TMS2006

135th Annual Meeting & Exhibition

■ *Linking science and technology for global solutions*

Meeting Information

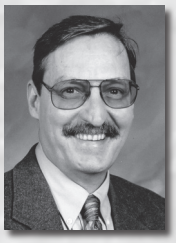
March 12-16, 2006
Henry B. Gonzalez Convention Center
San Antonio, Texas, USA

Technical Program Follows Meeting Information

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.

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

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- Free subscription to *TMS Letters*, the online technical journal providing updates to cutting-edge research in a concise format.
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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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Technical Program

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

WEDNESDAY

2006 Nanomaterials: Materials and Processing for Functional Applications: Nanomaterial Formation and Manufacture

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

Wednesday AM Room: 214C
March 15, 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
Dispersion and Alignment of Single Walled Carbon Nanotubes in Ceramic Slurries by Pot Milled Method: *Alok Vats*[1]; Haiping Hong[1]; Fernand D. Marquis[1]; James W. Sears[1]; [1]South Dakota School of Mines and Technology

Single walled carbon nanotubes have shown promising electronic, mechanical and thermal properties due to their unique structures and bonds. In this abstract, we report our attempts to understand the behavior of SWCNT in ceramic slurries modified through pot milling, sonification and other chemical routes. Since ball milling operations on SWCNT's show reduction in size and introduction of defect on the sidewalls, depending on the duration of milling which contribute to change the morphology and properties of single wall nanotubes, we tried to incorporate and disperse nanoscale ceramics onto the SWCNT through the above techniques. The preliminary results of the Transmission Electron Microscopy (TEM) show that SWCNT are dispersed very well by the treatment of pot milled method. More interesting electrical and thermal properties are expected by using these kinds of materials. It may open the new route towards better utilizing the SWCNT.

8:55 AM
Synthesis of Carbon Nanocapsules by an Electric Plasma in Ultrasonic Cavitation Field of Liquid Ethanol: *Ruslan Sergiienko*[1]; Etsuro Shibata[1]; Zentaro Akase[1]; Hiroyuki Suwa[1]; Takashi Nakamura[1]; Daisuke Shindo[1]; [1]Tohoku University

Nanoparticles of iron carbides wrapped in multilayered graphitic sheets (carbon nanocapsules) were synthesized by a newly developed method in which an electric plasma was flashed in an ultrasonic cavitation field in liquid ethanol. Ultrasonic cavitation causes a very highly localized high temperature and pressure field where tiny bubbles are collapsing. An ultrasonic cavitation field, with its many activated tiny bubbles, enhances electrical conductivity due to the radicals and free electrons formed within it. Thus, an electric plasma can be generated at relatively low power, even in insulating organic liquid mediums. The size of the carbon nanocapsules ranged between 5 and 600 nm. The crystal structures of the spherical cores were found to be orthorhombic Fe_3C and monoclinic χ-$Fe_{2.5}C$. Some spherical particle cores were discovered in the amorphous state. The magnetic hysteresis loop of the nanocapsules at room temperature showed the saturation magnetization of 51.36 Am^2/kg and coercivity of 3.4 kA/m.

9:15 AM Break

9:25 AM
Formation of Bulk Nanocrystalline Grains in Titanium by Friction Stir Processing: *Zhili Feng*[1]; Oleg M. Barabash[1]; Stan A. David[1]; Joe A. Horton[1]; [1]Oak Ridge National Laboratory

We report the formation of bulk nanocrystalline grains in commercially pure Titanium by friction stir processing (FSP). In the FSW experiment, a specially designed rotating refractory tool induces severe plastic deformation near the surface of the Ti specimen, accompanied by a considerable temperature rise as the result of high-strain rate deformation and friction. The translational movement of the rotating tool makes it possible to process a large surface area of the material. The hardness in the processed region exceeds 300 Hv, compared to the base metal value of less than 200 Hv. Transmission electron microscopy (TEM) analysis revealed that the grain size in the processed region is in the range of 20 to 60 nanometers (nm). The basic principles of FSW as applied to producing nanocrystalline grains will be described, as well as the microstructural features of the processed region.

9:45 AM
Nanostructures and Mechanical Properties Developed in Copper by Severe Plastic Deformations: *Michal Besterci*[1]; [1]Institute of Materials Research of Slovak Academy of Sciences

The development of the nanostructure in Cu and the strength and ductility after severe plastic deformation (SPD) with the technology of equal channel angular pressing are analyzed. Results showed strength and ductility can be increased simultaneously by SPD. The changes in grain size and yied strength followed in some range the Hall-Petch equation. The grain size decreased with the increase of deformation, after 10 passes to 100-300 nm. The stress-strain curve of the deformed test piece after 10 passes showed a "plato", showing the presence of superplastic like deformation mechanism, we suppose sliding and rotations of grains, and the same can explain the increase of the reduction of area after severe deformation. TEM analysis suggested the possible nanostructure formation mechanism by the formation of cellular grain structure, forming of subgrains and then forming of large angled nanograins with random orientation.

10:05 AM
Bulk Nanomaterials through Powder Consolidation by Twist Extrusion: *Victor Varyukhin*[1]; Yan Beygelzimer[1]; Viktor Tkach[1]; Viktor Maslov[1]; Sergey Synkov[1]; Aleksandr Sergeevich Synkov[1]; Viktor Nosenko[1]; [1]DonFTI

We propose a theory of powder consolidation by twist extrusion. It is based on the continuum model of powder body with representative volume element which satisfies the quadratic yield condition. We show that intensive shear under high pressure at the end points of the twist channel and the intensity of material flow in the cross-sections of the billet provide favorable conditions for powder consolidation. The TE technique has been used for consolidation of macroscopically ductile amorphous Al86Ni6Co2Gd6 ribbons produced by melt-spinning processing. The fully billets with dimensions of 14×23×40 mm^3 were produced at extrusion temperatures above 523 K, while the maximum microhardness (550 kgf/mm^2) was reached for the bulk billet consolidated at 513 K with the nanocomposite structure consisted of about 57% of Al-nanocrystals with size about 13 nm embedded in residual amorphous phase.

10:25 AM Break

10:35 AM
Nanostructure Materials Fabricated by Ion Beam Mixing: Sufian Abedrabbo[1]; *Dia-Eddin Arafah*[1]; Nuggehalli Ravindra[2]; [1]University of Jordan; [2]New Jersey Institute of Technology

Special nanostructure materials and thin films/alloys are processed utilizing the novel technique of Ion Beam Mixing (IBM). Examples include shallow alloy formation of engineered band gap Si-Ge for solar cell applications and GeO2 islands and continuous nano-dimensional films by subsequent annealing. Characterization processes include investigations of the structural variations noted due to Argon beam irradiation to various fluences by Rutherford Backscattering (RBS), shallow defects and deep trapping level states by Thermo luminescence (TL), X-ray Diffraction (XRD) are performed.

10:55 AM
Self-Assembly of Amphiphilic Triblock Copolymers into Various Nanostructures: *Wei Jiang*[1]; [1]Institue of Applied Chemistry

Self-assembly of ABA (P4VP-b-PS-b-P4VP) and ABC (PS-b-P2VP-b-PEO) amphiphilic triblock copolymers in selective dilute solution were

investigated and various novel nanostructures, such as ring-shaped aggregates, biomimetic segmented worm-like micelles, ring-ring nesting micelles, among others were obtained. Micellar morphology formation mechanism was explored by combining experimental and computer simulative method. Moreover, the micellar morphologies of ABA triblock copolymer in dilute solution were tuned by using many facile methods, including changing common solvent properties, hydrogen coupling between one of the block of copolymers and small molecular surfactants, binary blending of copolymers and homopolymer of core-forming blocks (PS blocks) addition method. The micellar morphologies can be conveniently tuned into spheres, rods, vesicles, et al by using these methods. Finally, we have successfully fabricated various metallized nanostructures by coating metallic nanolayers onto the copolymer micellar templates through simple electroless plating methods. In particular, bimetallic nanostructures have been obtained by using simple methods.

11:15 AM

Architectural Transformation on Nanoparticles to Nanowires in Applied Field: Harish Nathani[1]; *Jagdish Rawat*[1]; Devesh K. Misra[1]; [1]University of Louisiana at Lafayette

We present a novel room temperature fabrication of nanowires of about 8-10 micrometers in length and about 100-200 nm in diameter. The magnetic behavior was studied by Superconductivity Quantum Interface Device (SQUID) magnetometer. Most importantly, the nanowires exhibit higher coercivity at 2K and are paramagnetic. This behavior is in contrast to nanoparticles that indicated high remanence and low coercivity.

11:35 AM Break

11:45 AM

Optical Characterization of Ag Nanoparticles Embedded in Aluminophosphate Glass: Sergiy Lysenko[1]; *Jose A. Jimenez*[1]; Guangjun Zhang[1]; Huimin Liu[1]; [1]University of Puerto Rico

The physical properties of small metal particles, including their optical properties are strongly dependent on local structural and geometric parameters of particles caused by the preparation process. Silver nanoparticles (NP) embedded in aluminophosphate glass were prepared by melt and heat treatment processes. A red shift of the surface plasmon resonance (SPR) peak is observed as particle size increases, with the quadrupolar plasmon excitation being prominent for the more intensely heat treated samples. Low-temperature photoluminescence studies of Ag NPs embedded in the glass matrix show a broad band emission at ~410 nm with a dip which has a minimum at wavelength corresponded to dipole SPR mode in spherical Ag NP. Such a dip is assigned to reabsorption of Ag_2O emission by NPs. Time-resolved femtosecond pump-probe reflectivity measurements have shown the particle size-dependent nonlinear optical dynamics on a picosecond time scale. The relaxation mechanism of the electron-phonon system will be discussed.

