposition on ZnO surface was accompanied by formation of a Zn-containing volatile complex which passed in gas phase and decomposed on the substrates with isolation of final ZnO. The concentration of the Zn-containing volatile complex (N) in gas phase increases with UV pre-irradiation time (t) (without irradiation N=1,05 •10^{11} particles / cm^3; after t = 2 hours, N=5,19 •10^{11} particles / cm^3, after t = 4 hours, N=9,95 •10^{11} particles / cm^3, after t = 7 hours, N=1,86 •10^{12} particles / cm^3.

4:10 PM Break

4:25 PM
Preparation of SmCo5 Alloy by Chemical Co-Deposition and Reduction and Diffusion Method: *Guo Xueyi*[1]; [1]Central South University

This SmCo5 powder was prepared by chemical co-precipitation followed by improved reduction and diffusion method. In the chemical co-precipitation, the thermodynamic equilibrium analysis was conducted, then the precipitation were done to address all the factors on the behaviors of the precursor particle. The optimum conditions were determined, and the precursor particles with narrow size distribution, spherical shape or quasi-sphere were prepared. In the high temperature process, the precursor powder was treated to produce the SmCo5 alloy by the improved reduction and diffusion method. First, the Sm-Co oxide powder was produced by controlling the calcining temperature, atmosphere and time. Then, the Sm-Co oxide powder was treated to obtain the SmCo5 alloy under the hydrogen atmosphere, precise reduction and diffusion temperature and the rate for temperature rise. The effects of all the parameters on the high temperature process were addressed.

4:50 PM
Electrical Field Effects on Reactive Sintering: *Dat V. Quach*[1]; Lia A. Stanciu[2]; Vladimir Y. Kodash[1]; Joanna R. Groza[1]; [1]University of California; [2]Purdue University

Field-assisted sintering technique (FAST) is a novel processing method to process powder materials at lower temperature and in shorter time. The application of an external field can increase the mobility of atoms/ions involved in the diffusion process, thus enhancing not only neck formation and growth but also the rate of chemical reaction. Aluminum titanate (Al_2TiO_5) was prepared by FAST sintering of a mixture of amorphous sol-gel Al_2O_3 and TiO_2 powders at 1300°C in 10 minutes. Compared with conventional sintering that requires a longer sintering time (2 hours), FAST gives a higher yield of Al_2TiO_5 formation. To further investigate the effects of electrical field/current on the reaction between Al_2O_3 and TiO_2, basic diffusion couple studies were performed and the results will be reported.

Multicomponent-Multiphase Diffusion Symposium in Honor of Mysore A. Dayananda: Intermetallics and Ceramics

Sponsored by: The Minerals, Metals and Materials Society, ASM Materials Science Critical Technology Sector, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Yong-Ho Sohn, University of Central Florida; Carelyn E. Campbell, National Institute of Standards and Technology; Richard Dean Sisson, Worcester Polytechnic Institute; John E. Morrall, Ohio State University

Tuesday PM — Room: 203B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Marek J. Danielewski, AGH University of Science and Technology; Dennis D. Keiser, Idaho National Laboratory

2:00 PM Invited
Diffusion in Binary Intermetallic Compounds: *H. Mehrer*[1]; [1]Universität Münster

Diffusion studies on binary intermetallics including silicides and aluminides are reviewed. Particular attention is given to ^{99}Mo, ^{31}Si, and ^{71}Ge diffusion in single crystals of tetragonal $MoSi_2$2. Si and Ge diffuse six or more orders of magnitude faster than Mo suggesting that diffusion in the Si and Mo sublattice is decoupled. Diffusion is in all cases significantly faster perpendicular to the tetragonal axis than parallel to it. Monte Carlo simulations of correlation effects and anisotropy ratio of vacancy-mediated diffusion in the Si-sublattice lead to their rationalization. Diffusion in aluminides is reviewed with particular attention to iron-aluminides: interdiffusion coefficients were measured on binary diffusion couples. Tracer diffusivities of Al were deduced using the Darken-Manning equation, ^{59}Fe tracer diffusivities, and theoretical values of thermodynamic factors. Al and Fe diffusion are closely coupled in B2 structured FeAl. The diffusion of ternary alloying elements in FeAl and self-diffusion in NiMn is also discussed.

2:30 PM
Interdiffusion in Mo_5SiB_2 (T_2) Synthesized in a Mo_5Si_3/Mo_2B Diffusion Couple: *Sungtae Kim*[1]; John H. Perepezko[1]; [1]University of Wisconsin

Diffusion couples based upon Mo_2B and Mo_5Si_3 were used to determine the diffusion kinetics of T_2 phase development and the relative diffusivities controlling the kinetics. During the growth of the T_2 phase, Si and B atom movements were found to be coupled and driven by the Si concentration gradient. The activation energy for interdiffusion in the T_2 phase was evaluated to be larger than that for layer growth of Mo_5Si_3 in both Mo/Si and $MoSi_2$/T_2 diffusion couples. This larger activation energy in T_2 may result from the restricted jump possibilities in T_2 due to the coupled nature of Si and B atom movements. The relatively slow diffusion of the T_2 phase due to this high activation energy explains the improvement in the high-temperature creep properties of the Mo-Si-B alloy systems. The support of the AFOSR (F49620-03-1-0033) is gratefully acknowledged.

2:55 PM
Reactive Interdiffusion in the Binary System Ni-Si: Morphology of the Ni3Si2 Phase: *Delphine Borivent*[1]; Jérôme Paret[1]; Bernard Billia[1]; [1]L2MP

In binary multi-phase diffusion, it is generally admitted that interfaces between phases are necessarily plane. However, a few cases exist, as the binary diffusion couples Ni-Si, Mo-Si and Fe-Al, for which an intermediate phase of each system grows with an irregular needle-like morphology. To characterize the nonplanar growth of Ni3Si2, we have studied the behaviour of intermetallic compounds formation in Ni/Si bulk samples by in-situ X-ray microtomography, for different annealing times. Studying evolution of needles curvature radii with time, we show that these radii grow proportionally to the square-root of time. This result is compatible with an anisotropic diffusion model of the origin of this peculiar microstructure.

TUESDAY PM

3:20 PM Invited
Defect Interactions in Binary Compounds of Transition Metals at High Temperatures: *Ewa Maria Fryt*[1]; [1]AGH University of Science and Technology

In highly defective solids in which concentration of point defects is higher than 0.1%, interactions between defects become important influence on diffusion in its crystallographic lattices and many properties. For description of interactions between defects in highly defective solids it is necessary to introduce new terms in diffusion theory characterizing the influence of interactions between defects especially for temperature and pressure dependencies of activity and mobility, and hence on diffusivity. The above mentioned function is characteristic for a crystallographic lattice of a given compound, different types of defects and their relative concentrations, attractive or repulsive interactions between defects and for a given diffusion mechanism. This approach will be discussed using different examples of determined free energy of defect interactions and interpretation of its influence on many macroscopic properties of binary compounds of some transition metal oxides, sulphides and carbides at high temperatures.

3:50 PM Break

4:10 PM
Irradiation Enhancement of Interdiffusion in the Interaction Zone of U-Mo or U3Si2 Dispersion Fuel in Al: *Yeon Soo Kim*[1]; Gerard L. Hofman[1]; Steve Hayes[2]; Ho Jin Ryu[3]; M. R. Finlay[2]; [1]Argonne National Laboratory; [2]Idaho National Laboratory; [3]Korea Atomic Energy Research Institute

The main issue in the process of developing dispersion fuel containing U3Si2 or U-Mo particles in an Al Matrix is the interdiffusion zone growth at the interface between the fuel and matrix. To predict the zone width during irradiation, extrapolated values of the out-of-reactor correlation based on diffusion-couple test data at 350-550°C to reactor operation temperature (~150°C) must be enhanced to account for irradiation effect. The irradiation enhancement factor varies due to the changes in fission density and in deposition of fission fragment damage in the zone. Using the TRIM code, enhancement factors as a function of zone width were assessed for U-Mo/Al and U3Si2/Al dispersion fuels. These factors were compared to measured irradiation data. In addition, diffusion enhancement for ion beam irradiation in U-Mo/Al was measured. Comparison to the analytically obtained results showed that the ion beam test results were in line with those of analytical results.

4:35 PM
Interdiffusion in Ilmenite Solid Solutions of (Ni,Co)TiO_3: *Srinivasa Reddy*[1]; Lowell B. Wiggins[1]; Brian Sundlof[1]; [1]IBM Corporation

This paper reports the results of interdiffusion studies in (NixCo1-x)TiO3 solid solutions in the temperature range of 1473-1623K. In the ilmenite structure, the divalent (Ni,Co) cations and the tetravalent Ti occupy separate sub-lattice and the diffusion of ions in different sub-lattice are not strongly correlated with each other. At 1573 K, the interdiffusion co-efficient increases by more than an order of magnitude when the composition changes from NiTiO3 to CoTiO3. This indicates that the concentration of point defects responsible for diffusion increases with increasing Co in the solid solution. However, no data is available regarding the point defects or tracer diffusion of individual cations in these solid solutions and hence the applicability of Darken type relation is not known.

5:00 PM
Diffusional Analysis of a Multiphase Oxide Scale Formed on a Mo-Mo_3Si-Mo_5Sib_2 Alloy: *Voramon S. Dheeradhada*[1]; David Johnson[1]; Mysore A. Dayananda[1]; [1]Purdue University

Diffusional analyses were performed to understand the oxidation at 1300°C of a multiphase Mo-13.2Si-13.2B (at%) alloy. During oxidation, a protective glass scale formed with an intermediate layer of (Mo+glass) between the base alloy and external glass scale. Compositional profiles across the (Mo+glass) layer and the external glass scale were determined, and interdiffusion fluxes and effective interdiffusion coefficients were calculated by using "MultiDiFlux©" software. Interdiffusion coefficients for the various component in the Mo and glass phases were estimated on the basis of Mishin's analysis. The motion of the (alloy/Mo+glass) and (Mo+glass/glass) interphase boundaries after passivation was examined. Additionally, vapor-solid diffusion experiments at 1300°C were performed with multiphase Mo-10Si-10B(at.%) and single phase Mo_3Si or T2 specimens. These specimens were exposed to vacuum to induce silicon loss resulting in the formation of a Mo layer. An average effective interdiffusion coefficient of silicon in molybdenum at 1300°C was estimated at about 10^{-17} m^2/s.

5:25 PM
Oxygen Diffusion through Aluminum-Containing Amorphous Silica: *Yiguang Wang*[1]; Yi Fan[2]; Ligong Zhang[2]; Yong-Ho Sohn[1]; Linan An[1]; [1]University of Central Florida; [2]Chinese Academy of Science

Oxygen diffusion through vitreous silica has attracted extensive attention in last decades due to its scientific interests and practical importance. The process is the rate-control step for the thermal growth of dielectric silica thin film on silicon, which enabled technology for modern microelectronics and optoelectronics. The same process also determines the degradation of silicon-based ceramics (e.g. SiC and Si3N4) when they are used in high-temperature oxidizing environments. Oxygen diffusion through amorphous silica doped with small amount of aluminum is investigated using SIMS to profile the diffusion of 18O tracer. We demonstrate that aluminum-doping can significantly inhibit both interstitial transportation and network diffusion of oxygen. The activation energy of oxygen network diffusion for aluminum-doped silica is two times higher than that for pure silica. Contrary to conventional understanding, these results suggest a small amount of aluminum-doping could decrease oxygen diffusion by strengthening the network structure of otherwise pure silica.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials V: 3D, Fine Pitch and High Temperature/Low Temperature Interconnects in Electronics Packages

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee
Program Organizers: Katsuaki Suganuma, Osaka University; Douglas J. Swenson, Michigan Technological; Srinivas Chada, Jabil Circuit, Inc.; Sinn Wen Chen, National Tsing-Hua University; Robert Kao, National Central University; Hyuck Mo Lee, Korea Advanced Institute of Science and Technology; Suzanne E. Mohney, Pennsylvania State University

Tuesday PM	Room: 213B
March 14, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Hyuck Mo Lee, Korea Advanced Institute of Science and Technology; Jae-Ho Lee, Hong Ik University

2:00 PM Invited
Interconnection Process and Electrical Properties of the Interconnection Joints for 3D Stack Package with Cu Via: Kwang-Yong Lee[1]; Hye-Jin Won[1]; Teck-Su Oh[1]; *Jae-Ho Lee*[1]; Tae-Sung Oh[1]; [1]Hong Ik University

Stack specimen with three dimensional interconnection structure through Cu via of 75µm diameter, 90µm height and 150µm pitch was successfully fabricated using subsequent processes of via hole formation with Deep RIE (reactive ion etching), Cu via filling with pulse-reverse electroplating, Si thinning with CMP, photolithography, metal film sputtering, Cu/Sn bump formation, and flip chip bonding. Contact resistance of Cu/Sn bump and Cu via resistance could be determined from the slope of the daisy chain resistance vs the number of bump joints of the flip chip specimen containing Cu via. When flip-chip bonded at 270°C for 2 minutes, the contact resistance of the Cu/Sn bump joints of 100X100µm size was 6.7 mΩ, and the Cu via resistance of 75µm diameter, 90µm height was 2.3 mΩ.

2:30 PM
Electro and Electroless Copper Plating on Flexible Substrate for CoF Applications: *Jae-Ho Lee*[1]; Sung-Sup Byun[1]; Jung-Eun Kang[1]; [1]Hong Ik University

As electronic devices are getting smaller and lighter, the density of copper lines on flexible circuit board (FCB) is getting higher. Subtractive method is cost effective and most widely used method in copper line formation, however, as the line pitch is getting smaller, the lateral etching of copper cause serious problem. To replace the subtractive method, semi-additive method was used for fine pitch copper line. Lithography and copper electroplating are two key factors in this process. Polyimide film was used as flexible substrate and copper seed layer was deposited on polyimide film by magnetron sputtering method. For copper electroplating bath, low residual stress bath was used since flexibility of copper line is very important. The feasibility of electroless plating to replace sputtered copper seed layer was also investigated. The adhesion strength between polyimide and copper was improved by treating the polyimide surface with amines.

2:55 PM

Decapsulation Method for Flip Chip with Ceramics in Microelectronic Packaged Devices: T. I. Shih[1]; *Jenq-Gong Duh*[1]; [1]National Tsing Hua University

Decap of flip chip with ceramics in microelectronic devices is an arduous work especially accompanied by polishing techniques. Current processing for flip chip with ceramics is limited due to the ability of human and capability of equipment. After decap of flip chip package, the destructiveness of pad and clean level of samples are primary concerns that should be focused for any possible improvement. This study is aimed to provide an alternative methodology to decap the flip chip package of semiconductor microelectronic device by manual method and chemical reaction of selective acids. A novel processing approach is developed to well prepare the flip chip sample consistently and accurately. It can be demonstrated that this way of process suffices to increase sample through put and also to improve surface finishes with reduced consumables and preparation time.

3:20 PM

Deposition of Conductive Films on Electronic Substrates by Using Gold Nanopowders: *Tzu-Hsuan Kao*[1]; Jenn-Ming Song[2]; In-Gann Chen[1]; [1]National Cheng Kung University; [2]National Dong Hwa University

Since nano-sized metals exhibit a dramatically low melting point compared to bulk materials, nano-metallic particle fluid suspensions (NPFS) can be deposed and cured to obtain electric conductors at a relative low processing temperature. This implies that fabrication of micro-interconnects without using the conventional lithography-etching process is possible. In this study, gold particles with an average size of 5nm were selected to prepare NPFS. Utilizing spin coating and ink-jet printing, the NPFS was deposited onto several commonly used electronic substrates, Cu, Ni and Al. After a proper curing treatment, the degree of continuity and adhesion strength of the coated film on the Ni substrate was better than that on the Cu substrate, which is in turn better than Al substrate. Electrical resistivity of the gold film thus produced and the interfacial reactions between the films and the substrates were also examined. Finally, the optimal processing parameters were recommended.

3:45 PM

Study of Au-Si Wafer Bonding Technology for GaN/Si Wafer Bonding: *Hsiang-En Chiu*[1]; Cheng-Yi Liu[1]; [1]National Central University

A void-free and thermally stable wafer bonding is required for the fabrication of the state-of-the-art thin-GaN LED device. To meet this criteria, Au-Si bonding is one of good techniques to bond GaN wafer with Si wafer. However, it has been reported that a lot of voids formed at the interface between Au bonding layer and Si(111) surface due to the serious inter-diffusion between Si and Au. In this study, the GaN/Ti/Ni/Au wafers were bonded with Si substrates. By adjusting the process temperatures, bonding time, and thickness of Au bonding layer, a void-free Au-Si eutectic layer can be achieved. In this talk, we will also compare Au-Si bonding interface microstructure by using different Si substrates, such as, n-type and p-type high-doping (111) Si. The detail Au-Si reaction analysis will be given.

4:10 PM Break

4:20 PM

Development of Bi-Based High Temperature Pb-Free Solders with Second Phase Dispersion: Microstructure, Interfacial Reaction and Thermal Fatigue: *Yoshikazu Takaku*[1]; Ikuo Ohnuma[1]; Ryosuke Kainuma[1]; Yasushi Yamada[2]; Yuji Yagi[2]; Yuji Nishibe[2]; Kiyohito Ishida[1]; [1]Tohoku University; [2]Toyota Central R&D Laboratory, Inc.

Bi based alloys are candidates of high temperature Pb-free solder which can be substituted for conventional Pb-rich Pn-Sn solders. However, inferior properties such as brittleness should be improved for practical use. In this study, the microstructure of Bi-based alloys was designed by thermodynamic database, ADAMIS (Alloy Database for Micro-Solders), and their applicability for high temperature solder was investigated. Bi-based alloys were prepared by a rapid quenching method. The microstructure was controlled through liquid-phase separation to exhibit an in-situ composite of fine dispersed particles in the Bi-based matrix. Other composite samples which consist of Cu-based alloy powder in the Bi-based matrix were also fabricated. Semiconductor chips and substrates were soldered with the Bi-based composite alloys. After the soldering, the interfacial reaction was examined by SEM. Thermal cycles between -40°C and 195°C were subjected to soldered samples. The effect of the second phase dispersion on thermal fatigue properties was investigated.

4:45 PM

Thermal and Humidity Stability of Zn-xSn and Zn-30In Alloys as High Temperature Lead-Free Solder: *Jae-Ean Lee*[1]; Keun-Soo Kim[1]; Katsuaki Suganuma[1]; [1]Osaka University

The potential of the newly designed Zn-xSn(x=20, 30 and 40mass%) and Zn-30 mass%In alloys as a high temperature lead-free solder have been evaluated, especially focusing on the thermal and humidity stability. The hypereutectic solders showed two endothermic peaks in DSC, these peaks are well associated with the eutectic and liquidus temperature, and little undercooling was observed on cooling. In the Zn-Sn alloys, primary α-Zn grains were surrounded with β-Sn/α-Zn eutectic phase, in which fine Zn platelets disperse in a β-Sn/α-Zn matrix. The Zn-30In alloy did not find out Zn platelets in a porous β-In/α-Zn matrix. From the tensile test of alloys, UTS and 0.2% proof stress were not much difference but the elongation was slightly decreased as increasing Zn contents in the Zn-Sn alloys, showing ductile fracture mode. The Zn-30In alloy show lower tensile properties and seriously degraded as time goes under thermal and humidity conditions.

5:10 PM

Interfacial Reactions in the Joint of 80Au-20Sn/58Bi-42Sn Combination Solder: Chang Joon Yang[1]; Moon Gi Cho[1]; *Hyuck Mo Lee*[1]; [1]KAIST

The 80Au-20Sn/58Bi-42Sn combination solder is proposed for multichip module packaging. The bonding process between the 80Au-20Sn solder bump and the 58Bi-42Sn solder has been conducted at 200°C for 2 min. The intermetallic phases of $(Au,Ni)_3Sn_2$, Ni_3Sn_4 and $AuSn_4$ were observed to form at the interface between solder and Ni UBM, and the $AuSn_2$ phase together with a thin inner layer of AuSn was observed at the combination interface. The interfacial reactions were explained through thermodynamic considerations. The ball shear and lap shear tests after multiple reflows have also been performed and their behavior will be explained.

5:35 PM

Comparison of Sn-2.8Ag-20In and Sn-10Bi-10In Solders for Intermediate Step Soldering: *Sun-Kyoung Seo*[1]; Hyuck Mo Lee[1]; [1]KAIST

Sn-2.8Ag-20In and Sn-10Bi-10In (in wt.%) have been chosen as Pb-free solder materials for intermediate step soldering. Reactions between two solders and under bump metallugy (UBM) Au/Ni (Au: 1.5 µm, Ni: 3 µm) were investigated at 210, 220, 230 and 240°C for up to 4 min. All of the Au UBM was dissolved into the solder matrix as soon as the reaction started and resulted in formation of $Au(In,Sn)_2$ in the case of SnAgIn and $Au(In,Sn)_2$ as well as $Au(In,Sn)_4$ in SnBiIn. The exposed Ni layer reacted with the solder and formed $Ni_3(In,Sn)_4$ in both solders. The formation mechanism of intermetallic phases was thermodynamically explained. The growth rate of the interfacial $Ni_3(In,Sn)_4$ phase in SnBiIn was faster than that in SnAgIn. In the shear strength test, the shear strength values of both solders were similar.

Phase Transformations in Magnetic Materials: Magnetic Shape Memory Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Raju V. Ramanujan, Nanyang Technological University; William T. Reynolds, Virginia Tech; Matthew A. Willard, Naval Research Laboratory; David E. Laughlin, Carnegie Mellon University

Tuesday PM — Room: 213A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Michael McHenry, Carnegie Mellon University; Gernot Kostorz, ETH Zurich

2:00 PM Invited
Magnetic and Martensitic Transformation of Ni-(Co,Fe)-(Al,Ga) β Alloys: *Katsunari Oikawa*[1]; Ryosuke Kainuma[1]; Kiyohito Ishida[1]; [1]Tohoku University

Magnetic and martensitic phase transformation of the Ni-Co-Al, Ni-Fe-Al, Ni-Co-Ga and Ni-Fe-Ga systems were systematically investigated. The composition region of the β phase showing the shape memory effect in the ferromagnetic state of these systems was clarified. The spontaneous magnetization of these ferromagnetic shape memory alloy systems showed linear dependence with the magnetic valence introduced in the generalized Slater-Pauling curve. The β phase transformed from B2 to 2M structures in the Ni-Co-Al, Ni-Fe-Al and Ni-Co-Ga systems, and from $L2_1$ to 10M or 14M structures in Ni-Fe-Ga systems. In addition, the plastic workability of these alloy systems could be improved by the introduction of a small amount of the ductile γ (fcc structure) phase with control of the chemical composition and annealing temperature because the composition region of the ferromagnetic shape memory alloys in these systems appears near the β+γ two-phase region. This characteristic is one of advantage for practical application.