12:05 PM

Experimental Study on Particle Size and Crystalline Phase of Titania Synthesized by Vapor Phase Oxidation Route: *Wang Zhi*[1]; Yuan Zhangfu[1]; Guo Zhancheng[1]; [1]Chinese Academy of Sciences

Crystalline TiO2 particles were produced in a tubular flow reactor by chemical vapor reaction using titanium tetrachloride as a starting material in oxygen containing atmospheres. The resulting powders were characterized by scanning electron microscope, transmission electron microscope and x-ray diffraction. The influence of temperature rules on titania showed that the particles size was controlled by the reaction temperature, the oxygen preheated temperature and the cooling fashion, but the particle crystalline phase was predominately anatase and hardly changed with the variation of the temperature, which was supposed due to the short residence time.

3-Dimensional Materials Science: 3-D Atom Probe

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

Wednesday AM — Room: 205
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: David N. Seidman, Northwestern University; Chantal K. Sudbrack, Northwestern University

8:30 AM Invited

Applications of the Leap Tomograph to Three-Dimensional Materials Science: *Thomas F. Kelly*[1]; David J. Larson[1]; [1]Imago Scientific Instruments

Atom probe tomography provides 3-D atomic-scale structural and compositional analysis of materials that complements and supersedes other high-performance techniques such as TEM and SIMS. With recent developments in Local Electrode Atom Probe or LEAP® technology by Imago Scientific Instruments, the atom probe's compositional imaging capabilities are now available for a wide spectrum of materials. The materials to be analyzed can be prepared in either the traditional needle-shaped geometry or in the form of microtips residing on planar surfaces. Imago's LEAP technology also enables a cross-sectional area up to 100 nm and requires only minutes of data collection. The end result is that much larger volumes of material may be rapidly accessed with the atom probe microscope than has previously been possible or practical. In this talk, application of atom probe tomography to a wide range of materials studies including magnetic storage materials, semiconductor materials, and metals will be discussed.

8:55 AM Invited

The Benefits of 3D Microscopy at the Atomic Scale: *Alfred Cerezo*[1]; [1]University of Oxford

The 3-dimensional atom probe (3DAP) permits analysis of the nanochemistry of materials with near-atomic spatial resolution, and was one of the first 3D-microscopy techniques in materials science. With 3-dimensional data, it is far easier to interpret complex microstructures and not only investigate the shape and orientation of features but also determine the proximity between different phases and the degree of interconnectivity. Other types of topological characterisation also become possible, such as the fine-scale roughness or the fractal dimension of an interface. 3D shape information is important in making an accurate determination of interface segregation, and may also give some indication of relative surface energies. This paper will review a number of studies in which the 3-dimensional nature of the data from the 3DAP has been important in the study of nanostructured engineering materials.

9:20 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.

9:45 AM
Atom-Probe Tomography Study of Precipitation-Strengthened Aluminum Alloys: *Keith E. Knipling*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University

We seek to develop new castable and heat-treatable, precipitation-strengthened aluminum alloys with coarsening- and creep resistance beyond 375°C. Decomposition of supersaturated Al(Zr) solid solutions occurs initially by the formation of nanoscale metastable $L1_2$ Al_3Zr precipitates. However, segregation during peritectic solidification results in an inhomogeneous dendritic distribution of Zr, creating interdendritic precipitate-free zones in the precipitated microstructure. By adding Sc, a eutectic solute which forms $L1_2$ Al_3Sc, solute partitioning occurring during solidification improves the inhomogeneous dispersion of precipitates. Furthermore, in the presence of Zr, Al_3(Sc,Zr) precipitates form with improved thermal stability compared to binary Al_3Sc. We analyze via atom-probe tomography the temporal evolution during thermal exposure of the structure and chemistry of the complex precipitate structure formed from the nonuniform initial solute distribution. We compare our results with previous work on Al(Sc,Zr) alloys with low Zr/Sc ratios.

10:05 AM
3D Characterization of the Carbon Distribution in a Medium Carbon Steel: *Donald H. Sherman*[1]; Michael K. Miller[2]; Sudarsanam Suresh Babu[3]; Nan Yang[1]; [1]Caterpillar Inc; [2]Oak Ridge National Laboratory; [3]Edison Welding Institute

The microstructure of a medium carbon (0.26 wt% C), low alloy steel has been characterized by atom probe tomography. The microstructure of this predominantly martensitic steel was characterized in the water quenched and the quenched and tempered at 215C conditions. The carbon distribution was determined in a local electrode atom probe. This wide angle instrument enables significantly larger samples to be obtained more rapidly than in previous generations of three-dimensional atom probes. A discussion of the local variations in carbon level and formation of ultrafine carbides 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.

10:25 AM Break

10:45 AM
Temporal Evolution of the Sub-Nanometer Compositional Profiles across the γ/γ'-Interface in a Model Ni-Al-Cr Superalloy: *Chantal K. Sudbrack*[1]; Ronald D. Noebe[2]; David N. Seidman[1]; [1]Northwestern University; [2]NASA

Combining atom-probe tomography (APT) and lattice kinetic Monte Carlo (LKMC) simulations is a robust technique to analyze in 3D the earliest stages of solid-solid phase separation; that is, the genesis, microstructural evolution and compositional development of nanometer-sized precipitates. Employing sophisticated computer-based analytical methods, the early-stage phase separation in a Ni-5.2 Al-14.2 Cr atomic % superalloy, isothermally decomposing at 873 K, is investigated with APT. Sub-nanometer scale compositional profiles across the γ(fcc)/γ'($L1_2$) interfaces demonstrate that both the γ-matrix and the γ'-precipitate compositions evolve temporally. Observed chemical gradients of Al-depletion and Cr-enrichment adjacent to the γ'-precipitates are initially transient, consistent with well-established model predictions for time dependent diffusion-limited growth, and mark the first detailed observation of this phenomenon. Furthermore, it is shown that Cr atoms are kinetically trapped in the growing γ'-precipitates.

11:05 AM Invited
Local Electrode Atom Probe Characterization of Crept CMSX-4 Superalloy: *Michael K. Miller*[1]; Roger C. Reed[2]; [1]Oak Ridge National Laboratory; [2]Imperial College

The solute distribution in crept and annealed single crystal CMSX-4 nickel-based superalloy has been characterized from multi-million atom, three-dimensional data sets obtained with the local electrode atom probe. Solute-depleted and solute-enriched regions are evident on both sides of the γ- γ' interface. Rhenium partitioned to the γ matrix and acts as a solid solution strengthener. Ultrafine (~1 nm diameter) rhenium clusters containing up to ~10% Re were evident in the 10-nm wide Re-enriched region in the matrix close to the γ - γ' interface. No evidence of nanometer size Re clusters was observed in the central region of the matrix in any of the crept or annealed materials. 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.

11:30 AM Invited
GP Zones Structures in Al-Ag Alloys: *Emmanuelle A. Marquis*[1]; [1]Sandia National Laboratories

In the phase-separating Al-Ag system, intermediate metastable precipitates (Guinier Preston zones) are formed. However, the exact structure of the GP zones (ordering, Ag concentration) and the existence of temperature-dependent phases (ordered at low T and disordered at high T) is still matter of debate. Recent experimental observations have indicated that in the initial stages of decomposition, precipitates form with a surprising morphology consisting of a Ag-rich shell surrounding an Al-rich inner core. This particular structure had been hypothesized from SAXS experiments, but the exact crystallographic nature and Ag concentrations have not yet been determined. Through transmission electron microscopy and atom probe tomography measurements, the temporal evolution of the local Ag concentration in three-dimensions is investigated. These measurements provide key information for connecting to theoretical predictions of the faceting and interfacial sharpness, and provide a basis for explaining the interesting morphologies observed in this system.

11:55 AM
Dilute Al-Sc-Yb Alloys Studied by Atom-Probe Tomography: *Marsha E. Van Dalen*[1]; David C. Dunand[1]; David N. Seidman[1]; [1]Northwestern University

Small additions of Sc to Al lead to greatly improved mechanical properties due to the formation of nano-size Al_3Sc precipitates. In the present study, ytterbium was added as a ternary alloying element to further improve the mechanical properties of Al-Sc-X alloys. Utilizing LEAP atom-probe tomography, it is found that Yb forms precipitates in Al more rapidly than Sc, upon aging at 300°C. Initially, Al_3Yb precipitates form with radii of 1-2 nm. Al_3Yb precipitation is followed by slower diffusion of Sc to these precipitates. The resulting Al_3 ($Sc_{1-x}Yb_x$) precipitates show interfacial segregation of Sc to the a-Al/Al_3 ($Sc_{1-x}Yb_x$) heterophase interface. The number density of Al_3 ($Sc_{1-x}Yb_x$) precipitates formed is higher than that of binary Al-Sc alloys of similar volume fraction, thereby leading to higher hardness values.