2:35 PM
Martensite Transformation in Ni-Mn-Ga Ferromagnetic Shape-Memory Alloys: *Marc L. Richard*[1]; Jorge C. Feuchtwanger[1]; Patricia Lázpita[2]; Samuel M. Allen[1]; Robert C. O'Handley[1]; Manu Barandiarán[2]; [1]Massachusetts Institute of Technology; [2]Universidad del Pais Vasco/EHU

The crystal structure of Ni-Mn-Ga ferromagnetic shape-memory alloys is extremely sensitive to composition. Several martensitic structures including tetragonal (5-layered), orthorhombic (7-layered) and non-modulated tetragonal have been observed. A systematic exploration of the composition-structure relationship has been performed using x-ray diffraction on several single crystals with different compositions. Temperature-dependent magnetic measurements have revealed markedly different transformation behavior in the tetragonal and orthorhombic materials. The orthorhombic material shows a much larger difference between the martensite start and finish temperatures as compared to tetragonal martensite. The martensitic transformation processes have been observed with in-situ TEM and the defect structure present in each phase has been explored. Single-crystal and powder neutron diffraction have been employed to study the chemical ordering in the austenite and martensite phases. The difference in transformation character can be explained with a thermodynamic model by including the difference in the strain energy contribution for the two different martensite phases.

3:00 PM Invited
Influence of Composition and Microstructure on the Magnetic Properties of Ni-Fe-Ga Ferromagnetic Shape Memory Alloys: *Todd M. Heil*[1]; Matthew A. Willard[2]; William T. Reynolds[1]; [1]Virginia Tech; [2]Naval Research Laboratory

The martensite and magnetic transformations in Ni-Fe-Ga ferromagnetic shape memory alloys are very sensitive to alloy chemistry and thermal history. A series of Ni-Fe-Ga alloys near the prototype Heusler composition (X_2YZ) were prepared, and a $Ni_{53}Fe_{19}Ga_{28}$ alloy was subsequently annealed at various temperatures below and above the B2/$L2_1$ ordering temperature. Calorimetry and magnetometry were employed to measure the martensite transformation temperatures and Curie temperature. Compositional variations of only a few atomic percent result in martensite start temperatures and Curie temperatures that differ by about 230 K degrees and 35 K degrees, respectively. Various one-hour anneals of the $Ni_{53}Fe_{19}Ga_{28}$ alloy shift the martensite start temperature and the Curie temperature by almost 70 K degrees. Transmission electron microscopy investigations were conducted on the annealed $Ni_{53}Fe_{19}Ga_{28}$ alloy. The considerable variations in the martensite and magnetic transformations in these alloys are discussed in terms of microstructural differences resulting from alloy chemistry and heat treatments.

3:35 PM Invited
Analysis of Magnetoelastic Composites: *Perry H. Leo*[1]; [1]University of Minnesota

We consider the properties of high volume fraction magnetoelastic composites. Our analysis depends on new results related to Eshelby's inclusion problem. Specifically we prove the existence of special inclusions that induce uniform magnetic fields when they are uniformly magnetized under periodic boundary conditions. Similar properties hold for linear elasticity. These inclusions are in some sense the high volume fraction equivalent to isolated ellipsoidal inclusions under uniform magnetic field (or, in the elastic case, eigenstrain). By using these special inclusions we are able to estimate the properties of high volume fraction composites. We discuss the implications of our results to the Eshelby conjecture and to theconstruction of energy minimizing microstructures. This work is joint with R. D. James and Liping Liu.

4:10 PM Break

4:25 PM Invited
Martensitic and Magnetic Transformations of the Ni-Mn-Based Ferromagnetic Shape Memory Alloys: *Ryosuke Kainuma*[1]; Yuji Sutou[1]; Katsunari Oikawa[1]; Kiyohito Ishida[1]; [1]Tohoku University

Ferromagnetic shape memory alloys (FSMAs) such as Ni2MnGa and NiCoAl have attracted considerable attention as a new type of magnetic actuator materials. Recently, the present authors have reported some Ni-Mn based FSMAs in NiMnX (X: In, Sn and Sb) systems[1]. In the present paper, characteristic features on the martensitic and magnetic transformations of these NiMn-based FSMAs will be reported. [1]Sutou et al., Appl. Phys. Lett., 85(2004)4358.

5:00 PM Invited
Martensitic Transformations and Magnetic Shape Memory in Heusler Alloys: Gernot Kostorz[1]; Myriam Aguirre[1]; Debashis Mukherji[1]; *Peter Mullner*[2]; [1]ETH Zurich, Applied Physics; [2]Boise State University, Materials Science and Engineering

In L21-type ordered body-centered cubic alloys (Heusler alloys), martensitic transformations lead to various low-symmetry, non-modulated (tetragonal) or long-periodic modulated (10M and 14M) structures. Twinning rather than dislocation motion provides the lattice-invariant shear in most cases. Alloys around the stoichiometric composition Ni_2MnGa have recently attracted major interest because of their ferromagnetic order. Ferromagnetism in the martensitic phase provides a magnetically-induced mechanism for shape change (also called magnetic shape memory), if the crystal anisotropy leads to a sufficient driving force to move twin boundaries. Details of the size and reproducibility of shape changes under the influence of magnetic fields depend very sensitively on the microstructure. Polysynthetically twinned single crystals and polycrystals have been investigated, with a maximum strain of 10% for the 14M structure. Results on magnetomechanical properties will be presented and related to microstructural features obtained from electron diffraction and conventional as well as high-resolution electron microscopy.

5:35 PM
Phase Field Modeling and Simulation of Microstructure Evolution in Ferromagnetic Shape Memory Alloys: *Yongmei M. Jin*[1]; [1]Texas A&M University

The unusually large magnetomechanical responses of ferromagnetic shape memory alloys are correlated with the coupled magneto-elastic domain evolutions in these multiferroic materials. The ferromagnetic and ferroelastic domain structures actively respond to the change in external magnetic field, applied stress, and temperature. The kinetic pathways of domain evolutions play the key role in determining the materials properties. Even the basic mechanism of magnetic-field-induced shape memory effect is understood, little quantitative details of the processes are known. Phase Field model could be a valuable tool to understand these processes. It takes into account the strain and magnetic coupling of the structural and magnetic domains and their interactions with external magnetic field and applied stress. This method can be employed to investigate the quantitative details of magnetic and strain responses to the applied fields and to determine the main factors affecting the ferromagnetic shape memory effect. Ongoing computational effort will be discussed.

Point Defects in Materials: Other Diffusion

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Dallas R. Trinkle, U.S. Air Force; Yuri Mishin, George Mason University; David N. Seidman, Northwestern University; David J. Srolovitz, Princeton University

Tuesday PM — Room: 210B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Irina V. Belova, University of Newcastle

2:00 PM Invited
Diffusion and Viscous Flow in Bulk Glass Forming Alloys: *Franz Faupel*[1]; Volker Zoellmer[1]; Alexander Bartsch[1]; Klaus Raetzke[1]; [1]Kiel University

We review radiotracer diffusion and isotope measurements in bulk glass forming alloys from the glassy state to the equilibrium melt and compare diffusion and viscous flow. In the glassy as well as in the deeply supercooled state below the critical temperature Tc, where the mode coupling theory predicts a freezing-in of liquid-like motion, very small isotope effects indicate a highly collective hopping mechanism. Even in the supercooled state below Tc the temperature dependence is Arrhenius-type with an effective activation enthalpy, and diffusion is clearly decoupled from viscosity. Above Tc the onset of liquid-like motion is evidenced by a gradual drop of the effective activation energy and by the validity of the Stokes-Einstein equation. This strongly supports the mode coupling scenario. The isotope effect measurements show atomic transport up to the equilibrium melt to be far away from the regime of uncorrelated binary collisions.

2:30 PM
Kinetic Monte Carlo Simulation of Cesium Migration in UO2: *Thomas A. Patten*[1]; Chaitanya S. Deo[2]; Blas P. Uberuaga[2]; S. G. Srivilliputhur[2]; James F. Stubbins[1]; Stuart A. Maloy[2]; [1]University of Illinois; [2]Los Alamos National Laboratory

The migration of Cs in uranium dioxide, and its consequent segregation, is important to understand the operational containment of fission products. We have developed a kinetic Monte Carlo (kMC) model to study the long time evolution of Cs migration and defect clustering. In this model, the fission product Cs migrates on the Uranium sub-lattice with assistance of vacancy type defects. Atomistic models based on the Buckingham potential are used to obtain the catalog of Cs mechanisms, the energy barriers to their diffusion, as well as other point defect migration and cluster binding energies, in stoichiometric and non-stoichiometric UO2. Using our kMC model, we calculate the self-diffusion coefficient for point defects and the survival rate of defect clusters. The Cs self-diffusion coefficient is predicted for various system temperatures.

2:50 PM Invited
Defects and Self-Diffusion Mechanisms on SiO2: An Ab-Initio Study: *Yves Limoge*[1]; Guido Roma[1]; [1]Commissariat à l'Energie Atomique

Either as a pure material or as the main component of oxides alloys, SiO2 plays an inceasing role in various properties demanding technological fields. Here the atomic scale understanding of material properties becomes more and more the required level. In this work we will present the results of a study of defect formation and migration energies in crystalline and amorphous SiO2, using ab-initio methods in the framework of Density Functional Theory. We review the difficulties in predicting the absolute equilibrium concentration and charges of each type of defect (Si/O vacancies and interstitials) in real materials as a consequence of impurity concentrations. We discuss also the main self-diffusion mecanisms that, from our calculations, turn out to be dominant in specific conditions.

3:20 PM
Microscopic versus Macroscopic Activation Barriers for Diffusion in Solid Solutions: *Ramanathan Krishnamurthy*[1]; Mikhail Mendelev[2]; David J. Srolovitz[1]; Roberto Car[1]; [1]Princeton University; [2]Ames Laboratory

Diffusion in solid solutions is rich with varied physical effects, occurring on disparate length/time scales. At a microscopic level, individual atom/vacancy exchanges depend intimately on the local environment surrounding the exchanging pair. An accurate calculation of the activation energy for this exchange requires the use of density functional theory methods (or molecular statics where reliable potentials are available). The relevant length/time scale is nm/ps. Several atom-vacancy exchanges with differing microscopic energy barriers combine to give macroscopic diffusion, the activation energy for which is generally different from any of the exchange energies. Depending on the type of solid solution under investigation, the simulation size/time required to accurately predict long-time diffusivities is 10-1000 nm / 100 - 10^6 ns or even more. Kinetic Monte Carlo methods, with only the microscopic rates as input are eminently suited to address these time/length scale requirements. Here, we present two technologically important examples, oxygen diffusion in yttria stabilized zirconia and Fe impurity diffusion in Al where such a combination of simulation techniques has been employed to successfully predict modeling complex diffusion phenomena. Interesting physical effects that occur as the disparate time/length scales are bridged and their relevance in explaining experimental observations will be discussed.

3:40 PM Break

3:55 PM Invited
Using Molecular Dynamics Techniques for Simulations of Grain Boundary Diffusion and Mobility: *Diana Farkas*[1]; [1]Virginia Tech

In this talk we will discuss the use of large scale molecular dynamics techniques to study the mechanisms of diffusional transport along grain boundaries as well as grain boundary migration. Results are presented of molecular dynamics simulations of the diffusion process in ordered B2 NiAl at high temperature using an embedded atom interatomic potential. In this material, diffusion occurs through a variety of cyclic mechanisms that accomplish the motion of the vacancy through nearest neighbor jumps restoring order to the alloy at the end of the cycle. MD allows the study of the detailed time evolution of the jump sequence in these cyclic mechanisms, both in the bulk and along the grain boundary. A similar technique is used to study the process of grain boundary motion in a pure Ni digital sample containing initially 15 grains.

4:25 PM
Vacancy Formation and Migration Energies in Nonequilibrium Grain Boundaries: *Airat A. Nazarov*[1]; Ramil T. Murzaev[2]; [1]Ufa State Aviation Technical University; [2]Russian Academy of Sciences

Grain boundaries (GBs) in plastically deformed metals have a nonequilibrium structure characterized by the presence of extrinsic dislocations and disclinations. Such GBs are important elements of the structure of nanocrystals prepared by severe plastic deformation. In order to understand underlying mechanisms of diffusion along nonequilibrium GBs, vacancy formation and migration energies in these GBs are calculated by atomistic simulations based on the embedded atom method. Two types of nonequilibrium GBs containing (a) extrinsic grain boundary dislocations (EGBDs) and (b) wedge disclinations are studied. GBs subjected to uniform tensile stress are also considered. The uniform tensile stress and long-range stresses of extrinsic dislocations are shown not to influence significantly the characteristics of vacancies. Up to about 0.2 eV change of both the vacancy formation and migration energies is caused by disclinations on a large distance from the disclination core. Implications of the results to the diffusion in nanocrystals are discussed.

4:45 PM Invited
Segregation, Pre-Melting, and Melting Phenomena in the Cu-Bi System Studied by Radiotracer Grain Boundary Diffusion: *Sergiy V. Divinski*[1]; Christian Herzig[1]; Maik Lohmann[1]; Boris Straumal[2]; [1]University of Muenster; [2]Institute of Solid State Physics

Copper embrittlement by Bi is a typical example of a severe detrimental phenomenon in materials science. Strong segregation of Bi can induce significant changes in grain boundary structure and even cause a liquid-like film at the grain boundaries. The recent results on grain boundary diffusion of Cu-64 and Bi-207 in Cu-Bi alloys are presented. The measurements are performed in the single-phase (solid solution) as well as in the two-phase (solid + liquid) regions of the equilibrium phase diagram. Above certain temperature, the grain boundary diffusivities of both Cu and Bi in the two-phase region are by orders of magnitude higher than those in pure Cu. An abrupt increase of the diffusivities was observed at certain Bi contents which are unequivocally in the single-phase region. The present results convincingly showed the occurrence of the premelting phase transition in GBs of the Cu-Bi system.

Polymer Nanocomposites: Session II

Sponsored by: The Minerals, Metals and Materials Society
Program Organizer: Devesh K. Misra, University of Louisiana at Lafayette

Tuesday PM March 14, 2006 — Room: 217A — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jyoti Prakash Jog, National Chemical Laboratory; Quan Yuan, University of Illinois

2:00 PM Invited
Polymer Nanocomposites: Crystallization and Polymorphism Studies: *Jyoti Prakash Jog*[1]; [1]National Chemical Laboratory

Polymer nanocomposites is an emerging area of research and is of particular interest to scientists because of the molecular level interaction of the fillers with polymer chains. The nanocomposites may be comprised of nanoparticles, nanotubes or nanoclays. The dimensions of the second phase are close to the chain dimensions, and a number of studies are aimed to elucidate the effect of nano size filler on the crystallization of polymers. An attempt is made to address the few aspects of crystallization in polymer nanocomposites. We will be discussing the effect of the second phase in nano- dimension on the various aspects of crystallization such as isothermal crystallization, non-isothermal crystallization, spherulitic growth, phase transformation, polymorphism etc. The polymers, which are studied, include Polybutene and Polyvinylidene fluoride. The effect of type of filler and its concentration is investigated through isothermal crystallization, optical microscopy and X-ray diffraction.

2:30 PM Invited
Structural and Mechanical Characterization of Polymer Nanocomposites: *Xiaodong Li*[1]; [1]University of South Carolina

The extremely small dimensions of nanoscale reinforcements in polymer nanocomposites such as carbon nanotubes, nanoclays, and nanoparticles impose a great challenge for many existing characterization techniques and instruments. To understand and establish the reinforcing mechanisms for polymer nanocomposites, the morphology, dispersion and orientation of the reinforcements as well as the bonding between the reinforcements and matrix need to be well characterized. The nanoscale structure of single-walled carbon nanotube-reinfored epoxy composites, nanoclay-reinforced agarose composites, bamboo-like polymer/silicon nanocomposites, and natural seashell nanocomposites such as the shells of red abalone and pectinidae was studied by atomic force microscopy (AFM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Their mechanical properties were measured by nanoindentation, nanoscratch, and tensile and bending tests. The reinforcing mechanisms are discussed in conjunction with their structure and mechanical properties such as hardness and elastic modulus. The strengthening and toughening mechanisms of natural nanocomposites are also presented.

3:00 PM
Photocatalytic and Antimicrobial Active Polymer Nanocomposites Membrane for Water Purification: *Subhasis Rana*[1]; Jagdish Rawat[1]; Melinda Sorensson[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

Titania-coated nickel ferrite nanoparticles have been successfully dispersed in a porous polymer membrane with consequent formation of a hybrid polymer nanocomposite structure. This porous polymeric membrane acts as a filter for water purification characterized by photocatalytic and antimicrobial activity. Titania contributes to antimicrobial activity, while nickel ferrite facilitates removal of the membrane with the help of small magnetic field. The performance of the hybrid structure and the factors that influence its performance are discussed.

3:25 PM
Micromechanism of Stress Whitening during Tensile Deformation in Polymer Nanocomposites: *Raghunath R. Thridandapani*[1]; Harish Nathani[1]; Jyoti Prakash Jog[2]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette; [2]National Chemical Laboratory

The micromechanism and susceptibility to stress whitening during tensile deformation of polymer nanocomposites is studied by electron microscopy and compared with the unreinforced neat polymer. Mineral-reinforced composites exhibit significantly reduced susceptibility to stress whitening, and are characterized by lower gray level in the plastically deformed stress whitened zone. This behavior is attributed to the effective reinforcement of polymer by the mineral and the nucleating effect. Additionally, reinforcement of polymer alters the primary micromechanism of stress whitening.

3:50 PM
Processing and Property Optimization of Polycarbonate Matrix-Carbon Nanotube Reinforced Composites: *Jerimiah E. Robbins*[1]; [1]New Mexico Institute of Mining and Technology

We investigate multi-walled carbon nanotubes as reinforcement in polycarbonate matrix composites. Experimental data show relevant processing-property relationships via glass transition measurements, tensile testing, TEM imaging and SEM fractography. Addressed are processing issues such as dispersion, interfacial adhesion, and forming operations. Based on statistical data from designed experiments, we will discuss optimum processing parameters of the polymer nanocomposites.

4:15 PM Break

4:25 PM
The Relaxation Behavior of Polymer Nanocomposites: A Study of Dynamic Mechanical Analysis: *Qiang Yuan*[1]; S. Awate[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

The effects of nanoparticles on the relaxation behavior of polymer matrix are studied with dynamic mechanical analysis (DMA). In order to elucidate the mechanism of the effect of nanoparticles on the mechanical properties of polymer nanocomposites, the results of DMA combined with micromorphology of SEM and TEM are presented.

4:50 PM
Thermal and Mechanical Properties of PMMA/SiO_2 Nanocomposites with Controlled Particle/Matrix Interfaces: *Sarah L. Lewis*[1]; Chunzhao Li[1]; Robert G. Shimmin[2]; Brian C. Benicewicz[1]; Paul V. Braun[2]; Linda S. Schadler[1]; [1]Rensselaer Polytechnic Institute; [2]Univeristy of Illinois at Urbana-Champaign

In polymer/nanoparticle composites particle size and surface chemistry control the mechanical and thermal properties. Previous work has suggested that the addition of small particles can change the glass transition temperature and the addition of larger particles alters the ductility of the final composite. The "strength" of the interface controls the direction of change of the properties relative to the neat PMMA. To test these hypotheses and hence to better understand the physics of polymer chains adjacent to nanoparticle surfaces, SiO_2/PMMA composites have been made using monodisperse particles of two sizes, 15 and 150 nm. Different nanoparticle surface treatments to provide different "strengths" of particle/matrix interface were used. Preliminary results show that composites made with smaller particles with a weak interface (fluorinated) have a lower T_g than the neat polymer whilst those made with larger particles with the same surface chemistry show no change in T_g but do increase the ductility.

5:15 PM
On the Impact Strength of Clay Reinforced Polymer Nanocomposites: Mahesh Tanniru[1]; Raghunath Rao Thridandapani[1]; *Samrat Awate*[1]; Q. Yuan[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

The micromechanism of plastic deformation during impact loading of polymer nanocomposites is investigated through electron microscopy and atomic force microscopy and the behavior compared with the un-reinforced polymer under identical conditions of processing. The impact strength of composites is linked to structural studies by X-ray diffraction and atomic force microscopy observations. The adoption of clay in polymers has two primary effects: the reinforcement and the nucleating effect. The reinforcement effect generally increases the bulk crystallinity and modulus, while the nucleating effect decreases the spherulite size. The extent to which impact strength is retained on reinforcement depends on the polymer matrix.

Recycling - General Sessions: Electronics Recycling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling Committee
Program Organizers: Gregory K. Krumdick, Argonne National Laboratory; Cynthia K. Belt, Aleris Intl

Tuesday PM Room: 8B
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Gregory K. Krumdick, Argonne National Laboratory

2:30 PM Introductory Comments

2:35 PM
Metallurgical Recycling of Electronic Scrap: *Birgit Matl*[1]; Helmut Antrekowitsch[1]; Christine Wenzl[1]; Franz Prior[2]; Walter Spruzina[2]; [1]University of Leoben; [2]Centre of Excellence for Electronic Scrap Recycling and Sustainable Development

Nowadays, the pyrometallurgical treatment in copper smelters is the common process for the recycling of metals from the electronic scrap. The European Directive on the Waste from Electrical and Electronic Equipment (WEEE) and the Directive on the Restriction of Hazardous Substances (RoHS), increase the importance of electronic scrap recycling. A high amount of plastic and inert materials in these materials leads to problems at the processing. An optimisation concerning the internal recovery and the high amount of plastic is necessary to reach the recycling quota of the European directive. The majority of scrap is treated mechanically in order to separate plastics from metals. The quality of the different fraction often rather low and further treatment leads to higher costs. The Christian Doppler Laboratory for Secondary Metallurgy of Nonferrous Metals and the Institute of Nonferrous Metallurgy at the University of Leoben carried out investigations concerning the optimisation of the metallurgical recycling.

3:00 PM
Examining the Eco-Efficiency of Recycling Alternatives: A Case Study on End-of-Life Cathode Ray Tubes: *Jeremy Gregory*[1]; Jennifer Atlee[1]; Jaco Huisman[2]; Ab Stevels[2]; Randolph E. Kirchain[1]; [1]Massachusetts Institute of Technology; [2]Delft University of Technology

The disposal of end-of-life electronics is an issue receiving increasing scrutiny. This paper evaluates several alternatives for recycling cathode ray tubes (CRTs) within monitors and televisions with particular emphasis on their leaded glass. The primary recycling options for the glass, reuse in new CRTs or as a smelter flux, are examined from economic and environmental standpoints. The economics are modeled using a process-based cost model (PBCM) and include dependencies on processing technologies, transportation logistics, and incoming material composition. The environmental consequences are modeled using several methods including QWERTY, a framework that has been developed specifically to examine the recyclability of electronic products. Results are presented mapping out preferred processing alternatives across a range of technological and operational conditions using several evaluation methods. The modeling framework and results provide insights useful to stakeholders such as original equipment manufacturers, recyclers, and policy-makers.