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

Wednesday AM — Room: 214B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Craig Jensen, University of Hawaii; Carole Read, U.S. Department of Energy; Ragaiy Zidan, Savannah River National Laboratory

8:30 AM Invited
Materials Properties of Complex- and Perovskite-Hydrides: *Shin-Ichi Orimo*[1]; Yuko Nakamori[1]; Kazutaka Ikeda[1]; [1]Tohoku University

Complex hydrides have been regarded as potential candidates for advanced hydrogen storage media. Currently, two effective methods, 'par-

tial substitution by elements with larger electronegativities' and 'preparation of appropriate combinations', are investigated with the primary aim of destabilizing the complex hydrides for promoting their dehydriding reactions. On perovskite hydrides, fundamental properties such as formation abilities and dehydriding mechanisms are studied experimentally. We have then confirmed the occurrence of the dehydriding (and also rehydriding) reactions of the perovskite hydrides, similar to the reactions of the complex hydrides.

8:55 AM Invited

Synthesis, Stability and Crystal Structure of Al-Based Complex Hydrides: *H. W. Brinks*[1]; B. C. Hauback[1]; A. Fossdal[1]; M. S. Sørby[1]; C. M. Jensen[2]; [1]Institute for Energy Technology; [2]University of Hawaii

Alanates, metal hydrides based on the AlH4– unit, is one of the most promising groups of metal hydrides for reversible hydrogen storage at moderate temperatures, with possibilities of reaching 5 wt% reversible storage capacity. The pioneering work of Bogdanovic et al. in 1997[1] revealed that Ti additives increased the desorption kinetics of NaAlH4 and furthermore made re-hydrogenation possible. In order to get a better understanding of the reactions, detailed studies of the structure is essential. In particular, it is important to understand the nature of the additive in order to be able to improve the kinetics by design. Most of the Ti additive was after cycling found to be present as Al1-yTiy with y ≈ 0.15[2]. Structural studies and stability measurements will be shown for a number of other alanates, including bialkali-alanates like Na_2LiAlH_6 and K_2NaAlH_6. [1]B. Bogdanovic and M. Schwickardi, J. Alloys Comp. 253 (1997) 1.[2]H.W. Brinks, B.C. Hauback, S.S. Srinivasan, C.M. Jensen, J. Phys. Chem. B (2005) In press.

9:20 AM Invited

Physiochemical Pathway to Reversible Hydrogen Storage in Complex Hydrides: Jun Wang[1]; Armin B. Ebner[1]; *James A. Ritter*[1]; [1]University of South Carolina

The use of hydrogen as a renewable and clean energy source is one of the most exciting news items to hit this country in a long time. President Bush, during his State of the Union Address in February of 2003, pronounced 1.2 billion dollars to jump-start the Hydrogen Economy. The Hydrogen Economy represents not only freedom from the US dependence on foreign oil, which is a national security issue, but also a necessary step toward improving the environment by eliminating the release of carbon dioxide into the atmosphere due to the burning of fossil fuels. However, hydrogen storage is proving to be one of the most important issues and potentially biggest roadblock, when it comes to implementation of the hydrogen economy. Of the three options that exist for storing hydrogen, i.e., in a solid, liquid or gaseous state, hydrogen storage in a solid matrix is becoming more and more accepted as the only method to have any expectation of meeting the gravimetric and volumetric densities of the recently announced FreedomCar goals; and of all the known hydrogen storage materials, complex hydrides are showing considerable promise. To this end, a physiochemical pathway that facilitates reversible H2 storage in complex hydrides has been developed. The products of this physiochemical pathway represent perhaps the highest capacity, reversible H2 storage material known to date in low temperature ranges. This new pathway was utilized to create a 5 to 6 wt% reversible H2 storage material at less than 150°C from the complex hydride, lithium aluminum hydride. Moreover, this material reversibly stores approximately 3 to 4 wt% H2 at around 100°C. The unique feature of this off-board, physiochemical route is that it enables regeneration of this complex hydride material that to date has resisted regeneration through more conventional on-board routes that are being pursued with, for example, sodium aluminum hydride. This physiochemical pathway, or variations thereof, may also be amenable to fostering the reversibility of other higher capacity complex hydrides, like other alanates and boronates. With reversibility of the lithium aluminum hydride being achieved through this physiochemical pathway, optimization of this process is currently being studied, as is the applicability of this technique to other complex hydrides. The objective of this study is to present the most recent results obtained from the author's laboratory on the dehydrogenation and rehydrogenation properties and kinetics of lithium aluminum hydride and possibly other complex hydride systems.

9:45 AM Invited

Reaction of Hydrogen with DPB Organic Hydrogen Getter: *George L. Powell*[1]; [1]BWXT Y-12, L.L.C.

DPB (diphenyl butyldiyne typically mixed with 25 wt% C – 1 wt% Pd) is made into a hydrogen getter as porous pellets by Honeywell Federal Manufacturing and Technologies Kansas City Plant. The gettering reactions are very effective, and irreversible since the H_2 is converted into a hydrocarbon. A 2-volume system was used to titrate DPB pellets, as a series of pressure drop curves, from which the reaction rate was determined as a function of H_2 pressure and DPB reaction extent at ambient temperature. Methods are described for measuring and modeling reaction rate as a function of pressure and reaction extent, and the results compared to that for another organic hydrogen getter. Managed by BWXT Y-12 L.L.C. for the U. S. Department of Energy under contract DE-AC05-00OR22800.

10:05 AM Invited

AES and RBS Study of the Deactivation Mechanism of Palladium Coated Vanadium Alloy Membranes: *Stephen N. Paglieri*[1]; Venhaus J. Thomas[1]; Yongqiang Wang[1]; David R. Pesiri[2]; Robert C. Dye[1]; Craig R. Tewell[3]; Ronny C. Snow[1]; [1]Los Alamos National Laboratory; [2]SDC Materials; [3]Sandia National Laboratory

Metal membranes based on highly permeable metals such as vanadium may be used to purify hydrogen for use in fuel cells, compound semiconductor manufacturing, or the chemical industry. Foils of V-Cu coated with thin films of palladium or palladium alloy were fabricated and tested for hydrogen permeability and stability during operation at temperatures from 320-450°C. The vanadium alloy foils (~75 microns thick) were ion-milled and palladium coatings between 50 and 200 nm thick were applied to both sides in-situ, via electron beam evaporation PVD. The membranes were completely permselective for hydrogen. The rate of hydrogen flux decline at 400°C was gradual but accelerated quickly at higher temperatures. Hydrogen flux decrease was caused by metallic interdiffusion between the palladium coating and the vanadium alloy foil. Interdiffusion between various surface coatings and foils tested at different temperatures was characterized with AES depth profiles and RBS.

10:25 AM

Liquid Organic Hydrides for Hydrogen Storage: *Shinya Hodoshima*[1]; Yasukazu Saito[1]; [1]Tokyo University of Science

Liquid organic hydrides such as decalin, methylcyclohexane and tetralin have been proposed as hydrogen carriers for on-board or stationary application in this study. Both decalin and methylcyclohexane, which can store hydrogen with high capacities: decalin (7.3 wt%, 64.8 kg-H_2 / m^3); methylcyclohexane (6.2 wt%, 46.5 kg-H_2 / m^3), are suitable for operating hydrogen vehicles. Though the storage densities of tetralin are relatively low (3.0 wt%, 28.2 kg-H_2 / m^3), the rates of absorbing and desorbing hydrogen are much faster than the others, suggesting that tetralin is another choice for stationary use. Efficient hydrogen supply from organic hydrides under moderate conditions could be accomplished with the carbon-supported metal catalysts in the "superheated liquid-film states". Superheated catalyst layers gave high reaction rates and conversions at 210-280°C only under liquid-film conditions. Serious coke formation over the catalyst surface and excessive exergy consumption were therefore prevented simultaneously.

10:45 AM Break

11:00 AM Invited

Multiscale Heat and Mass Transfer Modeling and Optimization of Hydrogen Solid Storage Systems: Eustathios S. Kikkinides[1]; Maria Konstantakou[1]; Michael C. Georgiadis[2]; Theodore Steriotis[3]; *Thanos Stubos*[3]; [1]University of Western Macedonia; [2]Imperial College London; [3]National Center for Scientific Research, Demokritos

The operation of hydrogen solid storage tanks presents distinct challenges in process modeling and optimization. In order to make the method viable, the adsorption capacity of the absorbent must be such so as to allow storage of a sufficient amount of hydrogen at a relatively short filling time. Thermal limitations may need to be considered during the charging/delivery process to take account of potential safety and operational concerns. This work presents an integrated approach that formally exploits the benefits of material design and process design. Systematic simu-

lation and optimization studies are performed at two different length scales, adsorbent pore level and fixed bed adsorber level. A detailed mathematical model is developed, describing the hydrogen storage in the bed using as example a carbon-based material. The model is validated against experimental and theoretical literature results. Heat, mass and momentum transfer effects are modeled in detail.