3:25 PM
Filtration of Solar Cell Silicon Scrap: *Anne Kvithyld*[1]; Dilip Chithambaranadhan[1]; Arjan Ciftja[1]; Eivind Johannes Øvrelid[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology; [2]SINTEF

Silicon solar cells are attractive sources of energy. Recycling scrap from top and sides of ingots of multi crystalline silicon is addressed. SiC and Si3N4 inclusions are a major problem. Scrap from Scan Wafer ASA is melted in a high vacuum furnace at around 1500ºC. The filters, from commercial producers, are C and SiC with 10, 20 and 30 ppi. Characterisation of inclusions before and after filtration, and in the filter itself, is performed by microscopy. Dissolution of silicon in acid and counting of the un-dissolved inclusions is also carried out. In the filter Si3N4 needles and SiC inclusions were identified. The amount of these inclusions in the filtered silicon is much less than in the input. In conclusion, filtration using SiC and C filters in vacuum system in argon atmosphere removes SiC and Si3N4 inclusions from solar cell scrap. Flow of metal through the filters is not a problem.

3:50 PM
Leaching of Copper from Waste Printed Circuit Boards Using Electro-Generated Chlorine in Hydrochloric Acid Solution: *Eun-Young Kim*[1]; Min-Seuk Kim[2]; Jae-Chun Lee[2]; Jin-Ki Jeong[2]; Byung-Su Kim[2]; [1]University of Science and Technology; [2]Korea Institute of Geoscience and Mineral Resources

Electro-generated chlorine leaching of waste printed circuit boards was investigated in hydrochloric acid solutions. The pulverized printed circuit board contained about 45% of metal component, in which copper was about 84%. The current efficiency of chlorine generation was calculated by measuring the volume and composition of the generated gas. The leaching rate of copper was greatly affected by current density and agitation speed. The Leaching of copper up to 98% was achieved at 20 mA/cm2, 50°C, 180 minutes, and 600 rpm in 1M HCl solution. Increasing agitation and lowering current density enhanced utilization efficiency of electro-generated chlorine. Leaching of copper was suppressed at the initial stage, while the minor metal elements, such as aluminum, lead, and tin, were dominantly leached out.

4:15 PM Break

4:30 PM
Recovery of Tellurium from Retired Cadmium Telluride Photovoltaic Modules and Other Sources: *Wenming Wang*[1]; Vasilis M. Fthenakis[1]; [1]Brookhaven National Laboratory

Recycling of retired cadmium telluride (CdTe) photovoltaic (PV) modules has attracted attention due to new European environmental regulations. Recycling tellurium from CdTe PV modules makes economical sense because of the low availability of this metal. We investigated several possible treatment paths for reclaiming tellurium from leaching solution generated from processing CdTe PV modules. Our experiments showed that conventional neutralization/hydrolysis methods recovered only 50~60% of tellurium. However, by appropriately combining neutralization and sulfide precipitation, all the tellurium present in the leaching solution was precipitated and recovered, and the concentration of tellurium in the processed solution was reduced from 900-1000 mg/L to less than 5 mg/L, yielding a tellurium recovery of more than 99%. This method can be incorporated in the scheme of recycling spent CdTe PV modules and manufacturing wastes. It may be also an alternative to the current operations for removing tellurium from copper slimes leaching solutions.

4:55 PM
Recovery of Metals from Spent Batteries: *Masao Miyake*[1]; Masafumi Maeda[1]; [1]University of Tokyo

A recovery process of metals from spent nickel-metal hydride batteries was investigated. The batteries contain nickel hydroxide as positive electrode and hydrogen storage alloy comprising rare earth metals, nickel, cobalt, manganese, and aluminum as negative electrode. A leaching treatment with an ammoniacal solution was developed to remove the hydroxide and recover the alloy from their mixture. By the leaching treatment, only the hydroxide was dissolved in the solution and the alloy could be recovered without decomposition.

5:20 PM
A Clean-Lead Factory for Lead Batteries Recycling is Available by Means of the "Cleanlead" Process: *Carlos Frias*[1]; Nuria Ocaña[1]; Gustavo Diaz[1]; Tony Piper[2]; Brunon Bulkowski[3]; Andrzej Chmielarz[4]; Peter A. Claisse[5]; Steve Hemmings[6]; Luisa M. Abrantes[7]; Herman J. Jansen[8]; Joost van Erkel[9]; Ton Franken[10]; Zdenek Kunicky[11]; Teodor Velea[12]; [1]Tecnicas Reunidas S.A.; [2]Britannia Refined Metals Limited; [3]Orzel Bialy, S.A.; [4]Institute of Non-Ferrous Metals; [5]Coventry University; [6]Lafarge Plasterboard Ltd; [7]Universidade de Lisboa; [8]Magneto Special Anodes B.V.; [9]TNO - MEP; [10]Membraan Applicatie Centrum Twente; [11]Kovohute Pribram a.s.; [12]Institute for Non-Ferrous and Rare Metals

The lead-battery recycling is a major industrial activity (more than four million tonnes recycled lead per year) that generates large amounts of toxic wastes such as lead slag, mattes and acidic sludge, besides airborne emissions. This is a waste-producer industry that needs a definitive solution to avoid its negative environmental impact and getting a sustainable lead production. That is the principal aim of the CLEANLEAD technology, which properly combines both hydrometallurgical and pyrometallurgical routes aiming to get zero-waste production, reducing the use of energy and resources in all phases of battery reprocessing, and consequently greatly decreases the operating cost. There will be no toxic emissions to land, water or air, and obtained products are pure lead or pure lead oxide, and commercial gypsum instead of waste sludge. Obtained results and achievements at pilot plant level are very satisfactory, opening the way to further developments and potential applications.

5:45 PM
Cost Analysis of Used Battery Recycling in Taiwan: *Esher Hsu*[1]; Chenming Kuo[2]; [1]National Taipei University; [2]I-Shou University

In Taiwan, around 9,000 tons of batteries are consumed per year but only 10% of which were collected and transported to oversea for recovery. The dumping used batteries have become a potential environment problem. How to increase the collection efficiency of used batteries becomes an important issue for EPA in Taiwan. Besides, some treatment plants are on process to get permission for recovering used batteries. The objective of this study is to estimate the recycling cost of spent consumer battery and explore the feasibility of recovering used consumer batteries in Taiwan based upon the principle of cost-efficiency. The recycling cost including collection cost and recovery is estimated based on a survey. Results would provide some suggestions to Taiwan EPA regarding feasibility of recovering used batteries in Taiwan.

6:10 PM Concluding Comments

Simulation of Aluminum Shape Casting Processing: From Alloy Design to Mechanical Properties: Through Process Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Aluminum Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Process Modeling Analysis and Control Committee, TMS: Solidification Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Qigui Wang, General Motors Corporation; Matthew Krane, Purdue University; Peter Lee, Imperial College London

Tuesday PM Room: 6D
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: John T. Berry, Mississippi State University

2:00 PM Invited
Virtual Aluminum Castings: The Ford Experiment in Integrated Computational Materials Engineering: *John E. Allison*[1]; [1]Ford Research and Advanced Engineering, Ford Motor Company

This talk will describe Virtual Aluminum Castings, a comprehensive, integrated and experimentally verified suite of CAE tools for optimization of cast aluminum components and processes. Using VAC castings can be designed and virtually cast, heat treated and tested for durability, all on a workstation long before components are fabricated. VAC represents the Ford experiment in a new approach to component engineering, Integrated Computational Materials Engineering (ICME). ICME eeks to unify analysis of manufacturing, design and materials into a holistic system and to unify materials models across length and time scales and knowledge domains. This talk will review recent progress in ICME and, more specifically, our ability within VAC to integrate 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.

2:25 PM Invited
Study on Macro and Micro-Modeling of Solidification Process of Aluminum Shape Casting: *Baicheng Liu*[1]; Shoumei Xiong[1]; Tao Jing[1]; Qingyan Xu[1]; [1]Tsinghua University

Numerical methods to improve the computational efficiency and to extend the computational scale of the mold filling and solidification of aluminum die casting process were studied. For molding filling simulation, parallel computation method was studied, while for solidification simulation, an implicit finite difference scheme and a transient surface layer concept were studied. Three-dimensional simulation of the dendritic growth of multiple grains for aluminum alloy was studied by using phase field method coupling with thermal noise. In addition, modified cellular automaton (MCA) method was used to simulate the microstructure formation and evolution of Al alloy, including the grain structure and the dendritic microstructure and arm branching. Based on microstructure simulation, the mechanical properties were also predicted. The experimental results show that the models in the paper are reasonable for describing the formation and evolution of the dendritic microstructure.

2:50 PM Invited
A Mathematic Model to Predict the Cyclic Stress State of an A356 Automotive Wheel and Its Application to Fatigue Life Evaluation: Peifeng Li[1]; *Daan M. Maijer*[2]; Peter D. Lee[1]; Trevor C. Lindley[1]; [1]Imperial College; [2]University of British Columbia

Fatigue life is a key consideration in the design of cast aluminium alloy automotive wheels which offer both improved strength-to-weight ratio and fuel efficiency. The cyclic in-service (applied load and residual) stress distribution within a wheel is a limiting factor determining a wheel's fatigue performance. In this investigation, a through-process modelling methodology was applied to predict the cyclic stress state of an A356 automotive wheel subject to a bending moment. The predicted residual stresses arising during heat treatment and released through machining were

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validated by a destructive strain measurement technique. With the residual stress state used as an initial condition, the variation of the in-service stress state during a bending test was predicted. The measured strain variations within the wheel for a series of different bending loads was found agree with the predictions. The through process modelling approach is completed by assessing the fatigue performance of the wheel.

3:15 PM Invited

Modeling and Simulation of Semisolid Casting: *Gilmer R. Burgos*[1]; [1]Polytechnic University of Puerto Rico

Processing of metal alloys in their mushy state represents a new trend in metal processing. The process produces components with low porosity, high crack resistance, and superior mechanical properties. However, its implementation to industrial applications is hampered by technical problems due to the complex rheology of the material. The material behaves differently under slow and rapid transients and its response is time-dependent. In the present study, the thixotropic behavior of semisolid slurries is modeled using conservation equations and the Herschel-Bulkley fluid model. The model is implemented into a computer code to predict die filling. Results show that the final quality of the products depends on the processing conditions and the geometry of the die.

3:40 PM Break

3:55 PM Invited

Development and Applications of OPTCAST - A Thermal Boundary Conditions and Casting Process Optimization Tool: *Mei Li*[1]; John E. Allison[1]; [1]Ford Motor Company

OptCast is an integrated thermal/optimization tool coupling commercial casting simulation software MagmaSoft and ProCAST with optimization software iSIGHT. Two types of optimizations are realized in OptCast: one is using an inverse modeling approach to determine boundary conditions such as interfacial heat transfer coefficient, and the other is optimizing casting process variables by minimizing cycle time while maintaining casting quality. With OptCast heat transfer coefficients were developed from simple test casting and applied to V8 engine block for thermal analysis, microstructure and mechanical properties predictions and I4 block for porosity defect study. Heat transfer coefficients were also optimized for quenching process of cylinder head for residual stress analysis.

4:20 PM

Novel Solver Strategies for the Computational Modeling of Mould Filling in Very Complex Geometries: *Mark Cross*[1]; Nick Croft[1]; Diane McBride[1]; [1]University of Wales Swansea

Simulation of shape casting processes involves the simultaneous capture of the rapidly developing metal-air free surface and the liquid-solid moving boundary in the metal phase. Although a wide range of techniques have been developed to address this problem, there are occasions where existing techniques are limited, especially in very complex geometries where the mesh quality may inevitably be relatively poor at some key locations. These poor quality cells can seriously inhibit convergence. Here we describe a finite volume approach over unstructured meshes where the flow is solved at the cell or element vertex, and all other variables are solved at the cell centre (as in conventional CFD tools). This flow solver strategy is much more tolerant of meshes with poor quality elements and enables the solution of simultaneous free surface flows coupled with solidification in very complex meshes, thus enabling the capture of such behaviour in arbitrarily complex geometrical structures.

4:45 PM

A Thermal Model of the Low-Pressure Die-Casting (LPDC) Process and Its Application to Predict Porosity Formation in Aluminum Alloy Wheels: *Bin Zhang*[1]; Steve Cockcroft[1]; Daan Maijer[1]; Jindong Zhu[1]; [1]University of British Columbia

A mathematical model of the Low Pressure Die Casting process for the production of A356 aluminum alloy wheels has been developed to predict the evolution of temperature and the porosity formation in the wheel. The model was validated against temperatures measured at numerous locations in the die and wheel during production. A modified Niyama parameter has been employed to estimate the critical solid fraction (fsc) for liquid encapsulation and in turn, this has been used to estimate the volume fraction of porosity. Comparison of the predicted and measured porosity content within the wheel shows that the predicted porosity formation agrees reasonably well with the measurements. The application of LPDC pressurization sequence has also been discussed with respect to enhancing mass feeding to reduce porosity.

5:10 PM

Squeeze Casting Quality Control through Process Simulation: *Yun Xia*[1]; [1]SPX Contech

Squeeze casting process, because of availability of heat treatment, minimum metal flow turbulence and gas entrapment by steady bottom shot velocity, and minimum volumetric shrinkage by directional and spot solidification, has advantage over HPDC casting. It has been a very competitive process in the automotive industry. The casting quality is sensitive to the process control and gate and runner design. Usual defects found in squeeze casting production can occur during both the die-filling phase and the metal solidification phase of the process. These defects can be directly attributed to improper adjustment or lack of control for the following process parameters: metal filling velocity, temperature, dwell time, cooling pattern and casting design, etc. Some special techniques with process simulation have been used, which include pressurized runner and gate design, casting layout in the die, squeeze pin application, high thermal conductivity inserts and casting design and castability, spray and lubricant techniques.

Solidification Modelling and Microstructure Formation: A Symposium in Honor of Prof. John Hunt: Eutectic Growth

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Solidification Committee

Program Organizers: D. Graham McCartney, University of Nottingham; Peter D. Lee, Imperial College; Qingyou Han, Oak Ridge National Laboratory

Tuesday PM — Room: 6C
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: W. Kurz, Swiss Federal Institute of Technology; J. Perepezko, University of Wisconsin

2:00 PM Invited

Modeling of Cast Iron Solidification – The Defining Moments: *Doru Michael Stefanescu*[1]; [1]Ohio State University

There are many to contend that today human civilization has reached the age of engineered materials, yet the importance of iron castings continues to support the thesis that we are still in the Iron Age. Cast iron, the first man-made composite, is at least 2500 years old. It remains the most important casting material, with over 70% of the total world tonnage. This paper is a review of the mathematical models that describe the fundamentals of solidification of iron-base materials, from the seminal paper by Oldfield, the first to attempt modeling of microstructure evolution during solidification, to the prediction of mechanical properties. The latest analytical models for irregular eutectics such as cast iron as well as numerical models with microstructure output are discussed. Because of space limitations, the emphasis is on model performance rather than model formulation. The impact of solidification simulation on defect control is also discussed.

2:25 PM Invited

Columnar and Equiaxed Growth of the Eutectic in Al-Si Alloys: *Arne K. Dahle*[1]; Kazuhiro Nogita[1]; S. D. McDonald[1]; [1]University of Queensland

The Al-Si eutectic is an irregular eutectic with silicon being a faceted phase. During solidification, silicon is the leading phase of the eutectic interface. It has been shown that this eutectic system often displays equiaxed growth, such as in unmodified commercial alloys. Adding certain elements causes columnar eutectic growth to occur instead. The nucleation rate of the eutectic is expected to directly influence the eutectic spacing through its effect on the solid/liquid interfacial area, and thus the growth rate of the eutectic. Modification treatment, undertaken to refine the Al-Si eutectic in common foundry alloys, typically also changes the eutectic nucleation conditions. This paper analyses the solidification mechanisms in unmodified and modified Al-Si alloys from the perspective of columnar and equiaxed growth, particularly focussing on the roles of potent nucleant populations and solidification conditions.

2:50 PM
Secondary Ion Mass Spectroscopy Analysis of Trace Element Distribution during the Al-Si Eutectic Reaction: *Oyvind Nielsen*[1]; Christian Simensen[1]; [1]SINTEF

Additions of trace elements like Sr, Na, Sb, Ca, etc are well known to modify the plate-like Si-particles in Al-Si alloys into a benefitial morphology. However, such additions have been shown to increase the amount of porosity and change the porosity distribution. Reviewing the extensive literature on this topic in the last years, it is evident that the mechanisms for nucleation and growth of the Al-Si binary eutectic is still a matter of controversy, although a significant progress has been made in the understanding of the effect of modifying elements on the eutectic grain size. In the present work, Secondary Ion Mass Spectrocopy has been used to quantify the distribution of elements like Sr, Na, Ca, and P in Al-Si alloys with different nominal trace element contents. The results constitute new knowledge about the solubility of trace elements in Si particles and other intermetallic phases and the local segregation behaviour.

3:15 PM Invited
Spacing Selection in Ternary Eutectic Alloys: Experiments and Computer Simulations: *Markus Apel*[1]; Ingo Steinbach[1]; [1]RWTH Aachen, Access e.V.

The 1966 paper of Jackson and Hunt about eutectic growth is the fundamental work in understanding eutectic microstructures. Tying in with this tradition we will present our recent progress in understanding lamellar growth pattern formation in a ternary eutectic alloy Bi-In-Sn. In-situ observations of pattern formation in thin metallic films are performed using light microscopy. In a wide range of parameters a γ-Sn/$BiIn_2$/β-In/$BiIn_2$ lamellar sequence forms as a steady state growth mode. A scaling relation $\lambda^2 \sim 1/v$ holds even for this ternary lamellar structure. This particular stacking sequence was selected from a planar solid/liquid growth front by a series of nucleation events. The final sequence is independent of the phase of the initial planar growth front. By a combination of the in-situ technique and phase field simulations, nucleation undercooling for γ-Sn and β-In and the diffusion coefficients of Bi and Sn in the melt can be extracted from the experimental data.

3:40 PM Break

3:55 PM Invited
Phase-Field Modeling of Rapid Eutectic Growth: *Andrew Martin Mullis*[1]; James R. Green[1]; [1]University of Leeds

As the cooling rate (or conversely undercooling) is increased it becomes progressively more difficult for a eutectic front to adapt to the increasing growth rate. At high growth velocity this will lead to a breakdown of the eutectic front, often by the emergence of dendrites. However, a number of studies of undercooled bulk samples have shown that it is also possible for an anomalous, unstructured, eutectic to be formed. Structures reminiscent of this anomalous eutectic have been observed by Nestler et. al. [Physica D 138 (2000) 144] using phase field models. However, their model lacked anisotropy it was suggested that this was an artefact of the model and that if anisotropy were included dendrites would be observed instead. In this study we use phase-field modelling to analyses the effect anisotropy has upon the breakdown of eutectic solidification at enhanced growth velocities and comment upon the formation of anomalous eutectic.

4:20 PM
Modification of Semiconductor Phases in Metallic Alloys: *Simon N. Lekakh*[1]; Carl Loper[2]; [1]University of Missouri-Rolla; [2]University of Wisconsin-Milwaukee

A number of metallic alloys contain phases with semiconductor properties. A small amount of some additives can modify the shape faceted semiconductor phases into fibrous form in eutectics and into compact shape in hypereutectic alloys. The possible mechanism responsible for modification of semiconductor phases by donor type additives was described in this article. A possibility of decreasing latent heat and increasing electro conductivity of the semiconductor phases modified by donor elements follows from this mechanism. The decrease of latent heat by the addition of Na and P in Al-Si alloys was experimentally confirmed. The electro-conductivity of primary Si crystals was qualitatively analyzed by EBIC method. The suggested mechanism predicts the composition of additives which could produce the modification of semiconductor phases in different metallic alloys. The possibility of changing the shape of the primary AlSb phase in hypereutectic Al-Sb alloys by donor type additive of Te was demonstrated.

4:45 PM
Microstructural Evolution during Laser Resolidification of Fe3Ge Alloy: *Krishanu Biswas*[1]; Kamanio Chattopadhyay[1]; [1]Indian Institute of Science

The microstructural evolution of concentrated alloys is relatively less understood both in terms of theory and experiments. Laser remelting represents a powerful technique to study the solidification behaviour under controlled growth conditions. We have utilized this technique to experimentally probe the solidification and microstructural selection during solidification of concentrated Fe—25at%Ge alloys. Under equilibrium solidification condition, the alloy undergoes a peritectic reaction between B2 α_2 and it liquid leading to the formation of ordered hexagonal intermetallic phase e (DO19). In general microstructure consists of e phase and e-ß eutectic that forms due to incomplete peritectic reaction. We shall show that with increasing velocity, the solidification front undergoes a change leading to the selection of microstructure corresponding to metastable α_2/ß eutectic to α_2 dendrite + α_2/ß eutectic to non steady state doublon growth of the dendrite and finally compact seaweed patterns. We'll discuss the underlying basis for such microstructural selection.

5:10 PM
Characterization of Hypereutectic Al-Si Powders Solidified Far-From-Equilibrium: *Eren Yunus Kalay*[1]; Scott L. Chumbley[1]; Iver E. Anderson[1]; [1]Ames Laboratory, Iowa State University

Gas atomization was used to produce rapidly solidified hypereutectic Al-Si powders; Al-15Si, Al-18Si, Al-25Si, and Al-50Si (wt %), which were characterized by scanning and transmission electron microscopy (SEM, STEM) and x-ray diffraction (XRD). XRD revealed two phases (i) Si and (ii) supersaturated Al solid solution, with a comparison of measured and calculated lattice parameters. The sample powders exhibited four distinct microstructures with increasing particle diameter: microcellular Al grains, Al dendrites, coupled eutectic growth of Al and Si, and primary Si. TEM revealed a distinct nanocrystalline zone for small particles (<1µm) which may indicate solute rejection ahead of the solidification front after recalescence. The microstructures of Al-25Si particles with diameters of 2-3 µm consisted of Al cells, which were separated by thick boundaries rich in Si. Large faceted Si and fine eutectic structures were also resolved for different compositions by TEM. Support from DOE-BES, contract no. W-7405-ENG-82.

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Surfaces and Interfaces in Nanostructured Materials II: Liquid Phase and Biological Interactions

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
Program Organizers: Sharmila M. Mukhopadhyay, Wright State University; Narendra B. Dahotre, University of Tennessee; Sudipta Seal, University of Central Florida; Arvind Agarwal, Florida International University

Tuesday PM March 14, 2006 — Room: 209 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Sharmila M. Mukhopadhyay, Wright State University

2:00 PM
Chitosan-Hydroxyapatite Nanocomposite Coatings for Biomedical Applications: *Igor Zhitomirsky*[1]; Xin Pang[1]; [1]McMaster University

New method has been developed for the fabrication of nanocomposite chitosan-hydroxyapatite (HA) coatings. The method is based on the electrophoretic deposition of HA nanoparticles, prepared by a chemical precipitation technique, and electrochemical deposition of chitosan macromolecules. The deposit composition can be varied by the variation of HA concentration in chitosan solutions. X-ray studies revealed the preferred orientation of HA nanoparticles in the nanocomposites. Nanocomposite coatings were obtained on stainless steel, Ti and Pt foils, Ti wires, Ti meshes and graphite substrates. Deposition yield can be controlled by the variation of the deposition time. Coatings of various thickness in the range up to 50 μm were obtained. The method enables the formation of dense, adherent and uniform deposits on substrates of complex shape. Obtained coatings provide corrosion protection of stainless steel and Ti and can be utilized for the fabrication of advanced biomedical implants.

2:20 PM
Diffusion of Aqueous Solutions into Hydrophobic Nanoporous Thin-Films: Implications for Fracture: Eric Guyer[1]; *Reinhold Dauskardt*[1]; [1]Stanford University

Nanoporous organosilicate thin-films are being developed for use in a range of novel technologies ranging from biological to microelectronic devices. However, they are mechanically fragile and are prone to environmentally assisted cracking when exposed to aqueous environments. In this presentation, we demonstrate using novel diffusion studies that aqueous solutions can diffuse into highly hydrophobic nanoporous films containing interconnected porosity. The solution chemistry and presence of organic buffering agents significantly affects the ability of the solution to permeate into the nanoporous film. We further show that the diffusion of aqueous solutions into films can alter the films' stress state by changes in the surface stress of the internal pore surfaces. The resulting change in stress state has a profound effect on crack growth rate and has important consequences for reliability. Implications for the integration of nanoporous thin-films into advanced devices are considered.

2:40 PM Invited
Improvement of the Surface Bioactivity of Titanium Implants: *Mei Wei*[1]; Haibo Qu[1]; [1]University of Connecticut

Titanium has been widely used in orthopedic applications owing to its superior mechanical properties and higher corrosion resistance. However, as a bioinert material, titanium implants cannot bond directly to living bone as bioactive ceramics can, such as hydroxyapatite and Bioglass. As a result, there has been increasing interest in the recent years in the formation of a bioactive surface layer directly on titanium substrates, which will induce apatite formation in the living environment or simulated body fluid. In the present study, nano-hydroxyapatite was formed on the surface-modified titanium surface. The effects of both surface modification and simulated body fluid immersion conditions on the hydroxyapatite formation rate and the bonding strength between the hydroxyapatite and titanium substrate were investigated. A dense, strong hydroxyapatite layer was formed on the surface-modified titanium substrate within a short period.

3:20 PM
Ion Adsorption Behavior on SiO2 and Al2O3 Nanoparticles in Aqueous Electrolytes: *Gabriele Vidrich*[1]; Oliver Moll[1]; Hans Ferkel[2]; [1]Technical University Clausthal; [2]Volkswagen AG

The ion adsorption behavior on nanoparticles in aqueous electrolytes for deposition of dispersion-strengthened nickel was investigated. Measurements of the hydrogen formation during plating from pure nickel sulfamate electrolyte, electrolyte containing either 1.5 wt.-% SiO_2 or Al_2O_3 nanoparticles with particle diameter around 30 nm were carried out. These results were correlated with earlier investigations on the Zeta potential as well as on the dispersion behavior of the nanoparticles in diluted electrolytes. It was found that in case of pure electrolyte and dispersions containing SiO_2 nanoparticles the amount of H_2 collected at the cathode is nearly the same whereas in case of electrolyte containing Al_2O_3 about 3 times less H_2 evolves during plating. In conjunction with overpotential measurements, these results indicate that hydrogen is probably bonded on the co-deposited Al_2O_3. In case of SiO_2 the adsorption site on the particle surface are preferentially occupied by Ni^{2+} ion as discussed in earlier publications.

3:40 PM
Photo-Synthesis of Nano Gold Particles for Drug Delivery: *Yung-Chin Yang*[1]; Jun-Ying Lin[1]; Chang-Hai Wang[2]; Yeukuang Hwu[2]; [1]National Taipei University of Technology; [2]Institute of Physics, Academia Sinica

We reported a simple approach to generate gold colloidal from $HAuCl_4$ containing aqueous solution by synchrotron x-ray irradiation at room temperature, without a reducing agent. Gold colloidal were readily formed by the exposure of $HAuCl_4$ aqueous solution to synchrotron x-ray. It was also observed that the addition of $NaHCO_3$ tremendously modified the size and size distribution of gold nanoparticles. Under optimal conditions, the precipitated nanoparticles were well dispersed and with uniform size, ˜10 nm. The Au nanoparticle solution prepared with this simple, clean and very fast process without toxic reluctant is biocompatible – and could be used for surface modification and drug attachment procedures in the same bath. Our preliminary drug carrying experiment showed that thus produced gold nanopartricles could successfully carry anti-tumor drug and possess potential for drug delivery applications. Further studies on accessing the capacity of drug loading, delivery and release characteristics are currently underway.

4:00 PM
Energy Exchange in Nanopores: *Yu Qiao*[1]; Falgun B. Surani[1]; Xinguo Kong[1]; [1]University of Akron

According to a recent experimental study, with appropriate porous structure and surface properties, a nanoporous material can be infiltrated by a nonwetting liquid when the pressure is sufficiently high. As the pressure is reduced, for reasons that are still under investigation, the confined liquid would remain in the nominally energetically unfavorable nanopores, and thus the excess solid-liquid interface energy can be considered as being "absorbed", leading to a significant hysteresis of absorption isotherm. Due to the ultrahigh specific surface area of the nanoporous material that usually ranges from 100-1000 m^2/g, the energy absorption effectiveness of such a system can be higher than that of conventional energy absorption materials by orders of magnitude. The infiltration pressure can be adjusted by using chemical admixtures in the range of 0-20 MPa. The system recovery ratio can be as high as 75%.

4:20 PM
Investigation of Nanostructured Cadmium Sulfide in the Presence of Copper and Silver Salts: *Christopher Ryan Lubeck*[1]; T. Yong-Jin Han[2]; Alexander E. Gash[2]; Fiona M. Doyle[1]; [1]University of California, Berkeley; [2]Lawrence Livermore National Laboratory

Polymeric and oligomeric molecules have been used to template inorganics. In particular, nanostructured cadmium sulfide has been synthesized via a direct templating method in which self-assembled amphiphiles create structure within precipitated inorganic particles. Cadmium sulfide forms in the hydrophilic portion of the water/amphiphile liquid crystal and is excluded from the hydrophobic portion as particles grow. We investigate the transformation of this cadmium sulfide structure

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in the presence of silver and copper salts. Due to the high interfacial area of the nanostructured material, the rate of transformation is significantly faster than for bulk material. This method has allowed for the exchange of cadmium ions with copper and silver ions yielding silver sulfide and copper sulfide of different structures.

The Brandon Symposium: Advanced Materials and Characterization: Interfaces

Sponsored by: The Minerals, Metals and Materials Society, Indian Institute of Metals, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Srinivasa Ranganathan, Indian Institute of Science; Wayne D. Kaplan, Technion; Manfred R. Ruhle, Max-Planck Institute; David N. Seidman, Northwestern University; D. Shechtman, Technion; Tadao Watanabe, Tohoku University; Rachman Chaim, Technion

Tuesday PM — Room: 206B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Tadao Watanabe, Tohoku University; Yuichi Ikuhara, University of Tokyo

2:00 PM Invited
Thin Films in Ceramic Grain Boundaries: *C. Barry Carter*[1]; Jessica L. Riesterer[1]; Arzu Altay[1]; Jeffrey K. Farrer[2]; N. Ravishankar[3]; [1]University of Minnesota; [2]Brigham Young University; [3]Indian Institute of Science

Thin amorphous films are so often present along interfaces in polycrystalline ceramics that it is frequently assumed that such films are always present, similar in thickness in all ceramics, and in an equilibrium form. These assumptions are not generally valid and may be misleading. Classic liquid-phase sintering relies on the wetting of ceramic powder compacts by glass-forming additives with lower melting points. How the secondary phase wets the interface, how glass permeates these interfaces, and how this influences interface migration during processing is unclear. This talk will present results from two systems using a combination of microscopy techniques. Thin films of the second phase (anorthite or silica) are deposited onto sapphire or rutile substrates and then heated to 1400°C–1650°C. The second phase may dewet or continue to wet the surface. Particular attention will be paid to considering the importance of kinetics, and how they combine to control the wetting process.

2:25 PM Invited
Role of Interface on Structure and Properties of Multifunctional Epitaxial Oxides: *Xiaoqing Pan*[1]; Darrell Schlom[2]; Chang-Beom Eom[3]; [1]University of Michigan; [2]Pennsylvania State University; [3]University of Wisconsin

In this talk the effect of lattice mismatch (epitaxial strain), crystal defects, and chemistry at the film/substrate interfaces on the microstructure and properties of different systems will be addressed. The interfacial atomic structure and defects of the films were investigated by high-resolution transmission electron microscopy (HRTEM). The structure and formation mechanisms of misfit dislocations and their significance for the relaxation of epitaxial strain were systematically investigated. Quantitative HRTEM analysis of strained BaTiO3 predicts the enhancement of spontaneous polarization by 200%, which is consistent with the direct measurements of strained BaTiO3 thin films using epitaxial SrRuO3 electrodes. Furthermore, the effect of atomic steps on the substrate surface due to vicinal cutting on the epitaxial growth, microstructure and properties of perovskite oxide films will be described. It will be demonstrated that the existence of the atomic step flow on miscut substrate eliminates the formation of pyrochlore phases in epitaxial PMN-PT films.

2:50 PM Invited
Contact Deformation and Failure in Single Layer and Multi-Layer Columnar Hard Coatings: *Vikram Jayaram*[1]; S. K. Biswas[1]; [1]Indian Institute of Science

Hard, columnar films of TiN are used extensively in applications ranging from cutting tools to decorative coatings. When subject to contact loading, the films display one of two extreme responses: thin coatings on hard substrates display a mode of inter-columnar sliding in which the two phases deform compatibly, while thick coatings on hard substrates display extensive cracking, characterisitic of a large strain mis-match between film and substrate. This presentation will address the rationale behind this behaviour, the influence of multi-layering at the nanometric level and the implications for the design of hard coatings, through qualitative arguments as well as an analytical treatment of the problem of Hertzian contact deformation of a bi-material system using integral transforms.

3:15 PM Invited
Equilibrium Amorphous Films at Metal-Ceramic Interfaces: *Wayne David Kaplan*[1]; Amir Avishai[1]; [1]Technion-Israel Institute of Technology

Intergranular amorphous films (IGF) with a thickness of ~1nm exist in many ceramic systems. In this work the nature of IGF's at metal-ceramic interfaces was investigated via detailed microstructural characterization of model metal-alumina nanocomposites, and wetting/dewetting experiments. Cu-alumina and Ni-alumina composites, with and without glass-forming additives, were produced and characterized. The composites doped with glass-forming additives contained amorphous pockets at the triple junctions, as well as IGF's at all of the metal-alumina interfaces. No amorphous phase was observed in the undoped composites. In the doped composites the metal particles were found at triple junctions, grain boundaries, and as occluded particles within the alumina grains. Calculation of Hamaker constants for the metal-ceramic interfaces resulted in a stronger attractive force for IGF's at metal-alumina interfaces, compared to alumina grain boundaries, correlating to the film thickness measured in this work.

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4:00 PM
The Role of Partial Grain Boundary Dislocations in Grain Boundary Sliding: *Diana Farkas*[1]; Brian Hyde[2]; [1]Virginia Tech; [2]Northwestern University

Atomistic computer simulations were performed to investigate the mechanisms of grain boundary sliding in bcc Fe using molecular statics and dynamics with embedded atom method interatomic potentials. For this study we have chosen the Σ=5,(310)[001] symmetrical tilt boundary with tilt angle θ = 36.9°. Sliding was determined to be governed by grain boundary dislocation activity with Burgers vectors belonging to the DSC lattice. The sliding process was found to occur through the nucleation and glide of partial grain boundary dislocations, with a secondary grain boundary structure playing an important role in the sliding process. Interstitial impurities and vacancies were introduced in the grain boundary to study their role as nucleation sites for the partial grain boundary dislocations. While vacancies and H interstitials act as preferred nucleation sites, C interstitials do not. The role of pre-existing grain boundary dislocations in the sliding behavior will also be discussed.

4:25 PM Invited
Grain Boundary Excess Free Volume – Direct Thermodynamic Measurement: *Lazar Simhovich Shvindlerman*[1]; Günter Gottstein[1]; [1]RWTH Aachen

Grain boundary excess free (BFV) volume along with surface tension belongs to the major thermodynamic properties of grain boundaries. Unfortunately, our knowledge about grain boundary excess free volume is completely restricted by data of computer simulation, which, in its turn, is strictly limited by grain boundaries in the vicinity of special misorientation. However, for the grain boundaries due to the availability of the additional degree of freedom there is a way for correct thermodynamic measurement of this physical property. The special technique developed makes it possible to measure the BFV for practically any grain boundary and provides a way of estimating grain boundary excess free volume for grain boundaries of different classes with rather high accuracy. The knowledge of BFV is especially important for fine grained and nanocrystalline systems where it opens up new possibilities to control and design the physical properties and microstructure of such polycrystals.

4:50 PM Invited
Grain Boundary Populations as a Function of Disorientation and Boundary Normal: *Anthony D. Rollett*[1]; Jason Gruber[1]; Chang-Soo Kim[1];

Tricia Bennett[1]; Gregory Rhorer[1]; Nathalie Bozzolo[2]; [1]Carnegie Mellon University; [2]University of Metz

Studies of grain boundary populations that take account of the full five degrees of freedom, that is to say misorientation and boundary normal, reveal many interesting trends. Although the coincident site lattice (CSL) theory that is linked to Prof. Brandon's name has been a very useful tool for understanding interfaces, the grain boundary population data point to a much stronger connection to the properties of the two surfaces comprising the boundary. This connection is reinforced by computer simulation of grain growth that demonstrates the existence of a steady-state anisotropy in the population directly linked to the anisotropy of the grain boundary energy. This view is illustrated by reference to recent results for a variety of materials, including zirconium and iron.

The James Morris Honorary Symposium on Aluminum Wrought Products for Automotive, Packaging, and Other Applications: Automotive Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Recycling Committee
Program Organizers: Subodh K. Das, Secat Inc; Gyan Jha, ARCO Aluminum Inc; Zhong Li, Aleris International Inc; Tongguang Zhai, University of Kentucky; Jiantao Liu, Alcoa Technical Center

Tuesday PM — Room: 207A
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Subodh K. Das, Secat Inc; Jiantao Liu, Alcoa Technical Center

2:00 PM Invited
Implementation of Continuous Cast (CC) AA5754 Aluminum Alloy in Automotive Stampings and Hydroforms: *Sooho Kim*[1]; Anil K. Sachdev[1]; Alan A. Luo[1]; Raja K. Mishra[1]; [1]General Motors R&D Center

This study demonstrates that low-cost continuous cast (CC) aluminum alloys have slightly lower press formability than current production direct chill (DC) cast aluminum sheet, but the formability is sufficient for many automotive stampings like hood reinforcements, heat shields, skid plates etc. Tubes made of CC 5754 sheet showed similar forming strains in bending and hydroforming compared to conventional DC 5754 seam-welded and 6063 extruded tubes. The talk will describe the microstructures, post-forming properties of the hydroformed tubes and EBSD measurements of the texture distribution/evolution during deformation.

2:25 PM Invited
The Role of Microstructure on the Formability on Automotive Aluminum Alloy Sheet: *David S. Wilkinson*[1]; Xinjian Duan[1]; Jidong Kang[1]; Mukesh Jain[1]; Raj Mishra[2]; Anil Sachdev[2]; Sooho Kim[2]; [1]McMaster University; [2]General Motors R&D Center

We have studied the effect of microstructure on formability of aluminum alloys sheet (primarily AA5755, DC and CC variants). Fe-based intermetallic particles, found as isolated particles and in stringers, lower the ductility and formability of these alloys. They may also contribute to the lower ductility of CC alloys. These materials are very resistant to damage until just before fracture, so that this is not a particle-induced damage effect. We have studied the role of particles in the development of shear localization. I will outline a set of experiments and models which demonstrate the link between shear instability and fracture and the role played by hard particles. I will show how local strain mapping using digital image correlation helps to understand PLC band development and shear localization, while FEM calculations demonstrate the effect of particle density and morphology on shear localization. The aim is a new model that couples micro/macro effects.

2:50 PM
Study of Microstructure and Aging Behavior of Continuous Cast 6111 Aluminum Alloy: *Zhong Li*[1]; Steve Kirkland[1]; Shixi Ding[1]; Dave Thompson[1]; Paul Platek[1]; [1]Aleris International, Inc

Aleris International, Inc. has been developing continuous cast (CC) aluminum alloy sheets for different automotive applications over the last several years. Aleris has cast 5754, 5182, 5083 and 6111 using twin-belt casting technology for the research and development work towards automotive applications. In this paper, microstructures and aging behavior of continuous cast 6111 aluminum alloy after different routes of processing were studied and discussed.

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The Bauschinger Effect in Aluminum Alloy AA6111: *Henry Proudhon*[1]; Warren Poole[1]; David Lloyd[2]; [1]UBC; [2]Novelis Inc.

There is considerable current interest in developing mechanical property models which describe the yield stress and work hardening of 6000 series alloys which are used in automotive applications. This study examines the contribution of long range internal elastic stresses on the macroscopic hardening response. Internal stresses have been evaluated by a series of Bauschinger tests on alloys with different states of precipitation. It has been observed that there are significant differences between underaged and peak aged material compared to highly overaged material. This is attributed to the transition in the nature of the dislocation-precipitate interaction. For underaged and peak aged materials, precipitate shearing dominates and there are relatively small internal stresses. On the other hand, for the case of overaged samples with non-shearable precipitates, significant internal stresses are observed due to the storage of dislocations around the precipitates.

3:40 PM
AA6082 Feedstock Production for Thixoforging: *Yücel Birol*[1]; Ugur Bozkurt[2]; Mehmet Onsel[2]; [1]Marmara Research Center; [2]Bosphorus University

Thixoforming offers the possibility of forming complex aluminum parts with an exceptional quality and a reduction of processing steps. The production of a fine, equi-axed, globular microstructure is a must for the success of the thixoforming process. Strain induced melt activation (SIMA) process produces such a microstructure through recrystallization of heavily deformed billets and a subsequent heat treatment in the mushy zone. In the cooling slope (CS) route, on the other hand, molten metal with a suitable superheat is cast over a water-cooled, inclined surface into a permanent mould to produce the thixotropic billet. Both SIMA process and the CS casting route were employed to produce AA6082 thixotropic feedstock in the present work. The effect of cold work and heat treatment conditions and the effect of cooling length, casting temperature and reheating conditions on the final microstructures were investigated for the SIMA process and CS casting route, respectively.

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A Microstructural Examination of Aluminum Alloys Subjected to Incremental Forming: *Albert Krause*[1]; William T. Donlon[1]; Alan Gillard[1]; Sergey Golovashchenko[1]; [1]Ford Motor Company

Improvements in formability of certain aluminum alloys is required for utilization by the automotive industry for complex shaped body panels to reduce vehicle weight. One possible approach to increase formability is through incremental forming; a process of deforming the aluminum sheet in separate steps, with the sheet being subjected to a quick recovery heat treatment between each forming step, in order to partially undo the effects of work hardening. This process can significantly increase the total amount of deformation before fracture. The purpose of this work was to determine if the structural mechanism responsible for the partial recovery of both aging and non-aging aluminum alloys could be determined. The microstructure was examined through both optical and transmission electron microscopy for alloys subjected to a variety of temperatures and deformations.

4:40 PM
Characterization of AA 5754-O Sheet Metal Deformed under In-Plane Stretching: *Stephen W. Banovic*[1]; Mark A. Iadicola[1]; Tim Foecke[1]; [1]National Institute of Standards and Technology

Material behavior data for use in constitutive equations is often lacking to properly model material flow during stamping operations and accurately predict the material's response (limiting strains; surface roughness;

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friction; springback). In an effort to better understand the processing-structure-properties relationship of aluminum sheet, AA 5754-O is deformed under in-plane stretching, with measurement of true stress-true strain curves using a unique in situ X-ray diffraction system. Subsequent characterization is conducted using light optical and scanning electron microscopy. Diffraction techniques are used to measure the change in crystallographic texture. The results of this work are discussed with respect to implementation in existing and developing constitutive laws.

5:05 PM
Improving the Characterization of the Surface Morphology Generated in Deformed Aluminum Sheet: *Mark R. Stoudt*[1]; Joseph B. Hubbard[1]; [1]National Institute of Standards and Technology

Numeric predictions of mechanical behavior during forming are central to the automotive design process. Discrepancies between the predicted roughness and what is measured on real surfaces signify that improved predictive accuracy requires a better understanding of the fundamental relationships between deformation and the morphology of the free surface. Inaccurate predictions of surface character also imply limitations in the conventional surface roughness characterization methods. Most surface roughness assessments utilize tacit assumptions about the distributions of surface features within the roughness data. The validity of these assessments is further degraded by compressing complex surface information into singular quantities that are too coarse with respect to the length-scales of the relevant features. A different approach, based on ensemble averaging and rigorous statistical protocols, is being developed to improve the fidelity between roughness measurements and the original surface. Results from efficacy determinations performed on strained 6022 aluminum surfaces are presented and discussed.

5:25 PM
Overview of 5000-Series Aluminum Materials for Hot Forming in the Automotive Industry: *Eric M. Taleff*[1]; [1]University of Texas

Hot gas-pressure forming of 5000-series aluminum materials has recently been implemented for the mass production of automotive components by two major automobile producers. Product shapes used in these forming operations include both sheet, for body closure panels, and tubing, for subframe components. This presentation provides an overview of the important aspects of microstructure and mechanical behavior in 5000-series aluminum materials for these hot-forming operations. The elevated-temperature deformation mechanisms of grain-boundary sliding and solute-drag creep enable the large tensile ductilities necessary for hot-forming operations. Failure under forming conditions can be by either cavitation or flow localization. Cavitation is strongly influenced by the volume and size distribution of intermetallic particles. Aspects of microstructure which influence deformation and failure behaviors are discussed.

The Rohatgi Honorary Symposium on Solidification Processing of Metal Matrix Composites: Properties of MMCs

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Solidification Committee
Program Organizers: Nikhil Gupta, Polytechnic University; Warren H. Hunt, Aluminum Consultants Group Inc

Tuesday PM — Room: 207B
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Darrell R. Herling, Pacific Northwest National Laboratory; Somuri V. Prasad, Sandia National Laboratories

2:00 PM Invited
Friction and Wear Mechanisms in Aluminum Alloy Metal-Matrix Composites: *Somuri V. Prasad*[1]; Rajiv Asthana[2]; [1]Sandia National Laboratories; [2]University of Wisconsin-Stout

The major drawback of aluminum alloys in tribological (friction and wear) applications is their poor resistance to seizure and galling. The pioneering work of Rohatgi in the late 1960's has enabled us to synthesize a variety of cast aluminum alloy metal-matrix composites (MMCs) dispersed with solid lubricants, or ceramics (particulates, whiskers, or short fibers), or a combination of these to achieve the desired balances in physical, mechanical and tribological properties required for a tribocomponent. In this paper, we shall critically examine the mechanisms of lubrication in self-lubricating MMCs (e.g., Al alloy-graphite) and material removal mechanisms in abrasion-resistant MMCs (e.g., Al alloy-ceramic composites). Current and emerging applications of Al MMCs in aerospace and automotive industries will be discussed. *Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

2:25 PM Invited
Short-Fiber Cast Aluminum MMCs: Properties and Value: *Gerald A. Gegel*[1]; David Weiss[2]; [1]Materials and Process Consultancy; [2]Eck Industries, Inc.