11:20 AM Invited
Novel Organometallic Nanomaterials for Room-Temperature Reversible Hydrogen Storage: A First-Principles Prediction: *Shengbai Zhang*[1]; [1]National Renewable Energy Laboratory

First-principles total energy calculations reveal the mechanism for non-dissociative H2 adsorption on various organometallic nanomaterials ranging from (yet-to-be synthesized) transition metal-coated buckyballs and carbon nanotubes to (already-existing) metal carbide nanocrystals. Our study reinforces the notion that the relatively light-weighted 3d transition metal (TM) atoms are potential absorbers for H molecules, as the empty d orbital can bind dihydrogen with exceptionally high capacity at an energy range ideal for room temperature vehicular applications. However, upon H release, a collection of isolated TM atoms is unstable against spontaneous cohesion. We show that this difficulty may be overcome by "embedding" the TM atoms into various carbon-based organometallic nanostructures. In particular, the amount of H2 adsorbed on an Sc-coated C48B12 [ScH]12 could approach 9 wt%, with 30-40 kJ/mol binding energies. TM-coated carbon nanotubes also show similar binding characteristics although important differences do sometimes exist. Metallocarbohedrenes and TM carbide nanocrystals are different from the above TM coated carbon structures in that the TM atoms are coordinated into the carbon backbone. Still, a similar non-dissociative H2 binding mechanism is identified. These studies demonstrate the adaptiveness of the overall mechanism to different local chemical environments. Work was in collaboration with Y. Zhao, Y.-H. Kim, A. Dillon, & M. Heben and supported by the U.S. DOE/EERE under contract No. DE-AC36-99GO10337.

11:40 AM Invited
Characterization of Hydrogenation Behavior of NaAlH4 Utilizing Thermoelectric Power and Magnetic Analyses: *Angelique N. Lasseigne*[1]; M. Ashraf Imam[2]; Brajendra Mishra[1]; David LeRoy Olson[1]; [1]Colorado School of Mines; [2]Naval Research Laboratory

Hydrogenation behavior of the NaAlH4 intermetallics is investigated utilizing thermoelectric power and magnetic analyses. Using both thermoelectric power and magnetic susceptibility measurements on these intermetallics allows for confirmation and further insight into hydrogen's role in altering the material's electronic structure. Electron concentration changes with variations in stored hydrogen content, thus altering the filling of the electronic d-band and slope of the plot of the paramagnetic susceptibility as a function of the thermoelectric power coefficient. The electronic and magnetic property correlations to indicate the direction for further alloy development to optimize hydrogen storage will be discussed.

12:00 PM
Mechanism of Hydrogen Desorption in Li-N-H Hydrogen Storage System Catalyzed by Titanium Compounds: *Shigehito Isobe*[1]; Takayuki Ichikawa[1]; Satoshi Hino[1]; Hironobu Fujii[1]; [1]Hiroshima University

A solid-solid reaction between LiH and $LiNH_2$ catalyzed by a titanium compound proceeds with desorption of ~6mass% hydrogen and a quite small amount of ammonia. In this work, hydrogen desorption mechanism in the reaction from LiH+$LiNH_2$ to Li_2NH+H_2 under He flow atmosphere as a non-equilibrium condition was clarified by TDMS, TG and FT-IR analyses for the products replaced by LiD or $LiND_2$ for LiH or $LiNH_2$, respectively. The results suggested that the reaction progressed through two-step elementary reactions mediated by ammonia. Moreover, from the practical point of view, we carefully determined the amount of the ammonia contained in desorbed hydrogen for the Li-N-H system in a closed system. The results indicated that a quite small of ammonia thermodynamically coexisted in the desorbed hydrogen from the product although the ammonia could not be detected as emitted gas by TDMS under the non-equilibrium condition.

Alumina and Bauxite: Bayer Digestion 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

Wednesday AM — Room: 7B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Steve Healy, Alcan Queensland Research and Development Centre

8:30 AM Introductory Comments

8:40 AM
New Design for Digestion of Bauxites: *Rob Kelly*[1]; Dirk Deboer[1]; Mark Edwards[1]; Peter McIntosh[1]; [1]Hatch Associates

A number of process technologies are available for digestion of bauxites. Typically the major conceptual differences in these revolve around the design of the heating section employed to heat liquor and bauxite slurry to the desired digestion temperature, and in the design of the pressure vessels for extraction of alumina from the bauxite. However, the energy efficiency, operating and maintenance characteristics are determined by the overall design of the unit. This paper reviews technologies commonly used for both low and high temperature digestion. It then presents new designs that utilise tubular heating technology. Safety, technical, operational and cost attributes of the new designs are discussed.

9:05 AM
Digester Design Using CFD: *Jennifer Woloshyn*[1]; Lanre Oshinowo[1]; John Rosten[1]; [1]Hatch

In the Bayer process, dissolution of gibbsite and kaolinite and continued dissolution of desilication products occur in the digester train. Understanding the hydrodynamics of the digester is key to improving the extent of dissolution, and thus the extraction of alumina and re-precipitation of silica. Deviation from ideal plug flow results in a miscalculation of the slurry retention time, leading to losses of bauxite in the red mud, or an over-sized digester. To address this issue, the hydrodynamics of a digester train were modeled using computational fluid dynamics (CFD). The impact of column aspect ratio and inlet configuration on the slurry residence time distribution (RTD) were investigated. The RTD was used to estimate reaction extents and evaluate the effect of design parameters on performance. The modeling approach allows the inclusion of the digestion chemistry to directly evaluate the yield. Results show that the choice of slurry inlet configuration significantly impacts performance.

9:30 AM
Oxalate Removal by Occlusion in Hydrate: *Valérie Esquerre*[1]; Philippe Clerin[1]; Benoît Cristol[1]; [1]Alcan

Occlusion plays a significant role in the removal of oxalate at Alcan's Gardanne refinery. Up to 50% of the oxalate purge can be ensured by occlusion. This study investigates the occlusion phenomenon and identifies the action parameters that facilitate the control of the occlusion process. Successive precipitation cycle laboratory tests have been conducted in different operating conditions to ascertain the key factors. The addition of CGM appears to completely inhibit oxalate occlusion, whereas an increase in caustic concentration enhances the process. The impact of temperature is not clear, suggesting a counteracting effect of productivity and hydrate growth. Having a high level of precipitated oxalate doesn't ensure a high level of occluded oxalate. Possible mechanisms are proposed. The new operating instruction to maintain a high caustic concentration in the spent liquor has led to a high and lasting level of occluded oxalate.

9:55 AM
Organic Carbon in Indian Bauxites and Its Control in Alumina Plants: *Ramana Rao Venakata Kondapalli*[1]; Ram Nivas Goyal[1]; [1]Jawaharlal Nehru Aluminium Research Developement and Design Centre

The organic build up in alumina plants is known to cause not only various plant operational problems but also reduces the productivity and

quality of product hydrate. The organic situation in Indian alumina plants is still not alarming for the reason that the bauxites contain low organic carbon. However, every plant feels the impact of the organic build up as it becomes old. The rate of organic build up is plant specific and depends upon the digestion conditions such as digestion temperature and pressure, caustic concentration, TOC values and nature of organic compounds in the mineral. In this paper the influence of digestion parameters, TOC in Indian bauxites and influence of special additives on organic build up in Indian alumina plants are discussed. The details of various control methods investigated for removal of oxalates, humates and TOC for Indian plants are also presented.

10:20 AM Break

10:40 AM
Reaction Behavior between Calcium Silicate Hydrate and Aluminate Solution: Zhuo Zhao[1]; *Qingjie Zhao*[1]; Xiaobin Li[1]; [1]Chaclo

Calcium silicate hydrate is an important hydrate product of Portland cement. And it is also a very important substance in alumina production. If the silicate compound with no alumina, C-S-H, can be formed in the process of alkaline alumina production, aluminum and silicon will be separated completely. Using calcium hydroxide and sodium silicate as starting materials, two kinds of calcium silicate hydrates, CaO•SiO2•H2O and 2CaO•SiO2•1.17H2O, were hydrothermally synthesized at 120°C. The reaction rule of calcium silicate hydrate in aluminate solution has been investigated. The result shows that the reaction between calcium silicate hydrate and aluminate solution is mainly through two routes. Firstly Al replaces partially Si in calcium silicate hydrate. Secondly calcium silicate hydrate can react directly with aluminate solution, forming hydrogarnet and Na2O•Al2O3•2SiO2•nH2O. The concentration of Na2CO3 and NaOH have less influence on the stability of calcium silicate hydrates; meanwhile the Al(OH)4- concentration in aluminate solution affects those remarkably.

Aluminum Reduction Technology: Pot Control and Modeling

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

Wednesday AM Room: 7A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Gary P. Tarcy, Alcoa Inc

8:30 AM
The New Development of the ALPSYS® System Related to the Management of Anode Effect Impact: *Sylvain Fardeau*[1]; [1]Alcan

The development of automated process control has allowed aluminium producers to improve cell and potline control strategy. One of the features of the ALPSYS® system developed by Aluminium Pechiney is to manage and to correct abnormal or exceptional cell or potline situations. This particular point has already led to very significant improvements in anode effect rates. Given the impact of greenhouse gas (GHG) emissions from the aluminium industry, the ALPSYS® continuous improvement plan is now focusing on achieving even greater reductions in the frequency and duration of anode effects. This study compares the situation before and after ALPSYS® implementation at the St-Jean-de-Maurienne plant. It also shows the impact of feeding device crustbreaker chisel-bath contact equipment, optimized control cycle, and anticipated anode effect detection algorithms.