Metal matrix composite alloys combine the attributes of metals and ceramic reinforcements to provide materials engineered with low density and high specific mechanical properties. Most components do not require the high performance capability of aluminum MMCs throughout their entirety. Selective reinforcement, the reinforcement of only the high stress regions of a component, will reduce manufacturing costs. Pressure infiltration casting was used to produce test specimens from three short-fiber ceramic reinforcements. The measured mechanical properties of these materials and comparative cost analysis were used to accomplish a value analysis for each ceramic reinforcement. These property and value data may be used to facilitate component design and manufacture of selectively reinforced aluminum MMC components.

2:50 PM
Effects of Particle Size and Volume Fraction on Wear Behavior of A356 Aluminum Alloy/SiC Particles Composites: Martin Duarte[1]; *Jose Miguel Molina*[1]; Javier Narciso[1]; Enrique Louis[1]; [1]Alicante University

The wear resistance of A356/SiC composites with different particle sizes and volume fractions has been evaluated in the as-cast condition. The composites were fabricated trough direct mixing of the ceramic particle and the liquid metal. Wear experiments were carried out in a ball–on–disk machine. In an attempt to determine the wear mechanisms, the morphology of the wear track was examined by scanning electron microscopy. As found in previous studies, the wear performance increases with the particle size and volume fraction. The tribological properties of these materials are analyzed and compared against theoretical wear models. The power spectrum of the friction signal is analyzed by means of Fast Fourier Transform Algorithms. It turns out that the power spectrum shows a 1/f behavior. The fractal dimension of the signal is determined by means of a modified Counting Box Method. The results are compared with those obtained for steel and matrix alloy.

3:15 PM
Damping Characteristics of Al-Li-SiCp Composites: *Mirle Krishnegowda Surappa*[1]; Ranjit Bauri[1]; [1]Indian Institute of Science

Damping characteristics of 8090 Al alloy and its composites reinforced with 8, 12, and 18 vol pct SiC particles were investigated using a dynamic mechanical analyzer (DMA). Tests were done at different frequencies over a temperature range of 27°C to 300°C. Composites show higher damping capacity than the unreinforced alloy. Damping capacity is found to increase with decreasing frequency. An equation relating damping capacity with frequency has been proposed. The damping data are analyzed in the light of matrix microstructure and different operative mechanism.

3:40 PM
The Effect of Si and Mg Addition on Microstructure and Mechanical Properties of Al-60% SiC Composites Produced by Pressure Infiltration Technique: *Huseyin Cimenoglu*[1]; Ercan Candan[2]; Hayrettin Ahlatci[2]; [1]Istanbul Technical University; [2]Zonguldak Karaelmas University

In this study, effect of Si and Mg addition on the microstructure and mechanical properties of the Al-60 vol.% SiCp composites, produced by pressure infiltration, technique were investigated. Si and Mg content of Al matrix varied 0-8 wt.% while SiC particles had 23µm mean diameter. Mechanical properties of the composites were characterized by uniaxial compression, three point bending and impact tests. Si and Al3C4 intermetallics were present in the Al-Si matrix composites and whereas Mg2Si was also present in Al-Mg alloy matrix composites in addition to Si and Al3C4 intermetallics. Mechanical test revealed that maximum bending and compression strengths were achieved in Al-1.0 Si alloy/SiC composite. Impact resistance of Al-Si/SiC composites decreased with increasing Si content. As compared to Al-Si alloy matrix composites, Mg addition to Al matrix increased both bending and compression strengths whereas toughness was reduced notably. Fractured surfaces were examined by SEM after the mechanical tests.

4:05 PM Break

4:20 PM
Use of Dynamic Low Load Micro Hardness Indentation Technique in Studying Serrated Flow in Al-SiSp Composites: *Mirle Krishnegowda Surappa*[1]; Ranjit Bauri[1]; [1]Indian Institute of Science

Occurrence of plastic instabilities during depth sensing indentation in 8090 Al alloy and 8090 Al–SiCp composites containing 8 and 15 vol.% of SiC particles have been investigated by dynamic ultra low load micro hardness (DUH) technique. The unreinforced alloy shows instabilities (serrations) in the load–displacement curve in the as-extruded (AE) and solution-treated conditions. However, the magnitude of serrations becomes negligible after ageing. Al–Li–8% SiC composite shows signs of serrations in the solution-treated condition. On the other hand, Al–Li–15% SiC composite shows almost no or negligible serrated flow in all the conditions (as-extruded, solution-treated, and aged).

4:45 PM Invited
Laser Deposition of Metal Matrix Composites: *John E. Smugeresky*[1]; Baolong Zheng[2]; David Gill[1]; Yizhang Zhou[2]; Enrique J. Lavernia[2]; [1]Sandia National Laboratories; [2]University of California

Laser deposition via the Laser Engineered Net Shaping (LENS®) process is being used to explore the potential for in-situ synthesis of metal matrix composites. LENS® is a layer additive metal shaping process, that employs solidification as a mechanism for synthesizing materials with variations in compositions coupled with process parameter (e.g., substrate temperature, laser power, substrate traverse speed) modifications as a means to control microstructures and properties. With cooling rates in the 103 K per second, the LENS process enables the achievement of microstructure refinement and metastable phases. In this paper, we evaluate the effect of increasing additions of fine dispersoids into a metal matrix over a range of compositions and processing conditions on the microstructure and properties of the matrix metal. Characterization of the resultant microstructure includes metallographic analysis, composition profiles, and hardness using optical, SEM, EDS, XRD, and micro-hardness techniques. Mechanical properties of selected compositions will also be presented.

5:10 PM
Wear and Friction Behavior of Near Eutectic Al-Si +ZrO2 or WC Particle Composites: *Atef Awad Doud*[1]; Pradeep Rohatgi[2]; [1]CMRDI; [2]University of Wisconsin-Milwaukee

Dry sliding wear and friction behavior of cast near eutectic Al-Si alloy and composites containing 5 vol% WC or ZrO2 particles were studied by means of a three pins-on-disk (steel) apparatus. The results showed that the composites exhibited a superior wear resistance in comparison to the unreinforced alloy at load range of 18-180 N. The coefficients of friction for all the tested materials decreased with increasing applied load from 18 N to 90 N. However, at load levels of 90-180 N, the coefficients of friction increase as the load increases. SEM investigations revealed that in the load range of 18-90 N, the worn surfaces of the composites reinforced either by WC or ZrO2 were covered by iron oxides, which provided in situ lubrication. At load above 90 N, the WC or ZrO2 particles fractured and lose their ability to support the load. Therefore, the wear rates of the composites significantly increased.

5:35 PM Invited
Wear of Lead Free Cast Copper Alloy-Graphite Particle Composite: *Deo Nath*[1]; N. Prasad[1]; Pradeep Rohatgi[2]; [1]Banaras Hindu University; [2]University of Wisconsin - Milwaukee

Copper alloy-graphite particle composite (60Cu - 37.5Zn, 1.75 Graphite, 0.75Ti) was developed as a substitute for leaded copper alloy used in plumbing and bearings and wear tested under cast and extruded conditions. The wear volume and rate were determined as a function of sliding distance and lead. Optical and Scanning Electron Microscopy was used to study the wear debris and worn surfaces respectively. The wear volume and rate of the composite increased with increasing lead and sliding distance and was comparable to those of leaded copper alloy (60Cu - 37.5Zn, 1.75Pb, 0.75Sn) at higher loads. The size of wear debris decreased with increasing load. SEM studies indicated adhesive wear to operate.

Titanium Alloys for High Temperature Applications - A Symposium Dedicated to the Memory of Dr. Martin Blackburn: Microstructure and Properties of High Temperature Titanium Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Michael W. Peretti, Lyondell Chemical Company; Daniel Eylon, University of Dayton; Ulrike Habel, Munich; Guido C. Keijzers, Del West USA; Michael R. Winstone, DSTL

Tuesday PM Room: 201
March 14, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Ulrike Habel, Munich; Michael H. Loretto, University of Birmingham

2:00 PM Invited
Microstructure and Properties of High-Temperaturen Titanium Alloys after Rapid Heat Treatment: *Orest Ivasishin*[1]; Vadym Bondarchuk[1]; [1]Institute for Metal Physics, NAS of Ukraine

Enhancement of high-temperature strength and creep resistance of titanium alloys is an important issue that would allow to extend the field of their application. Mechanical properties of titanium alloys are to a great extent determined by a microstructure. The present paper addresses Rapid Heat Treatment (RHT) as an effective method to improve microstructure of titanium alloys and thus their high temperature performance. The RHT comprises fast (tens to hundreds degrees per second) heating into the single-phase beta field and results in a formation of a specific fine-grained beta-transformed microstructure unachievable with conventional heat treatments. This microstructure possesses a high creep resistance inherent to the conventional beta-transformed condition, but is free from drawbacks of the latter such as low ductility or fatigue resistance. The advantages of RHT over conventional treatments are discussed from the standpoint of microstructure/properties relationship and high temperature deformation mechanisms.

2:30 PM
Creep Behavior of TIMETAL®1100 at Elevated Temperatures and Its Automotive Applications: *Yoji Kosaka*[1]; Stephen P. Fox[1]; [1]Timet

TIMETAL®1100 (Ti-6Al-2.7Sn-4Zr-0.5Mo-0.45Si) was developed to maximize creep resistance with adequate strength and fatigue performance in aircraft jet engine applications. The combination of weight saving and the excellent high temperature properties of this alloy has opened an opportunity for the application of this alloy as exhaust valves of automotive and motorcycle engines. The service temperature for the alloy in this application appears to be much higher than the 1100°F (600°C) for which the alloy was originally designed. The present paper will introduce the application of TIMETAL®1100 for automotive engine valves and discuss the creep behavior up to 760°C or 1400°F.

3:00 PM
Creep and Recovery at Lower Temperatures in Titanium-Aluminum Alloys: *M. Brandes*[1]; Michael J. Mills[1]; [1]Ohio State University

Although titanium alloys are widely known to undergo a variety of creep and recovery processes at temperatures above 0.4 T_m, these types of phenomena occurring at lower temperatures are not well documented. Remarkably, it has been found that these materials, when deformed at lower temperatures, can exhibit recovery processes at temperatures as low as room temperature. This work addresses observations of the recovery of strain hardening in creep deformed Ti-6 wt% Al and Ti-6242 with the variation of several factors including plastic strain level, time spent in the unloaded state, exposure temperature, loading direction, and microstructure. It is proposed that the formation of intense b = <11–20> dislocation pile-ups at grain and/or phase boundaries, which stem from the planarity in these alloys, provide the driving force for a thermally activated, dislocation level recovery mechanism.

3:30 PM
Roller-Burnishing Effects on HCF Performance of Notched Ti-6242 at Ambient and Elevated Temperatures: *Tomasz Ludian*[1]; Wagner Lothar[1]; [1]Clausthal University of Technology

In Ti-6Al-2Sn-4Zr-2Mo-0.1Si, fully lamellar and duplex microstructures were processed through thermal and thermo-mechanical treatments, respectively. Resulting microstructures and textures were evaluated. Mechanical testing was performed at 20°C as well as 500°C. Fatigue tests were done in axial loading (R=-1) at roughly 100 Hz. Unnotched (kt = 1.0) and circumferentially slightly notched (kt = 1.5) as well as heavily notched (kt = 3.0) specimens were tested. In ball-burnishing, a hydraulically driven device utilizing various ball sizes and rolling forces was used. After burnishing, the changes in surface and near-surface properties such as surface topography, surface layer micro-hardness and residual stress-depth profiles were determined. For comparison, electrolytically polished conditions were prepared to serve as reference. The fatigue results are interpreted in terms of cyclic and thermal stability of the ball-burnishing-induced near-surface high dislocation densities and residual compressive stresses and their effects in turn on fatigue crack nucleation and micro-crack propagation.

4:00 PM Break

4:30 PM
Comparing Residual Stresses in Linear Friction Welded α-β and Near-α Ti Alloys: *Philipp G. Frankel*[1]; Mallikarjun Karadge[1]; Michael Preuss[1]; Greg Johnson[1]; Philip J. Withers[1]; Axel Steuwer[2]; Simon Bray[3]; [1]University of Manchester; [2]ESRF - ILL; [3]Rolls Royce Plc.

Residual stresses in linear friction welded (LFW) Ti-6Al-4V and Ti-6242 have been determined using high energy synchrotron X-ray diffraction. The chemical induced shift is accounted for by using the biaxial $\sin^2\Psi$-technique with a collimated laboratory X-ray source on thin slices cut from the centre region of a LFW sample of each material. Use of the Contour Method and hardness profiling also provide valuable information, as texture from the LFW process limits the effectiveness of diffraction based techniques directly at the weld line. Results show stresses near the weld line for the as-welded samples, with higher peak stresses in the Ti-6242 sample. After post weld heat treatment (PWHT) effective for stress relief in Ti-6Al-4V, stresses remain in the Ti-6242. Increasing the temperature by 100°C significantly lowers these stresses. This demonstrates that alloys designed for creep resistance at higher temperature, produce higher residual stresses during friction welding, requiring modified PWHT for effective stress relief.

5:00 PM
Heat-Resistant Titanium with Intermetallic Frame-Dispersion Strengthening: *Yaroslav Yurievych Kompan*[1]; Alexander Tarasovych Nazarchuk[1]; Igor Viktorovych Protokovilov[1]; [1]Paton Electric Welding Institute

The present paper is devoted to the magnetohydrodynamic and technological peculiarities of refining and homogenization of a cast structure of solid-solution multi-component heat-resistant titanium alloys with intermetallics. Round and rectangular ingots of these alloys were produced in magnetically-controlled electroslag melting (MEM). The key in the creation of the magnetically-controlled technology of melting of heat-resistant alloys of the new class is the different nature of hydrodynamic control of metal melting and ingot crystallization. The control is realized by external magnetic fields of different directivity, discrete cyclic action of melting electric current, and also by an integrated action of external fields and discrete electric currents. Alloy with a uniform frame and dispersion strengthening was melted on the solid-solution base of alloy VT22 with additions of tin intermetallics. Heat-resistance of this alloy is 330-400 MPa in heating up to 750-800°C.

5:30 PM
Visualization of Three-Dimensional Microstructures Reconstructed from Serial Sections in Ti-6Al-4V-TiB: *Scott I. Lieberman*[1]; Arun M. Gokhale[1]; Sesh Tamirisa[2]; [1]Georgia Institute of Technology; [2]Ohio University

Discontinuously reinforced titanium matrix composites (DRTi) and modified titanium alloys containing in-situ formed titanium boride whiskers (TiB_w) enhance the mechanical properties relative to unreinforced Ti-6Al-4V at room and elevated temperatures. In the development of these materials, characterization and visualization of the three-dimensional (3D) microstructure is of significant theoretical and practical interest. The properties and performance of the resultant material depend on the attributes of the 3D microstructural geometry. A recently developed montage-based serial sectioning technique has been utilized to visualize and recreate large volumes of 3D microstructure on a millimeter length scale at sub-micron resolution. This technique is useful for detecting and characterizing both short-range and long-range spatial patterns in non-uniform microstructures, and is of relevance for the increasingly diverse potential applications of Ti-6Al-4V-TiB materials.

Ultrafine Grained Materials - Fourth International Symposium: Microstructures and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
Program Organizers: Yuntian T. Zhu, Los Alamos National Laboratory; Terence G. Langdon, University of Southern California; Zenji Horita, Kyushu University; Michael Zehetbauer, University of Vienna; S. L. Semiatin, Air Force Research Laboratory; Terry C. Lowe, Los Alamos National Laboratory

Tuesday PM — Room: 217D
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Terry R. McNelley, U.S. Naval Postgraduate School; Hael Mughrabi, Universitaet Erlangen-Nuernberg; David Morris, CENIM; David J. Alexander, Los Alamos National Laboratory

2:00 PM Invited
Microstructural Mechanisms Governing the Fatigue Performance of Ultrafine-Grained Materials: *Hael Mughrabi*[1]; Heinz Werner Hoeppel[1]; Martin Kautz[1]; [1]University Erlangen-Nuernberg

The fatigue behaviours of different ultrafine-grained (UFG) metals and alloys (pure copper, commercial purity aluminium, alpha brass, an age-hardenable aluminium alloy) that were produced by the equal channel angular pressing technique, will be reviewed. Aside from details characteristic of the material considered, one important common finding is that the fatigue life of UFG materials, compared to materials of conventional grain (CG) size, is enhanced in the high-cycle fatigue (HCF) regime but lowered in the low-cycle fatigue (LCF) range. This behaviour is easily understood in a total strain fatigue life diagram. The LCF performance of UFG materials can be improved by a suitable annealing treatment (enhancing the ductility, retaining sufficient strength). However, the LCF performance generally remains below that of the CG materials. The dominant microstructural features of fatigue damage in different UFG materials will be reviewed and discussed with respect to fatigue life.

2:20 PM Invited
Microstructure and Texture Evolution in Commercial Purity Aluminum during High-Pressure Torsion Studied by OIM: *Alexander P. Zhilyaev*[1]; Keiichiro Oishi[1]; Terry R. McNelley[1]; [1]Naval Postgraduate School

An investigation was conducted to evaluate the microstructural evolution occurring in disks of commercial purity aluminum processed by high-pressure torsion (HPT) under constrained conditions. Microhardness measurements were taken to assess the variation in hardness across the diameters of disks subjected to different imposed strains and the microstructures were observed at the edges and in the centers of the disks using transmission electron microscopy. The results show the microhardness is lower and there is less grain refinement in the central regions of the disks in the initial stages of torsional straining but the microstructures become reasonably homogeneous across the disks at high-imposed strains. Thorough OIM study has been performed to reveal a relationship between microstructure and texture forming during high-pressure straining. Comparison with the results of OIM study of ECAP aluminum has been discussed.

2:40 PM Invited

Density of Stacking Faults and Twin Boundaries in Ultrafine-Grained Materials Determined by X-Ray Line Profile Analysis: *Tamás Ungár*[1]; Levente Balogh[1]; Gábor Ribárik[1]; Yuntian T. Zhu[2]; Zenji Horita[3]; Terence G. Langdon[4]; [1]Eötvös University; [2]Los Alamos National Laboratory; [3]Kyushu University; [4]University of Southern California

It has been reported that planar faults can play an important role in the defect structure in ultrafine-grained materials. The planar fault character (i.e. intrinsic- or extrinsic stacking faults or twin boundaries) can be distinguished by X-ray line profile analysis. Planar faults are crystal defects of "size" character in the sense that the line broadening caused by them is globally diffraction order independent. Line broadening and line shift caused by planar faults is highly anisotropic as a function of the hkl indices. This makes it possible to distinguish the line broadening components corresponding to planar faults, dislocations and subgrain-size. The numerical procedures developed so far for the determination of dislocation structures and subgrain-size are extended for determining planar faults. The defect structure of high pressure torsion specimens is presented with special emphasis on the planar fault structure.

3:00 PM

Effect of Grain Size Refinement and Particle Redistribution on the Mechanical Properties of an Al-7%Si Alloy Processed by Severe Plastic Deformation at Different Temperatures: *Maria Antonia Muñoz-Morris*[1]; Ivan Gutierrez[1]; David Gareth Morris[1]; [1]CENIM-CSIC

The microstructural evolution of an Al-7%Si alloy has been analysed after severe plastic deformation processes carried out both by equal channel angular pressing (ECAP) and forging at 20ºC and 200ºC. The as-cast microstructure is coarse and has a cellular distribution of Si particles which are broken and redistributed by the severe deformation processes at the same time as the reduction in grain size occurs. The two processes produce, however, different effects with respect to the particle redistribution and morfology of grains and particles which determine the final grain size. The latter reaches values ranging between 150-250 nm in the materials processed at room temperature but are slightly larger at a processing temperature of 200ºC. The increase in strength produced by the microstructural refinement has been analysed considering the different parameters leading to hardening in terms of their effectiveness as obstacles for dislocation motion.

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Low-Temperature Consolidation of Ultrafine Grained Al 6061-T6 Produced by Machining: Balkrishna Rao[1]; Richard McMillen[1]; W. Dale Compton[1]; Srinivasan Chandrasekar[1]; *Kevin P. Trumble*[1]; [1]Purdue University

Plane-strain machining has been used to produce Al6061-T6 particulate having grain sizes less than 100 nm and ~1.5 times higher hardness than bulk 6061-T6. Retaining the ultrafine structure through consolidation to bulk forms requires low-temperature densification/bonding routes. The presentation will focus on two main routes under investigation: room-temperature powder extrusion and polymer bonding. Extrusion of 1:1 blends of Al6061-T6 machining chip particulate and pure Al at room temperature and extrusion ratios up to 42 yielded densities greater than 97%, with intimate bonding as revealed by metallography. Preliminary tensile testing showed little or no plastic elongation, but tensile strengths of 150 to 200 MPa. Several routes for densifying and bonding the chip particles with various epoxy resins will also be presented. Metal fractions greater than 90 vol % have been achieved with no loss of hardness during the epoxy cure. Preliminary modeling on the hardness of these composites.

3:30 PM

Medium Carbon Steel Processed by Warm Equal Channel Angular Pressing: *Jozef Zrnik*[1]; Jaroslav Drnek[1]; Ondrej Stejskal[2]; Sergey Dobatkin[3]; Zbysek Novy[1]; [1]COMTES FHT; [2]West Bohemian University; [3]MISIS Moscow

In present study, the evolution ultrafine ferrite-pearlite microstructure during thermomechanical(TM)processing and subsequent warm severe deformation ECAP of medium carbon steel AISI 1045 was investigated. In preliminary step of straining very fine microstructure with high degree of strengthening has been achieved by means of a multistep open die forging. Fine dynamically recrystallized structure of ferrite and pearlite mixture with grain size of~ 2 μm resulted from hot press forging. The further grain refinement was obtained during severe warm deformation of preliminary forged specimens using ECAP following route Bc. The ECAP was performed with channel of 90 deg at temperature of 400°C and in two cycles (N=2).After the second ECAP pass the substructure was locally depended and presence polygonized substructure and submicrocrystalline structure was observed in strongly extended ferrite grains, and/or areas with desintegrated pearlite. The final mechanical properties of TM treated and ECAP processed specimens were designed.

3:45 PM

Scale-Up Experiments Demonstrate Benefits of ECAE Processing: *Karl T. Hartwig*[1]; Shabib J Kadri[1]; Robert E. Barber[1]; [1]Texas A&M University

The ability of ECAE to induce equivalent plastic strain irrespective of work-piece size is demonstrated. Square cross-section bars of large-grained as-cast CDA 101 Cu having columnar grains of 1-5 mm diameter and 10-20 mm length, and billet widths of 19 mm, 25 mm and 50 mm were extruded at room temperature through routes A, B, C and E for up to eight passes in sliding-wall 90° tooling. Characterization of as-worked and recrystallized microstructures via hardness and microscopy show independence from billet size. Fully worked material exhibits elongated grain fragments with widths of 100-150 nm and lengths of 200-300 nm. The fully-worked/recrystallized material has an average grain size of 4-5 microns. It is concluded that a larger billet cross-section gives greater load efficiency during ECAE processing, and that extrusion routes which incorporate a 90° rotation between passes lead to an increase in load as a result of texture affects.

4:00 PM Break

4:10 PM Invited

The Influence of Coarse Second-Phase Particles and Fine Precipitates on Microstructure Refinement, Structural Stability on Annealing, and Mechanical Properties of a Severely Deformed Al-Mg-Si Alloy: Maria A. Muñoz-Morris[1]; Ivan Gutierrez-Urrutia[1]; *David G. Morris*[1]; [1]CENIM-CSIC

An Al-Mg-Si alloy in various solutionised and aged states was severely plastically deformed. Materials were subsequently annealed and mechanical behaviour examined. Microstructural refinement and strengthening occur faster in the presence of particles. Coarse particles remain unaffected by severe deformation, and help stabilise the deformation microstructure, while fine precipitates dissolve. Microstructural refinement and strengthening depends more on solute content than on particle distributions. Solute precipitates on annealing, irrespective of the initial material state, but these particles play only a small role in stabilising deformation substructure. After significant particle and grain coarsening occurred, discontinuous grain coarsening may occur. Material strength is examined in terms of the contributions of loosely-arranged dislocations, many grain boundaries, and dispersed particles. Dislocation strengthening is significant in as-deformed and lightly annealed materials, with grain boundary strengthening providing the major contribution thereafter. Particle strengthening is not generally as important here as the other two strengthening contributions.

4:30 PM Invited

Formation Mechanism of Dimpled Fracture Surfaces of Nanocrystalline Materials: Zhiwei Shan[1]; J. A. Knapp[2]; D. M. Follstaedt[2]; Jorg Michael Wiezorek[1]; Eric A. Stach[3]; *Scott X. Mao*[1]; [1]University of Pittsburgh; [2]Sandia National Laboratories; [3]Purdue University

Dimple features much larger in size than the average grain size are often observed at the fracture surface of three dimensional nanocrystalline materials, but their formation mechanism is currently unclear. This study demonstrates that the in situ tensile straining transmission electron microscopy (TEM) technique can be used to reveal the underlying physical mechanism for the formation of the dimple features. At the onset of deformation of nanocrystalline Ni, we found that grain agglomerates with sizes much larger than the average grain size formed very frequently and rapidly in many locations, apparently independently of one another. HRTEM observations suggested that the grains in those grain agglomerates are most probably separated by low angle grain boundaries. Furthermore, both inter- and intra- grain agglomerate fractures were observed. Sandia is a multiprogram laboratory operated by Sandia Corporation, for the United States DOE's National Nuclear Security Administration under contract DE-AC04-94AL85.

4:50 PM Invited

Parameters Influencing the Limits of Microstructural Fragmentation during Severe Plastic Deformation: *Andreas Vorhauer*[1]; Reinhard Pippan[1]; [1]Austrian Academy of Sciences

Severe Plastic Deformation (SPD) is an effective method to produce ultra fine grained materials in bulk dimensions. In certain cases, many authors reported about the possibility to obtain submicrometer or even nanometer scaled microstructures in initially recrystallized, coarse grained base materials. This phenomenon is now well known since about more than ten to fifteen years. However, despite the continuously growing number of publications aimed to this topic and the increasing number of institutes, which are engaged in this field of materials science, the parameters, which are influencing the limits of microstructural fragmentation during severe plastic deformation at large strains, are still not fully understood. The aim of this paper is to gain a better understanding of such parameters, which are may be influencing the microstructural fragmentation and its saturation at large strains. Therefore, SPD by means of a high quality High Pressure Torsion tool was applied at different temperatures (between 77K and 723K), strain rates (between 2.5 x 10^-3 and 6.5x10^-2 s-1), hydrostatic pressures (between 0.85 and 5.4 GPa) on a wide range of different materials (pure metals: copper, iron, titanium, aluminium, molybdenum, nickel; technical relevant high alloyed steels: austenitic and ferritic steel). Measurements of flow curves in-situ during SPD have shown that a saturation of flow stress without any further work hardening is obtained at large strains, when SPD is performed at low homologous temperatures. At higher processing temperatures, the flow curves show a distinct maximum followed by a drop when strain exceeds the peak strain. These results, together with microstructural investigations in the scanning electron microscope (capturing of back scattered electron micrographs and orientation maps) at peak strain (maximum stress or plateau stress) have clearly shown, that the properties obtained by SPD are significantly dependant on parameters such as the homologous temperature and strain rate of materials processing and the purity of the material itself, whereby the applied hydrostatic pressure affects the results only for a small extent.

5:10 PM Invited

Preparing Multi-Component Hard Materials through Severe Plastic Deformation: *Jacob Chih-Ching Huang*[1]; C. J. Lee[1]; C. H. Chuang[1]; [1]National Sun Yat Sen University

It is intended to prepare much harder materials of multi-elements or multi-components through friction stir processing (FSP) or accumulative roll bonding (ARB). The first trial is to add 1-10% nano ceramic particles into the soft AZ61 Mg alloy via four FSP passes. The Hv hardness of the resulting nano-composites reaches over 110. The second trial is to fabricate multi-element intermetallic alloys of Mg50-80Al10-25Zn10-40 by three FSP passes. The Hv hardness of the resulting intermetallic alloys varies from 140 to 350. The third trial is to prepare the nanocrystalline and amorphous Zr based alloys through room temperature ARB from various elemental foils. After 40-100 ARB cycles, the elemental foils are refined into nanocrystalline phases and eventually transformed into amorphous alloys. The Hv hardness varies from 200 to over 500. The characterization of microstructures and mechanical properties of the resulting hard materials will be presented.

5:30 PM

Production of Ultrafine-Grained Copper by Cryogenic Torsion: *David J. Alexander*[1]; [1]Los Alamos National Laboratory

High-purity copper was strained at cryogenic temperatures by performing torsion of samples immersed in liquid nitrogen. The samples had a gage length of 51 mm and a diameter of 12.7 mm. Up to 16 rotations were applied before failure. Several combinations of forward and reversed torsion with a cumulative strain equivalent to 16 complete forward rotations were examined, including no reversals, a single reversal, and 8 reversals. Thin slices were taken from the deformed gage section, and heat-treated for various combinations of time and temperature. The resultant microstructures were examined by optical and scanning microscopy, and orientation imaging microscopy was used to determine the texture. The effect of strain, the number of strain reversals, and heat treatment conditions on the texture and grain size distribution will be discussed.

5:45 PM

Microstructural Development of "Recycled-Like" Alloys during ECAP – Particle Break-Up, Phase Transformations and Mechanical Properties: *Przemyslaw Szczygiel*[1]; Hans J. Roven[1]; [1]Norwegian University of Science and Technology

As a part of a comprehensive research program on recycling of aluminium, the present work deals with microstructural development of two generic alloys with recycled-like chemistries during severe plastic deformation (ECAP). Influence of deformation routes A and B were investigated, i.e. the microstructure evolution under different shearing patterns. Particle behaviour under complex stress conditions in the die corner was investigated and both qualitative and quantitative descriptions of the particle break-up processes are presented. Size distributions of different phases are given based on 2D image analysis and 3D measurements of particles extracted from the matrix using the "buthanol method". Some indications of possible pressure induced phase transformations are discussed. The influence of particles on deformation mechanisms, grain size distribution and evolution of grain boundaries was characterized applying HR-SEM and TEM. Post ECAP mechanical properties are examined and correlated with observed microstructure characteristics.

Ultrafine Grained Materials - Fourth International Symposium: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Shaping and Forming Committee
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Tuesday 5:15-7:00 PM   Room: 217D
March 14, 2006   Location: Henry B. Gonzalez Convention Ctr.

Influence of Equal Channel Angular Extrusion Processing Routes on Consolidation of Intermetallics: Anumalasetty Venkata Nagasekhar[1]; *Yip Tick-Hon*[1]; [1]Nanyang Technological University

Equal channel angular extrusion is the most promising severe plastic deformation technique for the fabrication of bulk ultrafine grain materials, and for consolidation of ultrafine powders. Thus, it has potential to enhance both physical and mechanical properties of the processed materials. In the current study an attempt was made to consolidate the recently invented intermetallic superconductor, magnesium boride (MgB2). By using commercial MgB2 powder as core and low carbon steel tube as the sheath, ECAE consolidation was carried at room temperature up to four passes via different processing routes: Route A, Route BC, Route BA, and Route C. Influence of processing route on consolidation was characterized in terms of density, and micro-hardness measurements. Consolidation force requirements for different passes were also calculated. Higher densification was achieved via Route A, and lower densification was achieved via Route BC. The result shows that ECAE offers a new possibility for the consolidation of intermetallic superconductors.

Deformation Resistance of Ultrafine-Grained Copper at Elevated Temperature: Yujiao Li[1]; Rajeev Kapoor[1]; *Jingtao Wang*[1]; Wolfgang Blum[1]; [1]University of Erlangen

The deformation resistance (flow stress, creep rate) of ultrafine-grained (UFG) Cu produced by equal channel angular pressing on route B_C at room temperature is investigated at elevated temperature (100 to 200 C). Compared to coarse-grained (CG) Cu the UFG Cu is stronger at small strains, but softer at large strains, due to its large content of high-angle grain boundaries. The virtual absence of small-angle grain boundaries in UFG Cu is apparent from TEM observation as well as from the absence of the pronounced transients upon change of deformation conditions. There is a distinct softening of UFG Cu with strain during deformation at elevated temperature beyond the maximum deformation resistance. The microstructural reasons for this softening and the resistance of UFG Cu against creep and stress relaxation are addressed.

Deformation Resolution of Aluminidies of Transition Metals by Severe Plastic Deformation: *Irina Grigorjevna Brodova*[1]; Irina Gennadjevna Shirinkina[1]; Irina Petrovna Lennikova[1]; Valery Aleksandrovich Shabashov[1]; [1]Institute of Metal Phisics

A special interest for studies has a deformation resolution of stable and metastable aluminidies of twice phase Al alloys with transition metals (Zr, Fe, Cr) by severe plastic deformation. It was determined a kinetic of deformation dissolution of the Al7Cr and Al13Fe4 stable aluminidies and the Al3Zr and Al6Fe metastable intermetallic-compound crystals. It was established that metastable crystals of Al3Zr fully dissolved in Al matrix and super saturated Al-base solid solution with ultra microcrystalline structure was formed in the Al-Zr alloy.Another aluminidies destroyed and only partly dissolved in Al matrix. After HPT these sizes decreased up to 10 – 20 nm and they regular distributed in ultra microcrystalline Al matrix. The hardness of these materials very high – 2000 –3000 GPa. According the Mössbauer spectroscopy data, HPT results to changes in the phase composition of structure constituents in the Al-Fe alloys.

Effect of Severe Plastic Deformation on Internal Friction in Fe3Al, AZ31 and Titanium Alloys: *Igor Stanislavovich Golovin*[1]; [1]Technical University

In the past, several attempts were undertaken to study anelastic properties of metals whose ultra fine grain structure was produced by severe plastic deformation (SPD). With rare exceptions, in most of them grain boundary effects were considered at elevated temperatures. Here we present mechanical spectroscopy data on low-temperature dislocation- and point-defect-related anelasticity of three materials: Fe-26Al and Ti deformed by high pressure torsion and Mg alloy AZ31 deformed by equal channel angular pressing. Several internal friction peaks with the activation energy in the range of 0.35-0.55 eV were found to be introduced or significantly enhanced by SPD. At least some of these peaks can be classified as Hasiguti peaks. Since the internal friction peaks observed are caused by different crystal lattice imperfections, which have different thermal stability, the mechanical spectroscopy provides a useful tool for studying early annealing stages of SPD processed alloys.

Effect of Very High Straining on the Stress-Strain Behavior of a Zinc-Aluminum Alloy: *Praveen Kumar*[1]; Cheng Xu[1]; Terence G. Langdon[1]; [1]University of Southern California

Specimens of the Zn–22% Al eutectoid alloy were processed by equal channel angular pressing (ECAP) through various numbers of passes up to a maximum of 24 passes at 473 K. Tensile specimens were cut from the unpressed and as-pressed billets and they were tested at strain rates of 1.0 $\times 10^{-2}$ and 1.0 $\times 10^{-3}$ s^{-1} at room temperature. The tensile tests show that higher elongations are recorded in the specimens processed by ECAP. However, samples processed through a very large number of passes show neither more strain hardening nor higher fracture strain than the samples processed through a small number of passes. This result is attributed to the relatively low melting temperature of the alloy since room temperature corresponds to a homologous temperature of about 0.4.

Effects of the Number of Equal-Channel Angular Pressing Passes on the Strain Rate Sensitivity of Ultrafine Copper: *Alexander Korshunov*[1]; Lev Polyakov[1]; Irina Vedernikova[1]; Tamara Kravchenko[1]; Irina Korotchenkova[1]; Andrey Smolyakov[1]; Vyacheslav Soloviev[1]; [1]RFNC-VNIIEF

Annealed tough-pitch copper was processed by eight passes of equal-channel angular pressing (ECAP) using two routes, BC and C. Pressed samples had a square section with a side length of 8 mm. Tensile tests were performed at static loading with strain rate variation by three orders. The material was examined in the as-received condition and after 1, 4 and 8 passes. All standard mechanical properties (conventional yield strength, tensile strength, elongation and contraction) were determined, and conditional and true deformation curves were plotted. Strength properties (conventional yield strength and tensile strength) in all cases grow as the strain rate increases, and plastic properties (elongation and contraction) remain practically the same, except for contraction after 8 ECAP passes, which grows as the strain rate increases.The strain rate sensitivity coefficient was found to grow with the number of ECAP passes.

Experimental Study and Computer Modeling of SPD Shape Forming Process of UFG CP Ti: *Vladimir Latysh*[1]; Gyorgy Krallics[2]; Irina Semenova[3]; A. Fodor[2]; Igor V. Aleksandrov[3]; [1]INTC ; [2]Budapest University of Technology and Economics; [3]Ufa State Aviation Technical University

Numerous experimental studies proved the efficiency of severe plastic deformation (SPD) realized by equal channel angular pressing (ECAP) used for producing bulk ultrafine-grained (UFG) CP Ti billets. However, the successful implementation of the method into the commercial production implies the satisfaction of various requirements to the technological processes. In particular, the most important parameters to be achieved are the decrease in the number of ECAP passes, fabrication of billets with the increased relation between the length and the size of the cross section, enhancement of structure and properties' homogeneity over the whole volume of processed billets. This report presents the results of experimental studies and computer modeling, aimed at the optimization of SPD regimes realized by the combination of ECAP and further broaching. The described approach enabled to find the optimal regimes and produce bulk billets with homogeneous UFG structure and extraordinary properties.

Fatigue Properties of Forged AA6061 after Severe Plastic Deformation: *Balakrishna Cherukuri*[1]; Prabir Kanti Chaudhury[2]; Rob Mayer[3]; Raghavan Srinivasan[1]; [1]Wright State University; [2]Orbital Sciences Corporation; [3]Queen City Forging Company

Severe plastic deformation (SPD) has emerged as a promising technique for creating ultra fine grained metals and alloys, with grain sizes of a micrometer or less. Mechanical properties of forged AA 6061 after severe plastic deformation was investigated. AA 6061 samples in solutionized (W) condition and in annealed (O) condition are severely deformed by Equal Channel Angle Pressing to produce ultrafine grain material of the order of 0.5 micron grain size. The SPD processed material was successfully forged at 650F and 700F as compared to the conventional forged material at 850F. Tensile, hardness and fatigue properties of the forged SPD material is compared against the conventional material. The results show marked difference between the starting material and the severely deformed ultrafine grain material. The results of this investigation will be presented in the light of enhanced properties and processing cost savings for industrial application of SPD material.

High Energy Milling as a Route for Obtaining Ultrafine Grained Duplex Stainless Steel: *Osvaldo Mitsuyuki Cintho*[1]; Cleverson Moinhos[1]; Evaldo Toniolo Kubaski[1]; José Deodoro Trani Capocchi[1]; Eduardo Franco Monlevade[1]; [1]State University of Ponta Grossa

In the present study, Iron, Chromium and Nickel powders were mixtured (Fe-19.5%Cr-5.0%Ni) and submitted to high energy milling in an Attritor type mill, with a ball-to-powder ratio was 50:1. Millings were conducted for 5, 9 and 15 hours under Argon atmosphere. After milling, even for long times, particles have a plate-like morphology. For longer milling times, microscopic platelike particles group into equiaxial agglomerates. The milled samples were then uniaxially pressed into pastilles form and heat treated for 1 hour at 900, 1050 and 1200°C. A complete dissolution reaction of Chromium and Nickel was only observed in the 15 hour milling samples. As a result, an ultra-fine grained (ca. 1 mm) duplex stainless steel sample was obtained in the samples treated at 1200°C. For shorter milling times, Chromium dissolution was shown to be substantially delayed when compared to Nickel dissolution in Iron.

Inhomogeneous and Anisotropic Deformation Behavior and Strain Hardening of Ultrafine-Grained Aluminum by ECAE: *Stijn Poortmans*[1]; Fouad El Houdaigui[2]; Anne-Marie Habraken[2]; Bert Verlinden[1]; [1]Katholieke Universiteit Leuven; [2]University of Liège

Hot-rolled AA1050 commercial pure aluminum was deformed by ECAE at room temperature following route BC for 8 passes. Mechanical testing at room temperature on both hot-rolled aluminum and aluminum after ECAE consisted of uniaxial tension, axisymmetric compression and shear by torsion. The phenomenological Hill's criterion identified from texture data accounts for the observed tension-compression asymmetry due to ECAE and predicts torsion yielding close to measurements. FE simulations of the compression tests are performed with Hill model or Minty micro-macro model and coupled with an isotropic Voce saturation hardening law. These simulations compute the inhomogeneous behavior due to barreling, the observed sample anisotropy and the force-displacement curve. Comparisons of numerical and experiments results provide a first identification of the hardening parameters and the friction coefficient during compression tests. Finally a general hardening model is proposed accounting for the softening during tensile tests and stress saturation in compressive and torsion tests.

Mechanical Alloying and Related Solid-State Flow and Mixing of Single-Crystal Tungsten Ballistic Penetrator and Steel Target Material: *Carlos Pizaña*[1]; Lawrence E. Murr[1]; Aditya Putrevu[1]; Thomas L. Tamoria[2]; H. C. Chen[2]; S. J. Cytron[3]; [1]University of Texas; [2]General Atomics; [3]U.S. Army

In this study optical metallography and SEM analysis (including elemental X-ray mapping) in combination with microhardness maps have elucidated the dynamically recrystallization (DRX)-facilitated flow and interaction of steel targets with penetrating, clad and unclad [001] single-crystal W long rods impacting at initial velocities ranging from 1 to 1.3 km/s. The ultra-fine DRX regime composing adiabatic shear zones in both the penetrator and target allows for mechanical mixing of the W and Fe in complex flow regimes which, in the extreme, melt in localized regions at the projectile/target interface, creating intermetallic phases within the intercalated flow zones. Optical metallographic observations also suggest that the addition of clad material to the projectile could contribute to the penetration deformation and to the projectile/target interaction and therefore the overall performance of the ballistic penetrator. (Supported by the U.S. Army-Picatinny Arsenal, prime Contract No. W15QKN-04-M-0267, project No. 1A4CFJER1ANG).

Mechanical Behavior and Microstructural Evolution in ECAP Copper: *Anuj Mishra*[1]; Bimal Kad[1]; Fabienne Gregori[2]; Robert Asaro[1]; Morgana Martin[3]; Naresh N. Thadhani[3]; Marc A. Meyers[1]; [1]University of California; [2]Laboratoire des Propriétés Mécaniques et Thermodynamiques des Matériaux - CNRS; [3]Georgia Institute of Technology

Microstructural evolution during ECAP has been investigated using TEM and EBSD techniques. Results on quasi-static compression and tensile tests, dynamic compression using Hopkinson bar and Reverse Taylor tests on ECAP samples after different number of passes (initial, 2, 4 and 8 passes) are presented. Saturation in hardness after 10 ECAP passes is shown to exist indicating a lower limit to the grain size that can be achieved using this technique. Deformation mechanisms in ultra-fine grained materials (grain size >100 nm) as produced by ECAP are discussed and compared with the ones that operate in the nanocrystalline regime (grain size < 100 nm).

Effect of Temperature on the Mechanical Properties of Bulk Nanocrystalline Ni Prepared by Electrodeposition: Anna Torrents[1]; *Manish Chauhan*[1]; Farghalli A. Mohamed[1]; [1]University of California

Variation in the hardness as a function of temperature in bulk nanocrystalline Ni having an average initial grain size of 20 nm, prepared by an electrodeposition technique, has been investigated by nano and microindentation. Hardness measurements were conducted on these 20 nm samples which were annealed at different temperatures, ranging from 323–1173 K, for various annealing times. It has been found that the hardness of the material increases with an increment in the annealing temperature and reaches its maximum value at 493 K, and decreases thereafter with further increase in the annealing temperature. This increase in the hardness until 493 K can be attributed to the relaxation of internal stresses around grain boundaries and triple junctions, and the bimodal grain size distribution obtained at this low annealing temperature.

Microstructure and Tensile Properties of Ultrafine Grained Pure-Ti: *Young Gun Ko*[1]; Dong Hyuk Shin[2]; Chong Soo Lee[1]; [1]Pohang University Science and Technology; [2]Hanyang University

A study was made to investigate microstructural evolution and mechanical properties of ultra-fine grained (UFG) pure-Ti produced by equal channel angular (ECA) pressings. The deformed structures were analyzed by finite element method and transmission electron microscopy with the increment of straining. After 4 isothermal ECA pressings, initial coarse grains (30µm) were significantly refined to ~0.3µm with homogeneous distribution of microstructure which was resulted from 180° rotation of the sample between pressings. UFG pure-Ti exhibited the considerable improvement in yield strength while losing strain hardening capacity as compared to coarse grained microstructure at ambient temperature, which was mainly attributed to ultra-fine grain microstructure with non-equilibrium grain boundaries. Such high strength and unusual strain hardening behavior of UFG pure-Ti were discussed in relation with two dislocation models based on dislocation bow-out and dynamic recovery associated with trapped lattice dislocations.

Modeling of Grain Refinement and Texture Evolution during Equal-Channel Angular Pressing by Means of Combined Visco-Plastic Self Consistent/Disclination Model: *Airat A. Nazarov*[1]; Igor V. Aleksandrov[1]; Irene J. Beyerlein[2]; Nariman A. Enikeev[1]; Tatiana S. Orlova[3]; Alexei E. Romanov[3]; [1]Ufa State Aviation Technical University; [2]Los Alamos National Laboratory; [3]Ioffe Physico-Technical Institute, Russian Academy of Sciences

Disclination model of grain subdivision incorporated into the visco-plastic self-consistent (VPSC) modeling of plastic deformation is used to simulate the grain refinement during equal-channel angular pressing (ECAP). Strain incompatibilities between a homogeneous effective medium and a grain calculated by VPSC are assumed to result in an accumulation of disclinations at the grain junctions. The stresses of these disclinations are then relaxed by growth of low-angle dislocation boundaries from the junctions. These boundaries split the grain into smaller, mis-oriented volumes which, with further deformation, can lead to its subdivision into new grains. The applied ECAP deformation histories are given by either a simple shear model, a fan model, or by finite element method calculations. The corresponding evolution of microstructure and texture during different ECAP routes are simulated. The calculated texture and intragrain misorientations evolution are found to be in good agreement with experimental data.