8:55 AM
Further Results from the Wireless Instrumentation of Hall-Héroult Cells: Micheal Schneider[1]; *James W. Evans*[1]; Paul Wright[1]; Donald Ziegler[1]; [1]University of California

Collaboration between Alcoa and UC, Berkeley has resulted in further experiments on of wireless sensors at Eastalco during 2004-5. The sensors were designed to measure the gas temperature in the pot exit duct and the heat flux through the pot shell. Dando (Light Metals 2004) has shown that the former (with wired sensors) signals open holes in the pot cover. Wireless measurements were intended to signal the same cell difficulty without the problems of making wired connections. The heat flux from the shell indicates the presence of sufficient ledge to ensure sidewall life. Sensors were connected to Crossbow MPR400 wireless motes running the TinyOS operating system. Early results were in a previous paper (Schneider et al. Light Metals 2005); the present paper reports on recent campaigns where duct temperatures and heat fluxes were measured over several days with results logged by a laptop and relayed by e-mail to the Alcoa Technical Center.

9:20 AM
Stability Analysis of Electrolysis Cells with MISTRAL: Morgan Le Hervet[1]; *Thierry Tomasino*[1]; [1]Alcan

The control of the electrolysis cell stability represents a major stake. For this reason, many numerical models were developed by considering a linear stability approach. The stability of the electrolysis cell was studied with an unsteady 3D model (MISTRAL) strongly coupling the equations of fluid mechanics and induction. Then, two specific magnetic configurations were analysed: one known to be stable, the other known to be unstable.From the evolution in time of the interface deformation associated to a spectral analysis (FFT), the stable case from the unstable one could be differentiated. A comparison with the linear stability model (ALUCELL) made it possible to bring up the differences as well as the advantages and disadvantages of the two numerical approaches.

9:45 AM
Busbar Sizing Modeling Tools: Comparing an ANSYS® Based 3D Model with the Versatile 1D Model Part of MHD-Valdis: *Marc Dupuis*[1]; Valdis Bojarevics[2]; [1]GeniSim Inc; [2]University of Greenwich

The main goal of a cell stability MHD model like MHD-Valdis is to help locate the busbars around the cell in a way that leads to the generation of a magnetic field inside the cell that itself leads to a stable cell operation. Yet, as far as the cell stability is concerned, the uniformity of the current density in the metal pad is also extremely important and can only be achieved with a correct busbar network sizing. This work compares the usage of a detailed ANSYS® based 3D thermo-electric model with the one of the versatile 1D model part of MHD-Valdis to help design a well balanced busbar network.

10:10 AM Break

10:20 AM
Comparison of MHD Models for Aluminum Reduction Cells: *Valdis Bojarevics*[1]; Koulis Pericleous[1]; [1]University of Greenwich

The self sustained waves at the aluminium-electrolyte interface, known as 'MHD noise', are observed in the most of commercial cells under certain conditions. The instructive analysis is presented how a step by step inclusion of different physical coupling factors is affecting the wave development in the electrolysis cells. The early theoretical models for wave development do not account for the current distribution at the cathode. When the electric current is computed according to the actual electrical circuit and if a sufficient dissipation is included, the waves can be stabilised. The inclusion of the horizontal circulation-generated turbulence is essential in order to explain the small amplitude self-sustained oscillations. The horizontal circulation vortices create a pressure gradient contributing to the deformation of the interface. The full time dependent model couples the nonlinear fluid dynamics and the extended electromagnetic field that covers the whole bus bar circuit and the ferromagnetic effects.

10:45 AM
Neural Model of Electric Resistance in Reduction Cells of Aluminum to be Applied on the Process Control: *Marcos Castro*[1]; [1]ALBRAS

This work describes the development and the implementation of a electrical resistance model for application in the control of voltage of aluminum Reduction cells. For the system modeling, and aiming to surpass the difficulties and limitations of using physical equations for the behavior of the system, it was considered the use of the Artificial Neural Net tech-

niques, that is capable to identify the variable through the acquisition of input and output signals of the process. Moreover, the model acts with equipment that is specific for tests called "JIGA", making possible the development, simulation and implementation of new control strategies without any direct interference in the process. This becomes system much more flexible and, with more trustworthy adjustments.

11:10 AM
PIV Measurements on Physical Models of Aluminium Reduction Cells: *Mark A. Cooksey*[1]; William Yang[1]; [1]CSIRO

A full-scale room temperature model of part of an aluminium reduction cell was used to study bubble behaviour and gas induced liquid flows. The model consisted of three industrially-sized anodes so that the effects of the walls did not dominate. The bubbles were generated by forcing air through a porous polymer that formed the base of each anode. The liquid flows were characterised using particle image velocimetry (PIV). The effect of a longitudinal slot in each anode was investigated and was shown to have a significant effect on the liquid flows.

11:35 AM
Numerical Analysis of the Collector Bar Current Distribution of a Reduction Cell: *Dariusz Kacprzak*[1]; Marcus Gustafsson[1]; Liren Li[1]; Mark Taylor[1]; [1]University of Auckland

The paper presents modelling results of current distributions in reduction cells. The work examines an idealised reduction cell to determine the design features responsible for non-uniformities in the collector bar currents and considers design modifications to improve the current distribution. The analysis showed that non-uniformity in the collector bar currents is often caused by unsuitable resistances connected in series and that the dominant role is played by the cathode bus bar and its connecting flexes. The upstream/downstream balance is influenced by longer and shorter current paths. The design work was aimed to reduce this first-order-effect. Further analysis showed that it is important to reduce the variation in the current distribution along each side. To reduce this second-order-effect, modifications of the flex geometries were requred. A before and after comparison of the cell electrical design and its impact on the riser currents and the magnetic fields is presented.

12:00 PM End

Amiya Mukherjee Symposium on Processing and Mechanical Response of Engineering Materials: Steady State Deformation of Materials - Part I

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

Wednesday AM Room: 217C
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Thomas R. Bieler, Michigan State University; Eric M. Taleff, University of Texas

8:30 AM Invited
The History of the Bird-Mukherjee-Dorn (BMD) Equation: *Avraham Rosen*[1]; [1]Mississippi State University

The talk will outline the scientific career of Prof. Amiya. K. Mukherjee from the time he was a post doctoral fellow in the lab of Prof. John E. Dorn. The author of this contribution knows him since then and cooperated with him on various research projects. Special emphasis will be given to the BMD equation that was presented for the first time at the Haifa 69' conference in Israel. In this equation the authors demonstrate, probably the first time, the importance of the structure and the sub-structure during steady state creep at high temperatures.

8:50 AM Invited
Comparisons and Contrasts in Creep Behaviour: *Geoffrey Wilson Greenwood*[1]; [1]University of Sheffield

Creep rate and ductility are uniquely related to stress, temperature and structure and correlations to assist in predicting the behaviour of many widely differing materials have emerged, particularly in the past 4 decades. Progress has also been made in the evaluation of anisotropic materials, on the influence of multi-axial stresses and of the influence of temperature changes. The extent and value of these correlations will be examined together with the identification of situations, especially in creep fracture, where chemical and microstructural features make predictive capability less reliable. The nucleation and development of creep damage continue to pose problems but useful information can be derived from experimental studies of notched specimens and from materials containing gas bubbles.

9:10 AM Invited
Inverse Problems in Stochastic Modeling of Mixed Mode Power-Law and Diffusional Creep for Variable Grain Size: *Rishi Raj*[1]; Jie Bai[1]; [1]University of Colorado

Analytical results for two kinds of inverse problems in mixed-mode creep are presently where the material behavior is strongly influenced by grain size distribution. In one problem the measurement of the power-law stress exponent is used to predict the grain size exponent. In the second problem the measurement of the stress exponent at two different strain rates, that are one to four orders of magnitude apart, is analyzed to estimate: (i) the standard deviation, and (ii) the median value of the grain size probability density function. Thus, for the first time, we have analytical methods for deducing the variability parameters of the microstructure from phenomenological measurements of material behavior.

9:30 AM Invited
Constitutive Modelling of Creep of Metallic Materials: Some Simple Recipes: *Yuri Estrin*[1]; [1]IWW, TU Clausthal

An approach to modelling dislocation glide controlled creep is presented. It is based on the evolution of dislocation density treated as an internal variable. The general constitutive modeling frame permits inclusion of microstructural features, such as grain size, volume fraction and size of second-phase particles, lamellae spacing in lamellar materials, etc. Examples of modeling creep in single-phase and particle reinforced metallic materials will be given. In the latter case, in situ evolution of particle population will also be considered. Finally, in addition to average quantities, effects of particle arrangement on the creep deformation will be discussed. A simple and transparent architecture of the models presented makes them to easy-to-use creep modeling tools.

9:50 AM Invited
On the Negligible Effect of Microstructural Variations Occurring during Steady-State Deformation on the Flow Stress: *Hael Mughrabi*[1]; [1]University Erlangen-Nuernberg

Experimental evidence shows that non-negligible microstructural variations occur during seemingly steady-state deformation in high-temperature creep and in cyclic deformation. These microstructural variations apparently have no significant effect on the flow stress. The experimental evidence will be reviewed. Reasons will be given why the flow stress is rather insensitive to these microstructural variations. It is proposed that, in steady-state deformation, there is a slow persistent increase of the overall dislocation density which occurs mainly in the form of geometrically necessary dislocations, giving, e.g., rise to increasing misorientations between neighbouring dislocation cells separated by cell walls which become increasingly thinner and sharper. In the Taylor flow-stress law, the increase of the dislocation density is more or less compensated by the lowering of the "arrangement" factor α, related to the reduced stress effectiveness of the dislocations.

10:10 AM Invited
On the Nature and Origin of Harper-Dorn Creep: *Farghalli A. Mohamed*[1]; [1]University of California

Harper-Dorn creep refers to the anomalous high-temperature deformation behavior first reported by Harper and Dorn in their studies on the creep of Al at very low stresses and temperature near the melting temperature. Experimental data obtained on Al under the condition of small strains

(~ 0.01) indicated that Harper-Dorn creep in Al was characterized by: (a) a creep rate that was linearly proportional to stress (Newtonian behavior), (b) an activation energy for creep that was nearly equal to that for self-diffusion, and (c) a creep rate that was independent of grain size. However, the results of recent experiments performed on Al under the condition of large strains (> 0.1) have revealed two important findings. First, Harper-Dorn creep is observed in Al only when the dislocation density in annealed samples is low. Second, Harper-Dorn creep not only is accompanied by boundary activities but also is influenced by purity level.

10:30 AM Break

10:40 AM Invited

Interfacial Creep in Thin Film Structures and Application to Interconnects in Microelectronic Devices: *Indranath Dutta*[1]; Michael Burkhard[1]; Chandrasen Rathod[1]; Vijay Sarihan[2]; [1]U.S. Naval Postgraduate School; [2]Freescale Semiconductor

Thin film structures in microelectronic devices can deform via unusual, scale-sensitive phenomena due to thermo-mechanical loads sustained during processing, or during service as part of a microelectronic package. In particular, large shear stresses may develop at interfaces during thermo-mechanical excursions, allowing the interface to slide via diffusional processes (i.e., creep). Such interfacial creep can be further augmented by superimposed electric currents (via electromigration) as the thin film features shrink to the nanometer regime in modern microelectronic devices, potentially causing severe reliability problems. In this paper, we will present experimental and modeling approaches for deriving constitutive laws for this phenomenon, discuss the potential role of interaction between electromigration and interfacial sliding, and present experimental and modeling results to provide insight into the effect of this phenomenon on potential damage/failure mechanisms within a microelectronic device. Supported by NSF grants DMR 0075281 and DMR 0513874.

11:00 AM

Transition of Creep Behavior in BCC, FCC and HCP Solid Solution Alloys of Binary Systems: *Hiroyuki Sato*[1]; Hiroshi Oikawa[2]; [1]Hirosaki University; [2]College of Industrial Technology

Creep behavior of binary solid solutions of cubic and hexagonal alloys are summarized and discussed in detail. Alloys which are strengthened by size misfit and show type-M(mMetal type)and type-A(Alloy type) behavior, are comppared with each other at the temperature range around 0.6Tm. Conditions of change in creep characteristics from type-A to type-M is overviewed from view points of both normalized critical stress and critical normalized strain rate. Concentration dependence of steady state creep rates, obtained experimentally by the authors in aluminum-magnesium (fcc and hcp), titanium-aluminum (hcp), titanium-vanadium (bcc) and iron-molybdenum (bcc) systems, are compared and discussed. Transition conditions between type-A and type-M are reasonably classified by either size-misfit parameter or non-dimensional normalized strain rate. Concentration dependence in type-M conditions is still inconsistent with investigators. Peculiar concentration dependence in alloys in type-M in high concentration solid solutions is also pronounced.

11:20 AM

Non-Contact Measurements of Creep Properties of Refractory Materials: *Jonghyun Lee*[1]; Richard C. Bradshaw[1]; Robert W. Hyers[1]; Jan R. Rogers[2]; Thomas J. Rathz[3]; James J. Wall[4]; Hahn Choo[4]; Peter K. Liaw[4]; [1]University of Massachusetts; [2]NASA MSFC; [3]University of Alabama; [4]University of Tennessee

State-of-the-art technologies for hypersonic aircraft, nuclear electric/thermal propulsion for spacecraft, and more efficient jet engines are driving ever more demanding needs for high-temperature (> 2,000°C) materials. At such high temperatures, creep rises as one of the most important design factors to be considered. Since conventional measurement techniques for creep resistance are limited to about 1,700°C, a new technique is in demand for higher temperatures. This paper presents a non-contact method using electrostatic levitation (ESL) which is applicable to both metallic and non-metallic materials. The samples were rotated quickly enough to cause creep deformation by centrifugal acceleration. The deformation of the samples was captured with a high speed camera and then the images were analyzed to estimate creep resistance. Finite element analyses (FEA) were performed and compared to the experiments to verify the new method. Results are presented for niobium and tungsten, representative refractory metals, at 2,300°C and 2,700°C respectively.

11:40 AM

Effects of Microstructure on Creep Properties of Commercial-Purity Molybdenum: *James R. Ciulik*[1]; Eric M. Taleff[1]; [1]University of Texas

The tensile creep behavior of commercial-purity, powder-metallurgy molybdenum sheet was determined using short-term creep tests at slow to moderate true-strain rates of 10^{-6} to 10^{-4} s^{-1} and temperatures between 1300°C and 1700°C. Standard strain-rate change (SRC) creep tests were used to measure the activation energy for creep (Qc) and the steady-state stress exponents within these ranges. Creep flow stresses at these temperatures were affected by dramatic microstructural changes that occurred during testing, resulting in sudden decreases in flow stress. Elongation-to-failure (EF) tests at constant strain rate illustrate this effect. The dramatic microstructural changes causing this behavior are attributed to dynamic abnormal grain growth (DAGG). The DAGG process is found to be temperature dependent and to require an initiation strain.

12:00 PM

High Temperature Microsample Characterization of Creep in Ruthenium Nickel Aluminide Alloys: *Daniel T. Butler*[1]; Brian Tryon[2]; Budhika Mendis[1]; Tresa M. Pollock[2]; Kevin J. Hemker[1]; [1]Johns Hopkins University; [2]University of Michigan

Modern thermal barrier coatings (TBCs) consist of four distinct layers: superalloy substrate, aluminide bond coat, thermally grown oxide (TGO) and ceramic top coat. Failure of the TBC is caused by spallation of the top coat. Bond coat plasticity has been shown to play an important role in this failure process, and the high melting temperature and increased dislocation activity observed in ruthenium aluminides make them promising candidates for bond coat applications. Here, we report on microtensile experiments conducted to measure the elevated temperature properties of (Ni,Ru)Al and (Ni,Ru)AlCoCr. Dramatic improvements in elevated temperature tensile strength, as compared to platinum modified nickel aluminide bond coats, have been realized. Stress relaxation experiments confirm similar enhancements in creep strength and have been successfully modeled using the Dorn description of power-law creep. The measured stress exponent (n=3) and activation energies (Q=150-250kJ/mol) will be discussed in light of TEM observations of the underlying dislocation structure.

12:20 PM

Characterization of Stress Rupture Behavior of Zr and Ti Alloys via Burst Testing: *Gollapudi Srikant*[1]; Brian Marple[1]; Indrajit Charit[1]; K. Linga Murty[1]; [1]North Carolina State University

An understanding of the stress rupture behavior of Zr and Ti alloy tubings is of primary importance in structural applications in energy technology. The stress rupture properties were evaluated using burst testing of closed-end thin-walled tubing at varied test temperatures and internal pressures. The rupture data are correlated using the Larson-Miller parameter. The uniform hoop strains were also measured along with rupture times from which the strain-rates were calculated. These results will be fit to Monkman-Grant relation with the aim of extrapolating to in-service stress levels. The activation energy for deformation will also be determined and the data correlated using Dorn parameters. Wherever possible, the results will be correlated with the biaxial creep data. Plans include investigation of deformation microstructures using TEM and the results to-date will be presented. This work is supported by the National Science Foundation grants DMR0101309 and DMR0412583.

Biological Materials Science: Bioinspired 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

Wednesday AM Room: 212A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Mehmet Sarikaya, University of Washington; Chris Orme, Lawrence Livermore National Laboratory

8:30 AM Invited
Molecular Biomimetics: Genome-Based Materials Science and Engineering: *Mehmet Sarikaya*[1]; [1]University of Washington

Functions of organisms are carried out by billions of proteins through predictable and self-sustaining interactions via molecular specificity and high efficiency leading to formation and self-assembly of controlled functional constructs, structures, tissues, and systems, at all scales of dimensional hierarchy. Through evolution, Mother Nature developed molecular recognition via successive cycles of mutation and selection. Structural control of inorganic materials at the molecular-scale is a key to synthesis of novel, practical material systems. With the recent developments of nanoscale engineering and the advances in molecular biology, we are now able to combine genetic tools with synthetic nanoscale constructs, and create a hybrid methodology, Molecular Biomimetics. We have adapted bioschemes, combinatorial biology, post-selection engineering, bioinformatics, and modeling to select and tailor inorganic-binding short, 7-60 aas, polypeptides, for use as nucleators, catalyzers, growth modifiers, molecular linkers and erector sets, fundamental utilities for nano- and nanobio-technology. Supported by ARO-DURINT and NSF-MRSEC Programs.