Modelling of Stress-Strain Distribution in ECAE by Analytical-Experimental Method: *Georgy Iosifovich Raab*[1]; Rimma Lapovok[2]; [1]UFA State Aviation Technical University; [2]Monash University

It is believed, that uniform simple shear is realized during ECAE resulting in uniformity of the stress-strain distribution and mechanical properties. However, some experimental facts as well as Finite Element (FE) simulation confirm a more complicated and non-uniform strain mode. Despite increased efficiency of FE packages accounting for temperature, friction condition, tooling configuration and complicated constitutive models, the finite shear strain can not be precisely simulated by plastic flow or incremental models. The simple and precise analytical-experimental method for direct evaluation of strain distribution during shear deformation, namely the combined grid method was adopted in this study. This paper describes the theoretical principles, the grid patterns and the calculation of the strains based on the grid node displacements and calculation of stress resulting on integration of equilibrium equations along flow lines. Results are compared with those of FE simulation.

Nanostructure Formation Induced by Explosively Driven Friction: *Hong Jin Kim*[1]; Andrew Emge[1]; Karthikeyan Subramanian[1]; David Rigney[1]; Peter Keightley[2]; Ron Winter[2]; [1]Ohio State University; [2]Atomic Weapon Establishment

There has been a surge of interest in ultrafine and nanograin microstructures formed by severe plastic deformation (SPD) methods such as Equal Channel Angular Pressing (ECAP), High Pressure Torsion (HPT), Repetitive Corrugation and Straightening (RCS) and even simple sliding. Explosively driven friction testing is a novel technique developed at the Atomic Weapon Establishment to produce large plastic strains in a short time. These tests involve driving a pair of metals obliquely against each other in a confined sleeve at velocities of 50-200 m/s. The metal-pair used here is aluminum/steel. Post-test characterization techniques such as Scanning Electron Microscopy (SEM), Focused Ion Beam (FIB) imaging and

Transmission Electron Microscopy (TEM), conducted at The Ohio State University, revealed SPD occurring on the aluminum side. The bending of fiducial line markers suggest large plastic strains (>14) adjacent to the sliding interface and this is structurally manifested by the formation of nano-scale structure.

Numerical Simulations of the ECAE Process Multi Pass: *Andrey Smolyakov*[1]; Alexander Korshunov[1]; Vyacheslav P. Solovyev[1]; [1]RFNC-VNIIEF

Numerical simulations of ECAE process carried using DRACON code (VNIIEF) based on variation-difference method of solving continuum mechanics equations have shown that satisfactory agreement between experimental and numerical data on deformed billet state can be achieved by using experimental data in the development of physical model. Issued analysis of passes number influence on uniform state in ECAE.

Processing of Long-Length Rods of TiNi Alloys with the UFG Structure and Increased Properties Using Multi-Step SPD Processing: *Dmitry Valerievich Gunderov*[1]; Vladimir Latysh[2]; Vladimir Pushin[3]; Alexander Lukyanov[1]; Irik Kandarov[2]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]INTC "Iskra"; [3]Institute of Physics of Metals

As the investigations have shown, formation of UFG structure by ECAP allows increasing strength and instrumental properties of TiNi alloys with shape memory effect (SME). But ECAP allows to process only cylindrical samples with the diameter 20-40 mm and the length up to 200 mm. However TiNi alloys are most widely used in medicine as pieces of thin rods and wire. Multi-step SPD processing that consists of the combination of ECAP, forging and drawing was applied to Ti49.4Ni50.6 alloy. As a result integral rods with the length up to 800 mm, diameter 6 mm and NC structure were successfully obtained. The given results show that the chosen treatment modes allowed to change the shape of an ECAP-billet and process integral long-length rods of NC TiNi alloy with high level of strength - 1400 MPa, that respectively implies high level of instrumental characteristics of SME.

Production of Ultrafine Grained Ferrite Structure through Multi-Pass Warm Caliber Rolling: *Venkata Suryanarayan Susarla Murty*[1]; Shiro Torizuka[1]; Akio Ohmori[1]; Kotobu Nagai[1]; [1]National Institute for Materials Science

Ultrafine grained steel bars having a cross section of 18mm square were fabricated through a multi-pass warm caliber rolling for a 0.15%C-0.3%Si-1.5%Mn steel. Average ferrite grain sizes of 0.43 μm, 0.70 μm and 1.2 μm were obtained in the isothermal rolling processes at 773K, 823K and 873K respectively. Even though caliber rolling results in inhomogeneous strain distribution, multi-pass caliber rolling to large accumulated strains of 2 or 3 can be uniformly introduced in to bar samples. Strain accumulation due to multi-pass warm deformations was confirmed by comparing microstructural evolution through multi-pass deformation with that of single pass deformation. The hardness and the evolved grains size of the ultrafine grained structures formed through severe warm deformation depend on the Zener-Hollomon parameter. The similarity of the microstructural evolution with single pass deformation reveals that the multi-pass warm deformation is an effective method to obtain ultrafine grained ferrite structure in bulk materials.

Structure of Ti-5Al-5Mo-5V Alloy Deformed by Heat Twist Extrusion: *Tatyana Alexeevna Ryumshyna*[1]; G. K. Volkova[1]; L. V. Loladze[1]; T. E. Konstantinova[1]; V. N. Varyukhin[1]; [1]Donetsk Physics and Technology Institute

X-ray, optic metallography, has investigated features of the structure formed under heat twist extrusion in Ti-5Al-5Mo-5V alloy. The twist extrusion was realized after annealing of samples in one-phase state (100% ß). After a heat annealing at same conditions the a –phase precipitations were obtained in number 49% and one had an elliptical form 5 mcm in size. The morphology of structure had been changed essentially after twist extrusion. The part of the precipitated a – phase was 55%. Two types of precipitations had observed: thin layers of deformation martensite and fine dispersed precipitations up to 300 nm in size. The forming by twist extrusion the structure with ultra fine precipitations of a – phase in ß – grain leads to a reduction of strength and to a rise plasticity.

Microstructural Refinement of Ti Billets by Equal Channel Angular Pressing: *Vladimir Latysh*[1]; Gulnaz Salimgareeva[2]; Irina Semenova[2]; Irek Kandarov[1]; Ruslan Valiev[2]; Yuntian Theodore Zhu[3]; [1]Innovative Scientific Technological Centre "Iskra"; [2]Ufa State Aviation Technical University; [3]Los Alamos National Laboratory

Preliminary deformation is one of the ways to increase the efficiency to refine bulk billets microstructure at equal channel angular pressing (ECAP). In the present paper preliminary deformation by isothermal forging in special heads has been used for CP Ti Grade 2 billets with diameter 40 mm. The investigations of microstructure evolution and mechanical properties of the billets during the forging and further ECAP after 1, 2, 3, and 4 passes have been carried out. It has been shown that decrease in the initial grain size from 50 to 1 μm already after 2 ECAP passes makes it possible to form a uniform equilibrium ultrafine-grained structure with average grain size of a-phase 0.6 μm compared to the grain size after 8 ECAP passes without the preliminary deformation. Moreover, this kind of treatment has allowed increasing Ti strength up to 750 MPa in contrast to 680 MPa after 8 ECAP passes.

Superplastic Behavior of Ultrafine-Grained Ti-6Al-4V ELI Processed by Severe Plastic Deformation: *Irina Semenova*[1]; Liliya Saitova[1]; Georgy Raab[1]; Ruslan Valiev[1]; Terry C. Lowe[2]; [1]Ufa State Aviation Technical University; [2]Los Alamos National Laboratory

The present paper considers the investigation results of the Ti-6Al-4V ELI alloy mechanical behavior in both coarse-grained and ultrafine-grained (UFG) state at elevated temperatures (500-800°C). It has been shown that combined severe plastic deformation induced by equal-channel angular pressing in combination with the further extrusion resulted in a considerable enhancement of Ti-6Al-4V ELI superplastic behavior. The Ti-6Al-4V ELI alloy demonstrates the superplastic characteristics even at 600°C. The following elongation parameters have been processed at relatively low temperatures and high strain rates: 286% at T = 700°C, strain rate = 10-2 s^{-1} and 516% at T = 800°C, strain rate = 10-2 s^{-1}. It has been established that UFG Ti-6Al-4V ELI after superplastic deformation demonstrates the extraordinary strength value of 1500 MPa at room temperature.

Theory of Non-Equilibrium Grain Boundaries and Its Application for Nano- and Microcrystalline Materials: *Vladimir N. Chuvil'deev*[1]; [1]Physical-Technical Research Institute of Nizhny Novgorod State University

Basic principles and methods of non-equilibrium grain boundary theory are considered. Concepts according to which the non-equilibrium grain boundary state depends on the grain boundary free volume changes caused by their interaction with lattice defects, in particular with dislocations, are in the basis of the theory. Non-equilibrium states of the grain boundaries in microcrystalline metals and alloys processed by equal channel angular pressing were analyzed. It was shown that non-equilibrium grain boundary state in microcrystalline materials can be provided by initial non-equilibrium state, as well as by non-equilibrium state caused by embedding dislocations into the boundary during grain boundary migration and/or materials deformation. Influence of the non-equilibrium grain boundary state on recovery, recrystallization, grain boundary sliding, solid solution decomposition and second phase particles precipitation in microcrystalline Al-Mg-Sc, Al-Zn-Mg-Sc, Mg-Al-Zn and Cu-Cr alloys is investigated. This research was supported by International Scientific and Technical Center (Grant No. 2809).

Ultrafine Grained Magnetic Shape Memory Ni2MnGa – Based Alloys: *Vladimir Pushin*[1]; Nikolay Kourov[1]; Alexander Korolev[1]; Ludmila Yurchenko[1]; Dmitry Gunderov[2]; Ruslan Valiev[2]; Yuntian Theodore Zhu[3]; [1]Institute of Metal Physics; [2]Ufa State Aviation Technical University, Institute of Physics of Advanced Materials; [3]Los Alamos National Laboratory

In this work we report the results of investigations of ferromagnetically ordered Heusler Ni2MnGa-based alloys. Our recent studies have shown that superrapid solidification (SRS) from melt and severe plastic torsion deformation (SPD) under high pressure could be effectively used for formation of homogeneous ultrafine grained structures (nano- and submicrocrystalline) and even amorphous state in alloys. For the first time nanostructured alloys were produced using complex combined methods (SRS+SPD+thermotreatment). Structural states and their thermal stabil-

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ity, and also martensitic transformations have been analyzed by X-ray and neutron diffraction and transmission electron microscopy. We identify in submicro-SRS and nano-SPD alloys a premartensitic state, structures of the low-temperature 5M and 7M martensites and their temperature evolution. The physical properties were determined by measuring of electrical resistance, magnetic susceptibility and dilatometry. We also demonstrate in this work that mechanical and shape memory properties can be enchanced essentially by forming ultrafine grained structures in these alloys.

Thermomechanical Conditions of Nanocrystalline Structure Formation and Functional Properties of Severely Deformed Ti-Ni Shape Memory Alloys: Sergey Prokoshkin[1]; Irina Khmelevskaya[1]; Sergey Dobatkin[2]; *Irina Trubitsyna*[1]; Evgeny Tatyanin[2]; Sylvain Turenne[3]; Vladimir Brailovski[4]; Vladimir Stolyarov[5]; Egor Prokofiev[5]; [1]Moscow State Steel and Alloys Institute; [2]Baikov Institute of Metallurgy and Material Science of RAS; [3]Ecole Polytechnique de Montreal; [4]Ecole de Technologie Superieure; [5]Institute of Physics of Advanced Materials

Structure formation under conditions of high pressure torsion in dependence on deformation temperature, pressure and post-deformation annealing in Ti-Ni-based alloys were studied. The tendency to form an amorphous structure under SPD conditions depends on relative positions of the deformation temperature and M_s temperature. The upper limiting deformation temperatures for amorphous and nanocrystalline structures formation were determined for aging and non-aging Ti-Ni alloys. To obtain a nanocrystalline structure under equal-channel angular pressing (ECAP), the ECAP temperature should be below 350°C. As a result of ECAP of Ti-Ni alloys at 350-500°C in 6-8 passes, a submicrocrystalline austenite structure was obtained with the grain size of 0.1-0.2 microns after ECAP at 350°C, 0.2-0.4 microns after ECAP at 450°C and 0.3-0.5 microns after ECAP at 500°C. The highest functional properties were obtained after ECAP at 350°C which exceed the properties provided by traditional thermomechanical treatment.

Combined Reaction Processing of FePd Intermetallics with UFG Microstructure: Anirudha R. Deshpande[1]; *Andreas Kulovits*[1]; Vincent M. Sokalski[1]; Paul Ohodnicki[2]; Jorg Michael Wiezorek[1]; [1]University of Pittsburgh; [2]Carnegie Mellon University

It has been shown in numerous works that ordering heavily deformed Fe-50at%Pd via a combined reaction transformation mode leads to a ultra-fine grained equiaxed structure with enhanced magnetic properties as compared to the polytwinned micro - constituent that forms upon conventional ordering of undeformed γ Fe-50at%Pd. In this study a Fe-50at%Pd alloy was severely plastically deformed in the austenitic γ - FePd state by an equal channel angular pressing or ECAP operation. The structural evolution of the phase - transformation was investigated by means of XRD, SEM and TEM and compared to the combined reaction product of heavily rolled Fe-50at%Pd. The differences in texture evolution during the solid state reactions after different processing routes were measured. The change in magnetic properties during the phase – transformation mode was monitored via VSM. The magnetic domain structure was measured and correlated to the underlying microstructure by means of MFM and AFM.

Effect of Strain Path on Grain Refinement of Aluminum Sheet: *Tetsuo Sakai*[1]; Koki Mori[1]; Hiroshi Utsunomiya[1]; Eimei Nakayama[1]; [1]Osaka University

It is known that recrystallization behaviors of work hardning materials are influenced by not only the amount of strain but also the mode of strain. In the present study, 3 different modes of deformation - only compression, only shear, and the combination of compression and shear - are introduced into commercially pure aluminum sheet, and then we studied about the effects of deformation mode on recrystallization temperature, recrystallization grain size, and texture. In the case of constant annealing temperature , compared with only shear and only compression of single strain mode, material of combination strain mode has smaller recrystallization grain size. According to this result, combination strain sample has lowest annealing temperature in the three samples. These results show that the deformation of different strain mode promotes the refinement of recrystallization grain.

Grain Refinement Limit during ECAP-Deformation: *Vladimir I. Kopylov*[1]; Vladimir N. Chuvil'deev[2]; [1]Physico-Technical Institute, National Academy of Sciences of Belarus; [2]Physical-Technical Research Institute of Nizhny Novgorod State University

The purpose of the present investigation is to determine minimum grain size which can be achieved using intensive plastic deformation. The investigations of the grain refinement limit Dm, which can be attained by ECAP-deformation, were carried out for pure metals, Al-alloys, Fe-Ni alloys and other materials. The experiments have shown the magnitude Dm for metals is varied in the interval 80-200 nm and depends mainly on the ECAP-deformation temperature, strain rate and diffusion parameters of materials. The model of grain refinement limit based on the theory of the nonequilibrium grain boundaries and the classical theory of fragmentation during severe plastic deformations, is developed. According to the model, the process of fragmentation is considered as one of the accommodation processes leading to increasing the power of defects, which are accumulated on the grain boundaries during deformation. The authors thank International Scientific-Technical Center (ISTC grant No. 2809) for support.

Influence of Multi-Step SPD Processing by ECAP, Cold-Rolling and Subsequent Annealing on the Structure of Ti-50.2%Ni Alloy: *Dmitry Valerievich Gunderov*[1]; Vladimir Pushin[2]; Alexander Lukyanov[1]; Egor Prokofiev[1]; Georgy Raab[1]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]Institute of Physics of Metals

The titanium nickelide alloys (TiNi) have high strength, ductility, corrosion resistance and such a property as shape memory effect (SME). This makes them important for application in technology and medicine as long-lasting materials implanted into a body. Equal-channel angular pressing (ECAP) allows to process bulk TiNi samples with UFG structure (grain size ≈ 250nm) and enhanced strength and instrumental properties. The investigations have also shown that rolling after ECAP allows to additionally refine the structure and enhance the properties of TiNi. As a result of rolling mode optimization integral band samples were processed. The alloy structure after rolling with the strain level of 80%-90% may be described as amorphous-nanocrystalline. The following controlled annealing of these states allows forming homogeneous nanocrystalline structures. With the degree of rolling increasing dislocation yield stress and strength increase, reaching 2000 MPa. The processed samples are materials with high reactive stress – up to 1500 MPa.

Advanced ECAP Techniques with Higher Intensity of Strain per One Pressing Cycle: *Georgy Iosifovich Raab*[1]; [1]UFA State Aviation Technical University

Severe plastic deformation (SPD) by equal channel angular pressing (ECAP) makes it possible to considerably enhance mechanical properties of commercial metals and alloys by forming a submicrograined (SMG) structure in billets for 4-12 processing cycles. However this process has a number of disadvantages. Among them are: a low materials utilization rate which is less than 0.5 and high labour intensity caused by high-cycle treatment. In this connection in the paper a number of ECAP methods has been investigated that allows to reach true strain Σ≥ 1,5 for one processing cycle and therefore intensifies the structure-forming process. Peculiarities of both flow and material strain state of the suggested ECAP techniques have been studied by experimental and finite element (FE) simulation. The examples of high-efficiency solutions to form a SMG structure during SPD have been shown. Recommendations for applications of the given investigations are presented.

Development of ECAP-Conform to Produce Ultrafine-Grained Ti: *Georgy Iosifovich Raab*[1]; Fidus Safin[1]; Yuntian Zhu[2]; Terry Lowe[3]; Ruslan Valiev[1]; [1]UFA State Aviation Technical University; [2]Los Alamos National Laboratory; [3]Metallicum

Recently we have shown an ability to process submicrograined aluminium rods with enhanced properties by the new technique that is ECAP-conform. However the production of long-length rods from ultrafine-grained Ti is of great interest, for example for medical applications. In the present work finite element (FE) simulation of a stress-strain state, the influence of tribological regimes, temperatures and some other processing parameters on a material flow and on strain uniformity in the long-length rods have been studied that enables us to design a new die-set for processing Ti by ECAP-conform. The most effective ways for a commercial development of ECAP-conform are discussed as well.

The Influence of ECAP on Microstructure and Mechanical Properties of AM60 - Magnesium Alloy: *Rinat Islamgaliev*[1]; Olga Kulyasova[1]; Bernhard Mingler[2]; Erhard Schafler[2]; Georg Korb[3]; Hans Karnthaler[2]; Michael Zehetbauer[2]; [1]Ufa State Aviation Technical University; [2]University of Vienna; [3]Austrian Research Center Seibersdorf GmbH

The AM60 alloy has been processed by ECAP at different temperatures (150-350°C), and the resulting microstructures have been investigated by means of TEM, DSC and XRD. ECAP reduced the grain size of cast alloy down to 2 mm. The majority of Mg17Al12 precipitates with a size of 0.05–0.2 mm were observed to be situated close to the grain boundaries, whereas precipitates with a size of 10–20 nm were found in the interior of the grains. The ECAP processing at 150°C led to enhancement of strength from 120 MPa to 310 MPa while the original ductility (15%) has been retained. Observations of deformation relief suggest processes of dislocation slip and grain boundary sliding to be responsible for the considerable ductility. Experimental results indicate that in the ECAPed materials the phase transition from solid solution to the two-phase region occurs at lower temperatures than in the undeformed material.

Initial Observations of the Effects of Sample Size on Microstructure and Properties of HPT Processed Titanium: *Rinat Islamgaliev*[1]; Vladimir Latysh[2]; Maxim Murashkin[1]; Askar Kilmametov[1]; Ruslan Valiev[1]; Yuntian Zhu[3]; [1]Ufa State Aviation Technical University; [2]Innovation Scientific Technical Center “ISKRA”; [3]Los Alamos National Laboratory

High pressure torsion (HPT) is a well established severe plastic deformation method to produce nanostructured states in metallic materials. This work presents the new developments of HPT technique to produce larger samples from various metals and alloys under higher imposed pressure. Using this new installation we revealed that SPD leads in pure Ti not only to structure refinement with increase of both the dislocation density and mean-root-squared strains but also to a-w phase transition at room temperature. In the case of Al-based alloys through refining microstructure up to 60-80 nm and precipitation hardening we could reach extraordinary strength and ductility needed for practical application of SPD produced materials.

On the Evolution of Microstructure of Tantalum Induced by Severe Plastic Deformation: *Qiuming Wei*[1]; Kaliat T. Ramesh[2]; K. T. Hartwig[3]; Ruslan Z. Valiev[4]; Laczlo J. Kesckes[5]; [1]University of North Carolina at Charlotte; [2]Johns Hopkins University; [3]Texas A&M University; [4]Ufa State Aviation University; [5]Army Research Laboratory

We have investigated the evolution of microstructure of a body-centered cubic refractory metal, tantalum, induced by equal channel angular press (ECAP). Commercial purity tantalum bars have been subjected to different number of ECAP passes and different type of routes. The grain refinement, grain boundary nature, etc. have been studied as a function of route type and number of passes of ECAP processing. It was found that even after 16 passes of ECAP, the grain refinement was still not saturated. We observed ghost diffraction spots and rings from the severely deformed state the smallest {h,k,l} indecies of which are {0,1/2,1/2}. The interpretation of the appearance of such ghost diffractions calls for further work. It was also found that the ghost diffraction intensity in the selected area electron diffraction increases with number of passes. This ghost diffraction is absent in the undeformed, annealed state.

Anisotropic Mechanical Properties of Ultra-Fine Grained Aluminum Alloy: Gyorgy Krallics[1]; *Arpad Fodor*[1]; Ahmed Ajina Agena[1]; [1]Budapest University of Technology and Economics

Bulk materials, subjected to SPD, are considered to be semi-finished components. They are subjected to additional processing in order to create different kinds of products. The present paper is focused on the workability and anisotropy parameters, required for the subsequent plastic forming processes. Samples for testing were prepared from work pieces subjected to the ECAP process, using various pass schedules. The tensile samples were taken in the longitudinal direction while the samples for upsetting were taken in longitudinal and two perpendicular directions. The mechanical investigations indicated fairly good measurable anisotropic behaviour of the material. Using continuum mechanics, the parameters of the constitutive equation were determined, assuming the material to be rigid-plastic. Complementary investigations, using special specimens to determine the formability limits in different stress states, were also performed. The fracture criteria of isotropic materials were thus modified.