9:00 AM Invited
Printing Biological and Biomimetic Materials: *Paul Calvert*[1]; David Kaplan[2]; [1]University of Massachusetts; [2]Tufts University

Inkjet printing can be used to pattern layers of material on a substrate with a thickness of about 100 nm and a lateral resolution of about 20 microns. It can be seen as comparable to the building of biological tissue, layer by layer, by a sheet of cells. This talk will cover printing of materials by chemical reaction between layers of different inks, by self-assembly and by direct printing of proteins. The printing of yeast and human cells will also be discussed.

9:30 AM
Biocompatible, Osteoconductive, and Resorption Properties of Synthetic Bone Substitutes Derived from Marine Structures: *Kenneth Scott Vecchio*[1]; Xing Zhang[1]; Jennifer Massie[1]; Mark Wang[1]; Choll Kim[1]; [1]University of California, San Diego

This project is focusing on optimizing the hydrothermal process to convert the aragonite/calcite to hydroxyapatite HAP or ß-tricalcium phosphate (ß-TCP), while maintaining the architecture of the marine structures, making the possibility of dense, well-structured, synthetic bone or strong, porous bone structures a realizable goal. Three important features of bone substitutes are their biocompatibility, osteoconductivity, and bioresorption properties, to facilitate bone in-growth and achieve a resorption rate comparable to the bone growth rate. Results from an in-vivo study using distal femur defects filled with the synthetic bone samples will be presented. Radiographically the position of the implants, possible absorption and evidence of fibrosis tissue can be determined during the implant period. Following the in-vivo implant period, electron microscopy and histology are used to establish new bone growth around and through the implant material, as well as the ability of the implants to serve as bone growth scaffolding.

9:50 AM
Biomimetic Syntheses of Inorganic Optical Materials Directed by Specific Peptides Isolated from a Phage-Displayed Library: *Matthew B. Dickerson*[1]; Gul Ahmad[1]; Ye Cai[1]; Kenneth H. Sandhage[1]; Nils Kroger[1]; Rajesh R. Naik[2]; Morley O. Stone[2]; [1]Georgia Institute of Technology; [2]Air Force Research Laboratory

Growing attention is being paid to the preparation of nanostructured oxide materials through biogenic or biomimetic approaches. Proteins isolated from biomineralizing organisms, or biological analogues (i.e., poly-l-lysine) to such proteins, have been used to direct the formation of silica or calcium carbonate. Although study of these naturally-occurring materials is certainly important, other oxide materials are often desired for advanced optical applications. In this work, a combinatorial phage-displayed peptide library was used to isolate and identify peptides exhibiting a binding affinity for selected inorganic optical materials, such as germania. Synthetically-produced peptides identified from the phase-displayed library, and the peptide-expressing phage, have been used to direct the nucleation and growth of optical oxides from precursor solutions. Correlations between the amino acid sequences of isolated peptides, and the nanoscale to microscale morphologies of oxide structures produced under their influence, will be discussed. The optical properties of the peptide-assembled oxides will also be presented.

10:10 AM Break

10:30 AM Invited
A Molecular View of Adsorbate Effects during Biomineralization: *Chris Orme*[1]; Jennifer Giocondi[1]; Molly Darragh[1]; Roger Qiu[1]; Jim De Yoreo[1]; [1]Lawrence Livermore National Laboratory

Recent research has shown that biologically inspired approaches to materials synthesis and self-assembly, hold promise of unprecedented atomic level control of structure and interfaces. With the advent of in situ probes such as atomic force microscopy, surface processes can be imaged in real-time, providing an unprecedented view of how organic molecules modulate growth. Direct measurables such as the atomic step velocity and the critical length for step motion, can be used to distinguish whether adsorbates induce energetic changes (for example by modifying the step-edge free energy or acting as surfactants) or whether adsorbates change kinetics (for example, by pinning the step motion or acting as catalysts). Using examples from calcium carbonate and calcium phosphate crystallization we show that the organic molecules that modulate biomineral formation exhibit all of these behaviors and collectively can be used to control macroscopic crystal shape and growth rate through step-specific interactions.

11:00 AM Invited
Tailoring of Inorganic Specific Polypeptides via Post Selection Genetic Engineering: *Candan Tamerler*[1]; Mehmet Sarikaya[2]; [1]Istanbul Technical University; [2]University of Washington

It is now well established that inorganic-specific polypeptides can be selected using combinatorial biology protocols, such as cell surface and phage display methods. While the first generation peptides selected as such can directly be used in biofabrication and assembly of inorganics, we now have adapted genetic engineering protocols to further tailor polypeptides with enhanced functionalities. In this process, combinatorially selected peptides are characterized experimentally and their molecular structures, sequence similarities are evaluated using computational modeling and bioinformatics. Similar to the natural evolution process, where recursive cycle of mutation and selection resulting in the progeny with improved features, these adapted procedures and second generation libraries are used iteratively for identification of binding domains critical in dictating the recognition mechanism. Example applications engineered polypeptides in nano- and nanobiotechnology include: i. Inorganic biofabrication through catalysis; ii. Molecular erectors, and iii. Synthetic/peptide hybrids. Supported by US-ARO-DURINT,and NSF-MRSEC program and State Planning Organization of Turkey.

11:30 AM
Effect of Polyethylene Pretreatments on the Biomimetic Deposition and Adhesion of Bone-Like Calcium Phosphate Films: *Kevin C. Baker*[1]; Jaroslaw Drelich[1]; [1]Michigan Technological University

Improving the deposition characteristics of biomimetic calcium-phosphate (CaP) films on polymeric substrates has the potential to enhance the lifetime and performance of many orthopedic devices on the market today. The effect of increasing the hydrophilic nature of polyethylene substrata on the deposition and adhesion of calcium-phosphate (CaP) films will be discussed. Ultrasonically cleaned samples undergo oxidation via either ultra-violet (UV) treatment, corona discharge (CD) processing, or immersion in chromic acid for specified times and conditions. After the conversion of predominantly hydrophobic polyethylene to a more hydrophilic surface, the samples are immersed in a supersaturated calcium phosphate solution (SCPS) to grow porous CaP coatings. The CaP coating coverage and morphology are examined with scanning electron microscopy (SEM). Coating compositions are determined by x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and energy dispersive x-ray spectroscopy (EDS). Substrate-coating adhesion is studied by performing scratch tests with a nanoindenter.

11:50 AM
Shock-Induced Index of Refraction Changes and Dynamic Cavitation Threshold in Water: *J. Michael Boteler*[1]; [1]NSWC-Indian Head

Applications of cavitation within the medical community range from lithotripsy to the use of bubble collapse jetting to penetrate cell sites for the purpose of drug delivery. As the physical understanding of microbubble collapse matures, emphasis for new experimental diagnostics and improved precision of existing diagnostic techniques becomes paramount. Specifically, many optical scattering techniques (Raman and IR spectroscopy etc.) require a precise understanding of the index of refraction (IOR) of the medium under investigation. Shock waves are known to alter the IOR and thus play a critical role. Modeling the complex phenomena of bubble nucleation, collapse, and jetting has proved difficult. A precise, dynamic value for the cavitation threshold is integral to the modeling effort. Purified water is examined in this study to quantify it's dynamic cavitation threshold, shock-wave induced IOR, and the feasibility of using bubble collapse and micro-jetting to react with dispersed 5 micron oxidized aluminum powder.

Bulk Metallic Glasses: Physical Properties

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

Wednesday AM Room: 217B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Robert W. Hyers, University of Massachusetts; Matthew J. Kramer, Iowa State University

8:30 AM Invited
Finding New Fe Based Bulk Amorphous Alloys with Minimum Number of Components: J. Zhang[1]; H. Tan[1]; *Yi Li*[1]; [1]National University of Singapore

Fe based bulk amorphous alloys have tremendous potential either as a structural material or as a magnetic material for functional application. Most of the large sized Fe based bulk metallic glasses discovered so far are multi-component with at least 6 elemental components and most of them are based on Fe, B and Rare earth. Starting from the simplest ternary Fe-B-Y system, new bulk metallic glass up to 1 mm was discovered using our recently developed pinpointing strategy. More importantly by carefully monitoring the microstructure changes, amorphous alloys with critical size large than 5 mm in Fe-B-Y based quaternary alloys have been discovered, which has never been reported before in a quaternary system. These alloys also have exceptional high fracture strength. We will discuss how these have been systematically found using our simple and practical strategy and the potential for further findings in Fe based alloys.

8:55 AM Invited
Measuring the Thermophysical and Structural Properties of Glass-Forming and Quasicrystal-Forming Liquids: *Robert W. Hyers*[1]; Richard C. Bradshaw[1]; Jan R. Rogers[2]; Anup Gangopadhyay[3]; Kenneth F. Kelton[3]; [1]University of Massachusetts; [2]NASA MSFC; [3]Washington University

The thermophysical properties of glass-forming and quasicrystal-forming alloys show many interesting features in the undercooled liquid range. Some of the features in the thermophysical property curves are expected to reflect changes in the structure and coordination of the liquid. These measurements require containerless processing such as electrostatic levitation to access the undercooled liquid regime. An overview of the state of the art in measuring the thermophysical properties and structure of undercooled liquid glass-forming and quasicrystal-forming alloys will be presented, along with the status of current measurements.