Characterization of Nano and Ultra-Fine Grains Formed during Friction Stir Processing of Ti: *Oleg M. Barabash*[1]; Zhili Feng[1]; Stan A. David[1]; Joe A. Horton[1]; Rozaliya Barabash[1]; [1]Oak Ridge National Laboratory

Microstructural changes of commercially pure Ti due to Friction Stir Processing (FSP) were analyzed by means of optical, electron (TEM), orientation imaging microscopy, mono- and polychromatic X-ray micro diffraction. It was established that after FSP a surface layer with nanocrystalline structure is formed in which the grain size is between 20-60nm. Beneath the nanocrystalline grain layer is a thermomechanically affected zone (TMAZ). The grain size increases sharply (by two orders of magnitude) from nanocrystalline surface region to TMAZ, reaching micron size (5-30 micron) in the TMAZ. Possible causes and mechanisms for the formation of the nanocrystalline and ultra-fine grains will be discussed in light of the microstructure analysis results.

Texture Evolution and Monotonic and Cyclic Response of ECAE Processed Interstitial Free (IF) Steel at Room Temperature: Guney Guven Yapici[1]; *Steven Sutter*[1]; Ibrahim Karaman[1]; Hans Jurgen Maier[2]; [1]Texas A&M University; [2]University of Paderborn

Interstitial Free steel is commonly utilized in automotive industry for producing large body panels and other structural components due to its high formability. Apart from high strength levels, improved fatigue performance is a prerequisite for some applications. Present work focuses on the severe plastic deformation of IF steel using ECAE. IF steel was processed up to 16 passes using various ECAE routes to investigate the processing-microstructure-property relationships. An extensive program was undertaken to characterize microstructure, crystallographic texture and mechanical properties of successfully extruded billets. Eight pass extrusion following route Bc resulted in yield strengths close to 700 MPa exhibiting a ten-fold increase compared to the annealed materials. Low cycle fatigue experiments on the eight pass sample demonstrated a more than two-fold increase in both the cyclic strength and number of cycles to failure. Effect of ECAE texture on the monotonic and cyclic response is discussed together with cyclic microstructural evolution.

Characterization of Severe Plastic Deformation Processed Commercially Pure Al-2024: Andreas Kulovits[1]; Anirudha R. Deshpande[1]; Brian Webler[2]; *Jorg Michael Wiezorek*[1]; [1]University of Pittsburgh; [2]Carnegie Mellon University

Nano-crystalline microstructures with average grain sizes below 100nm have been produced for an Al-Cu-Mg base high strength age hardening aluminum alloy (commercially pure Al-2024) by severe plastic deformation processing. High reductions in thickness were achieved by cold-rolling and subsequent cold-rolling and stacking of the deformed sheets. This variation of the accumulated roll bonding (ARB) method resulted in locally bonded layered compacts. Changes in hardness and microstructure have been carefully monitored by means of micro-hardness measurements, XRD and TEM. The microstructural investigations indicated a truly nanocrystalline microstructure in the locally bonded sections of the layered compact. Microstructural changes associated with the SPD processing of this Al-alloy have been studied with a special focus on the nature of precipitation products and are discussed.

Effect of Severe Plastic Deformation via Equal Channel Angular Extrusion on Shape Memory Response of a Ti-Ni-Pd High Temperature Shape Memory Alloy: *Jae Il Kim*[1]; Ibrahim Karaman[1]; Benat Kockar[1]; Jeff Sharp[2]; [1]Texas A&M University; [2]Marlow Industries

In order to extend the utility of TiNi shape memory alloys for high temperature applications, higher martensitic transformation temperatures (M*) than 100°C, lower temperature hysteresis, and better cyclic reversibility are required. The addition of Pd is effective in increasing the M* and decreasing the temperature hysteresis, however, it also leads to the degradation of thermal cyclic response under stress at elevated temperatures. In this study, the effect of severe plastic deformation (SPD) via equal channel angular extrusion (ECAE) on shape memory properties of a TiNiPd alloy was investigated. Multi-pass extrusions using several different ECAE routes were performed at temperatures as low as 300°C. Shape memory properties were investigated before and after ECAE. Thermal cycling under constant stress levels revealed that cyclic reversibility was significantly improved and critical stress for slip was increased after ECAE. TEM observations revealed that this improvement is caused by ultrafine and nano-scale grains (<300nm).

Design of Microstructure and Texture in NbZr and Ti Using Severe Plastic Deformation for Desired Mechanical Properties in Biomedical Applications: *Guney Guven Yapici*[1]; Ibrahim Karaman[1]; Gagan Singh[1]; Ralph Dieckmann[2]; Hans Jurgen Maier[2]; [1]Texas A&M University; [2]University of Paderborn

NbZr and Ti are widely used in biomedical applications ranging from cardiovascular stents to bone implants due to their high corrosion resistance and excellent biocompatibility. These applications require high strength and elastic modulus combined with enhanced ductility. This talk will summarize findings on the ECAE of NbZr and Ti to achieve target mechanical properties by engineering microstructure and texture. NbZr was processed up to 16 passes at room temperature while Ti was processed up to 12 passes at 300C without any shear localization. Post-processing of extruded billets with conventional forming techniques assisted by low temperature annealing treatments led to the formation of microstructures yielding both high tensile strengths and uniform strains. Series of experiments are conducted with the aim of capturing the yield locus evolution during ECAE. This enables us to demonstrate the effect of ECAE on the yield surface evolution and help developing models to predict the mechanical behavior.

Influence of Ultrafine Grain Size on Yield Strength of Ball-Milled Iron Alloys: *Chol K. Syn*[1]; Donald R. Lesuer[1]; Oleg D. Sherby[2]; [1]Lawrence Livermore National Laboratory; [2]Stanford University

Extended ball millings of iron powders have been shown in recent studies to generate ultrafine grains, as small as 5 nm, and create nano-sized iron oxide particles, leading to extraordinary hardness. Detailed analyses of these studies on ball-milled powders and the consolidated powders show that nano-oxide particles are the major contributors to the strength of ball-milled iron. Ultra-fine grains, although stabilized by the presence of the nano-oxide particles, do not seem to contribute significantly to the strength. This is because grain boundary sliding of the high-angle boundary ultrafine grains prevent dislocation pile-up and the Hall-Petch mechanism becomes less effective at grain sizes below 2 mm. It will be shown that the particles' contribution to the yield strength can be predicted by the surface-to-surface inter-particle spacing. It is proposed that adiabatic shear banding and phase transformation are the basis for creation of the ultrafine oxide particles.

Effects of ECAP Processing on the Recrystallization of FCC Copper and BCC Tantalum: *Joel W. House*[1]; James M. O'Brien[2]; Philip Flater[1]; Robert DeAngelis[3]; William F. Hosford[4]; [1]U.S. Air Force; [2]O'Brien and Assoc; [3]University of Florida; [4]University of Michigan

Deformation by Equal Channel Angular Pressing (ECAP) has been used to develop ultra-fine grain structures in metals. Generally these metallic structures are associated with increases in strength and/or ductility depending on the specific deformation mechanism. Previously we have reported the hardness of ECAP processed OFE Copper and Tantalum (99.95%) after 1, 2, 4, 8 and 16 passes using route BC. No hardness increase was observed after eight passes for either of these two metals. Studies of the effects of extensive plastic strain on metallic structures have correlated an increase frequency of high angle grain boundaries with increasing strain. The present study investigates the effects that the increase frequency of high angle grain boundaries has on the recrystallization of Copper and Tantalum. Detailed comparisons of the recrystallization characteristic of these metals following ECAP processing to eight and 16 passes will be presented.

Microstructural Evolution of Inconel-718 Alloy during Equal Channel Angular Extrusion Processing—Simulation and Experimental Validation: Padma Dhulipala[1]; Vadim Protasov[1]; *Shankar M. Sastry*[1]; [1]Washington University

Inconel-718 alloy was processed by equal channel angular extrusion (ECAE) at 900°C and 925°C for micro structural refinement and mechanical property modifications. Finite element method based DEFORM 3D code was used to simulate the ECAE process and microstructural evolution during ECAE. The simulation predictions of strain distribution in the ECAE processed samples, the extent of metadynamic recrystallization and the recrystallized grain size were in good agreement with the experimental measurements. Fully recrystallized microstructure with an average grain size of 3μm was obtained by ECAE processing the alloy at 925°C at an extrusion rate of 0.4 inch/min. further grain refinements are possible by using multiple pass ECAE processing.

Residual Stresses in Ultrafine Grained Flowformed Ti-6Al-4V: *Ibrahim Ucok*[1]; Gabriel J. Hostetter[1]; Hao Dong[1]; Lawrence S. Kramer[1]; Mehmet N. Gungor[1]; Troy Tack[1]; [1]Concurrent Technologies Corporation

The flowforming (FF) is a novel method for manufacturing near-net-shape tubes. FF is generally performed at room temperature and provides a thickness reduction up to 80%, which yields enhanced tensile properties in the as-flowformed material. However, the large amount of cold work also generates significant residual stresses that may cause problems during final manufacturing processes, such as machining, cutting and welding. In this study, Ti-6Al-4V structural tubes in both the as-flowformed and stress relieved conditions were subjected to residual stress measurement by X-ray diffraction method. The effect of heat treatment on the magnitude of residual stresses, resultant microstructure and mechanical properties will be presented 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 U.S. Navy Manufacturing Technology Program. Approved for public release; distribution is unlimited.

Microstructure and Strength of Ultrafine Grained fcc Metals Produced by Severe Plastic Deformation: *Jeno Gubicza*[1]; Nguyen Quang Chinh[1]; Tamas Ungar[1]; Terence G. Langdon[2]; [1]Eotvos Lorand University; [2]University of Southern California

Nanostructured materials produced by equal channel angular pressing (ECAP) have very high strength owing to their small grain sizes and high dislocation densities. To understand the mechanical behavior of materials produced by ECAP it is necessary to characterize their microstructure. In this work the microstructure of ultrafine-grained face centered cubic (fcc) metals (e.g. Cu, Ni, Al and its alloys) produced by ECAP was studied by X-ray diffraction line profile analysis. High resolution X-ray diffraction experiments were performed using a special double-crystal diffractometer with rotating Cu anode. The crystallite size distribution and some characteristic parameters of the dislocation structure (e.g. density, character and arrangement of dislocations) were obtained from the fitting. The correlation between the parameters of the microstructure and the mechanical behavior is studied and discussed. The effect of alloying and precipitation on the evolution of microstructure during ECAP was also investigated.

Microstructure and Mechanical Properties of an Ultrafine Grained Dual-Phase Steel: *Hongwei Song*[1]; Bi Shi[1]; Junbao Zhang[1]; Xiufang Wang[1]; [1]Baosteel Iron and Steel Company, Ltd.

The tensile ductility of ultrafine grained (UFG) steels grow rapidly worse when the grains is refined down to less than 5 microns. When this happens, the UFG steels appear to lose the capacity for strain hardening or uniform deformation. On the other hand, dual-phase (DP) steels consisting of ductile ferrite matrix and martensite islands show superior mechanical properties, such as continuous yielding, high work hardening rate, and high uniform elongation. It is reasonable to combine UFG with DP microstructure. In the present work, an UFG DP steel was produced by means of severe intercritical-rolling. The results indicate that the grains of ferrite and martensite are equiaxed or polygonal with average grain diameters of 4 and 2 microns, respectively. The steel exhibits both high strength and good ductility, which have been attributed to the grain refining effect and the DP microstructure, respectively.

New Concepts for Severe Plastic Deformation: *David J. Alexander*[1]; [1]Los Alamos National Laboratory

Several concepts for new methods of severe plastic deformation have been devised at Los Alamos National Laboratory. Some of these are simple extensions of conventional methods, such as equal channel angular extrusion (ECAE). This includes ECAE of long billets, or ECAE of flat plates. Some are primarily surface-deformation techniques, while others are bulk deformation processes. The new methods will be described, along with efforts to reduce the methods to practice. Possible benefits and shortcomings of the new concepts will be discussed.

Mechanical Anisotropy due to Microstructural Morphology and Bauschinger Effect in Equal Channel Angular Extrusion (ECAE) Processed Cu: *Mohammed Haouaoui*[1]; Ibrahim Karaman[1]; Hans J. Maier[2]; [1]Texas A&M University; [2]University of Paderborn

The effect of strain path and level on the flow stress anisotropy and Bauschinger effect (BE) in UFG OFHC copper was investigated considering grain morphology, dislocation mean free path and texture evolution. The material was deformed via multipass ECAE using a 90º square die to different strain levels following several routes (A, B, C, C' and E). The room temperature experiments were conducted under tension and compression along three perpendicular directions in each billet. Bauschinger experiments were conducted in both forward tension/reverse compression and forward compression/reverse tension modes. The strong anisotropy in flow stress, softening and BE were correlated to grain morphology and texture. It was found that as opposed to previous observations in which UFG materials have higher strength under compression, the observed stronger tension and tension/compression asymmetry is not only due to the effect of hydrostatic pressure on dislocations and it is also a function of grain morphology.

Deformation Microstructure and Grain Stability in Al-RE Alloys Produced by ECAP: *Michael Ferry*[1]; Nanang Burhan[1]; [1]University of New South Wales

This paper describes the evolution of microstructure in Al-RE alloys generated by equal channel angular pressing (ECAP) and the subsequent microstructural changes associated with elevated temperature annealing. As expected, the alloys develop a submicron grain (SMG) structure during severe plastic deformation with the amount of RE additions strongly influencing the stability of the deformation substructure. It was shown that nanosized RE dispersoids have a strong influence on grain stability with discontinuous grain coarsening suppressed at high annealing temperatures despite the general non-uniformity of the grain boundary character associated with the deformation microstructure. A simple analytical model is proposed that takes into account the effect of both fine particles and orientation gradients on grain coarsening in SMG alloys with the model confirming the observed coarsening behaviour.

Structure and Properties of Ni-20Cr Produced by Severe Plastic Deformation: *Judson S. Marte*[1]; Richard DiDomizio[1]; P. R. Subramanian[1]; Dheepa Srinivasan[1]; Michelle Othon[1]; [1]GE Global Research

Nanostructured and ultrafine grained materials have been receiving attention because of their superior mechanical properties. This paper will examine two severe plastic deformation (SPD) processes for producing nanostructured and ultrafine grained materials: multi-axis forging and equal channel angular extrusion (ECAE). The two processes were applied to a Ni-20 wt.% Cr alloy to determine the extent of grain refinement achievable via SPD. The multi-axis forging process resulted in an equiaxed microstructure with an average grain size of 1 micron. The ECAE process resulted in a heavily dislocated structure with shear bands and sub-grain sizes of approximately 100-300 nm. Heat treatment of the ECAE'd material resulted in dislocation-free structures and substantial grain growth. The evolution of microtexture was examined by electron backscatter diffraction. Low levels of microtexture were observed after heat treatment. Tensile tests were performed on the SPD processed Ni-20Cr. A significant increase in tensile strength was observed after SPD.

Wechsler Symposium on Radiation Effects, Deformation and Phase Transformations in Metals and Ceramics: Irradiation Pressure Vessel

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Korukonda L. Murty, North Carolina State University; Lou K. Mansur, Oak Ridge National Laboratory; Edward P. Simonen, Pacific Northwest National Laboratory; Ram Bajaj, Bettis Atomic Power Laboratory

Tuesday PM — Room: 208
March 14, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Ram Bajaj, Bechtel Bettis Inc; Roger E. Stoller, Oak Ridge National Laboratory

TUESDAY PM

2:00 PM
Local Electrode Atom Probe Characterizations of Neutron Irradiated RPV Steel Welds: *Michael K. Miller*[1]; Kaye Russell[1]; Randy K. Nanstad[1]; Mikhail A. Sokolov[1]; [1]Oak Ridge National Laboratory

An atom probe tomography characterization of the microstructure of two high copper, high nickel, high manganese alloys was performed with particular emphasis on the nature of the copper-enriched precipitates. The materials studied were a 0.20 wt% Cu 1.27% Mn, 1.20% Ni weld from the Palisades reactor that was irradiated to fluences of 1.4 and 3.4 x 10^{23} m^{-2} at 288°C and a 0.56 wt% Cu, 1.36% Mn, 1.66% Ni Rolls Royce weld (WV) that was irradiated to a fluence of 1.6 x 10^{23} m^{-2} at 290°C. Phosphorus and carbon were detected at different segments of the dislocations in the Palisades weld. The extents of the manganese, nickel and silicon distributions in the precipitates were larger than the extent of copper. Nickel and manganese were preferentially located at the precipitate-matrix interface. Silicon was uniform within the precipitate. Phosphorus was located both within the precipitate and at the interface.

2:20 PM
Positron Annihilation Characterization of Nanostructural Features in Reactor Pressure Vessel Steels and Model Alloys: *Brian D. Wirth*[1]; Stephen C. Glade[1]; G. Robert Odette[1]; Michael K. Miller[2]; [1]University of California; [2]Oak Ridge National Laboratory

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 and welds that provide insight into CRPs and MNP behavior, as well as their composition and magnetic properties. The positron results are compared to corresponding small angle neutron scattering, combined electricial resistivity-Seebeck coefficient and atom probe tomography data.

2:40 PM
Irradiation Induced Precipitates in Model Fe-Cu-Mn Alloys: A Unified Cross Comparisons of Multiple Characterization Techniques: Mike Miller[1]; Brian D. Wirth[2]; *G. Robert Odette*[3]; [1]Oak Ridge National Laboratory; [2]University of California, Berkeley; [3]University of California, Santa Barbara

Cu rich precipitates (CRPs) in iron alloys are important in several technological applications and are of fundamental scientific interest. Copper precipitation in both thermally aged and irradiated Fe-0.80Cu and Fe-

0.78Cu-1.05Mn alloys was characterized by several complementary microanalytical techniques, including: atom probe tomography, small angle neutron scattering, combined electrical resistivity-Seebeck coefficient measurements and positron annihilation coincidence Doppler broadening orbital electron momentum spectroscopy. A major objective was to help resolve the controversy about Fe in the CRPs, found or not found, by various methods. Detailed inter-comparisons showed very good overall consistency in the CRP volume fractions, sizes, number densities and even compositions. Unification of the results suggest nearly pure solute CRP cores are surrounded by shells with dilute concentrations of Cu and, especially, Mn. Significant thermal precipitation occurs at 290°C in only 7200h, indicating anomalously fast solute diffusion. Irradiation slightly enhances the solute content of the interface shell, perhaps by cascade mixing.

3:00 PM

A Unified Constitutive Model for Irradiated RPV Steels: Increases in Intrinsic Strain Hardening: *Takuya Yamamoto*[1]; G. Robert Odette[1]; [1]University of California, Santa Barbara

RPV steels irradiated at ˜300°C up to 0.1 dpa harden due to a high density of nanometer-scale Cu-Mn-Ni coherent precipitate and defect-solute cluster complex dislocation obstacles. Higher yield stress is accompanied by modest reductions in strain hardening and uniform strains, usually assumed to be due to dislocation cutting-destruction of the obstacles. A unified constitutive model is developed that properly combines all strength contributions (grain boundary, stress fields, pre-irradiation precipitates, obstacles and dislocation structures) between linear and root sum square superposition limits, and accounts for dislocation storage and annihilation mechanisms. Notably, although the net observed strain hardening is lower, due to superposition effects the intrinsic dislocation hardening (and density) is actually higher after irradiation, even assuming no obstacle destruction, probably due to reductions in the annihilation rate by suppressed cross slip. The database, novel experiments to measure flow stress at high strains and microstructural characterizations underpinning the model are described.

3:20 PM

Modeling Cascade Aging and Dose Rate Effects in Ferritic Alloys: *Brian D. Wirth*[1]; Paul Monasterio[1]; G. Robert Odette[1]; [1]University of California

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 Fe, Fe-Cu and Fe-Cu-Ni 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-solute 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 underconditions relevant to fusion reactor materials.

3:40 PM Break

4:00 PM Invited

Synergistic Effects of DSA and Radiation Defects on Mechanical and Fracture Characteristics of Pure Iron and Steels: *Indrajit Charit*[1]; Chang-Sung Seok[2]; K. Linga Murty[1]; [1]North Carolina State University; [2]Sungkyunkwan University

Ferritic steels commonly used for pressure vessels and reactor supports in light water reactors (LWRs) are known to exhibit radiation embrittlement in terms of decreased toughness and increased DBTT following exposure to neutron radiation. The superimposed effects of dynamic strain aging (or Portevin LeChatelier effect) on radiation embrittlement were considered first by Wechsler, Hall and others. We summarize here some of our work on these aspects with emphasis on synergistic effects of interstitial impurity atoms (IIAs) and radiation induced point defects, that result in interesting beneficial effects of radiation exposure at appropriate temperature and strain-rate conditions. A variety of ferritic steels are considered including pressure vessel steels as well as relatively pure (Armco) iron using tensile, 3 point bend and CT specimens. The superimposed effects of radiation on these steels and iron will be presented along with studies on fast vs. total (thermal+fast) neutron spectra that revealed unexpected results.

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On the Effects of Alloy Chemistry on the Composition of Copper and Manganese-Nickel Rich Precipitates in Irradiated Reactor Pressure Vessel Steels: *G. Robert Odette*[1]; Jonathan Smith[1]; Takuya Yamamoto[1]; Brian D. Wirth[2]; [1]University of California, Santa Barbara; [2]University of California, Berkeley

Irradiation embrittlement of reactor pressure vessel steels is primarily due to hardening caused by a high number density of Cu or Mn-Ni rich nm-scale precipitates. Modeling embrittlement requires understanding the thermodynamic relation between the alloy chemistry and the corresponding composition of the Cu-Mn-Ni-Si precipitates that form under irradiation. Predictions of both mean field and atomistic thermodynamic models, accounting for non-equilibrium effects of the interface at the nm-scale, are in good general agreement with observation, but they have not been rigorously verified by systematic experiment. In this work, we develop an experimental non-equilibrium Fe-Mn-Ni-Cu phase diagram for coherent transition phase precipitates at around 290°C, from both small angle neutron scattering and combined electrical resistivity-Seebeck coefficient measurement data on a large matrix of irradiated alloys with systematic variations in composition. The roles of Si and irradiation temperature are also discussed. The experimental phase diagram is used to fine-tune the thermodynamic models.

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Interaction of Plastic Deformation and Phase Transformations in Zr-Based Alloys at Temperatures of (α+β)-Region of Phase Diagram Zr-Nb: Yuriy Perlovich[1]; *Margarita Isaenkova*[1]; Sergey Kropachev[2]; Mikhail Shtutca[2]; Vladimir Filippov[2]; Oleg Bocharov[3]; [1]Moscow Engineering Physics Institute (State University); [2]Chepetckiy Mechanical Plant; [3]All-Russia Research Institute of Inorganic Materials

Development of plastic deformation in Zr-based alloys at temperatures of β- and (α + β)-regions of phase diagram Zr-Nb was studied by X-ray methods. Mechanisms, responsible for plastic deformation development by different temperature-rate regimes, were determined by texture changes, taking place in deformed samples. Among these mechanisms there are crystallographic slip in grains of β-Zr and α-Zr as well as diffusion displacements of crystallites along interphase boundaries. The latter mechanism becomes more intense under conditions of phase transformations and lies in the basis of superplasticity. Both α-Zr and β-Zr by deformation at temperatures of (α + β)-region show a phase instability, which can result in repeated phase conversions, accompanied by fragmentation of crystallites and intensifying of intergranular slippage. The latter leads to scattering of the texture, formed by operation of crystallographic slip. The greater is the input of slippage by interphase boundaries, the more uniform is the deformation of samples.