9:20 AM Invited
Anisotropic Free Volume Creation in a Metallic Glass during High Temperature Creep: R. T. Ott[1]; *Matthew J. Kramer*[1]; S. Bulent Biner[1]; M. F. Besser[1]; D. J. Sordelet[1]; [1]Iowa State University

Zr41.2Ti13.8Cu12.5Ni10Be22.5 metallic glass was homogenously deformed under isothermal uniaxial tensile loading at 598 K, 25° below the glass transition temperature. Total strain exceeded 80% for nominal tensile stresses of 250 and 400 MPa. High-energy synchrotron X-ray scattering and differential scanning calorimetery were used to examine strain-induced structural disordering. 2D detector allows for accurate measurements of the total scattering function in the longitudinal and transverse direction. The diffraction data and reduced radial distribution function demonstrate that the free volume parallel to the loading axis increases with increasing macroscopic strain. The free volume in the direction normal to the loading axis remained essentially constant for smaller strains. When the total strain exceeds 60%, the stress state becomes more triaxial due to the necking, resulting in an increase in the free volume in the transverse direction. DSC confirms that the amount of excess free volume is dependent on the gradient in plastic strain.

9:45 AM
Critical Analysis of Al-RE-Ni Glasses Using a Wedge-Casting Technique: W. S. Sanders[1]; J. S. Warner[1]; *Daniel B. Miracle*[1]; [1]U.S. Air Force

The critical thickness of Al glasses with rare earth (RE) and Ni additions has been established. Twelve RE additions were studied including Ce, Dy, Er, Eu, Gd, Ho, La, Nd, Pr, Sc, Sm, and Zr. A wedge-casting technique was used to ascertain maximum glass-forming ability of each composition and differential scanning calorimetry and TEM were used to evaluate glass thermal and structural characteristics. A strong linear correlation is found between the atomic radius of the RE element and the maximum amorphous thickness obtained in each ternary system. The maximum amorphous thickness ranges from <100 μm for Al-Zr-Ni to 780 μm for Al-La-Ni. These results reveal new insights into possible mechanisms of glass formation and structure, and offer a refinement of glass-forming criteria in Al-based glasses. New metallic glasses have been discovered in the Al-Ho-Ni, Al-Eu-Ni, and Al-Sc-Ni systems. The results obtained will be reported and discussed.

10:05 AM
Fabrication of Amorphous Alloy Surface Composites by High-Energy Electron-Beam Irradiation: *Kyuhong Lee*[1]; Sunghak Lee[1]; Nack J. Kim[1]; [1]Postech

Surface composites were fabricated with amorphous alloy powders by high-energy electron-beam irradiation, and their microstructure, hardness and wear resistance were investigated. Amorphous powders were deposited on a metal substrate, and then electron-beam was irradiated on these powders to fabricate one-layered surface composites. Two-layered surface composites were also fabricated by irradiating electron beam again onto the powders deposited on the one-layered surface composites. The microstructural analysis results indicated that a number of coarse crystalline phase particles were formed in the one-layered surface composite layers, whereas a small amount of fine and hard crystalline particles were homogeneously distributed in the amorphous matrix of the two-layered surface composite layers. Owing to these fine and hard crystalline particles, the hardness and wear resistance of the two-layered surface com-

posite layers improved over the one-layered surface composite layers. These findings suggested the possibility of applying amorphous alloy surface composites to wear- and thermal-resistant coatings or parts.

10:25 AM Break

10:35 AM

Metallic Glass Layers Produced by High Power Lasers – Applications and Opportunities: *David T. A. Matthews*[1]; Vasek Ocelik[1]; Jeff T. H. DeHosson[1]; [1]University of Groningen

The concepts surrounding glassy metallic alloys have been directed towards the production of thick (= 250 micrometer) amorphous surface layers on light metals such as aluminium and titanium, by harnessing the processing power of high power Nd:YAG lasers to achieve the inherently high cooling rates required to form many of today's bulk metallic glasses. Microstructural and chemical observation techniques include secondary electron microscopy, transmission electron microscopy, and X-ray diffraction, which reveal fully amorphous layers are attainable. Coating to substrate adherence is achieved by virtue of a functionally graded, often amorphous matrix, interlayer around 50 micrometer in depth. Thermo-dependant properties have been explored by differential scanning calorimetry and in-situ heating with transmission electron microscopy. Hardness and nano-indentation profiles reveal hardnesses up to 13 GPa over the full depth of a coating, coupled with elastic modulus around 150 GPa. Tribological tests have also been conducted and possible applications explored.

10:55 AM

Numerical Prediction and Experiments on Casting Fe-Based Bulk Amorphous Strips on a Twin Roll Caster and a Horizontal Single Belt Strip Caster: *Roderick I. L. Guthrie*[1]; Donghui Li[1]; Mihaiela Isac[1]; Roderick I. L. Guthrie[1]; Jim Wright[2]; Caian Qiu[2]; Francois G. Hamel[3]; Serge Turcotte[4]; [1]McGill University; [2]QuesTek Innovations LLC; [3]National Research Council of Canada; [4]IMI

The purpose of this work was to investigate the possibility of casting Fe-based bulk amorphous strip material using either twin roll and/or horizontal belt strip casting processes. Three kinds of Fe-based alloy were studied: ORNL-A1 (Fe-15B-7Co-7Mo-2Y-8Zr At.%), Darva101 (Fe-6B-15C-15Cr-14Mo-2Er At.%) and Darpa-Q21 (Fe-20B-2Zr-2Nb-1.5Cr-4.5V-2Y At.%), respectively. Maximum thicknesses of amorphous strip cast were predicted for various operating conditions for the two processes, and compared with corresponding experiments. Strip samples of 0.2~2mm thick were obtained. Darva101 alloy were cast on pilot-plant-scale twin roll caster. The strip samples' microstructures were analyzed by microscope, X-ray diffraction and SEM. Helium atmospheres were considered to be essential for enhancing cooling rates and lowering melt temperatures below the Tg values noted above. The possibility of casting Fe-based amorphous strip via the two casting processes was confirmed.

11:15 AM

Production of Bulk Metallic Glass Components by Low-Pressure Injection Die Casting: *Kevin Laws*[1]; Bulent Gun[1]; Michael Ferry[1]; [1]University of New South Wales

This study applies a repetitive low-pressure die-casting technique for optimizing the processing parameters for producing amorphous Mg-base alloy samples of cross section 3mm x 7mm and length 125mm for use in studies of elavated temperature mechanical behaviour. The aim of the present work is to establish the optimal combination of casting parameters to generate high quality cast components and, hence, reduce the variation in quality from a minimum number of experiments. In particular, the work investigates the effects of various die-casting parameters, including melt temperature, injection pressure and injection velocity on maximum cast length, porosity and internal microstructure of Mg-base alloys. The work has generated a comprehensive set of processing maps showing the optimized casting conditions for the production of these amorphous alloys.

11:35 AM

Recent Advances in Developing Large BMGs by the MANS Research Team: J. Xu[1]; C. L. Dai[1]; H. Ma[1]; Q. Zheng[1]; Y. Li[2]; *Evan Ma*[3]; [1]IMR, CAS; [2]National University of Singapore; [3]Johns Hopkins University

Encouraged by our recent success in developing inch-diameter Mg-based BMGs, the MANS research team has launched a new round of explorations to locate large BMGs in other systems. Cu-based systems, due to their strong potential for ductilization, are singled out as the next group worthy of special attention. Concurently, the team has been striving to derive decent plasticity from the large BMGs discovered. The over-riding theme is to invent BMGs that not only are large and inexpensive (based on engineering metals), but also possess mechanical properties (strength and ductility) that are practically useful in structural applications. This talk highlights some of the recent progress in these directions.

11:55 AM

Solidification Behavior of Fe-Base Amorphous Alloys during Twin-Roll Strip Casting: *Yoon S. Oh*[1]; Jung G. Lee[1]; Nack J. Kim[1]; [1]Pohang University of Science & Technology

Bulk amorphous alloys usually possess ultra-high strength, which are quite desirable for application as structural components. However, there are several obstacles which prevent their widespread applications such as high costs due to exotic alloying elements and limited fabrication methods and limited plasticity. In the present study, Fe-base bulk amorphous alloys were fabricated by twin-roll strip casting, which has been shown to be a cost efficient method for fabricating bulk amorphous alloy sheet products. Fe-base bulk amorphous alloys are interesting in that they are relatively inexpensive as compared to other bulk amorphous alloys. To improve the plasticity, microstructure of the alloys was modified to contain second phase of crystalline particles within amorphous matrix by control of alloy compositions and twin-roll strip casting conditions. Cooling sequence during twin-roll strip casting and continuous cooling transformation (CCT) diagram were simulated to determine the optimum process conditions at which desirable microstructures could be achieved.

Carbon Technology: Cathode Properties/ Refractory Materials

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

Wednesday AM
March 15, 2006

Room: 8A
Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Glen Goeres, Alcoa Aluminum

8:30 AM

Influence of Internal Cathode Structure on Behavior during Electrolysis Part III: Wear Behavior in Graphitic Materials: *Pretesh Mahendrabhai Patel*[1]; Margaret Hyland[1]; Frank Hiltmann[2]; [1]University of Auckland; [2]SGL Carbon GmbH

An extensive laboratory study has been undertaken to determine the effect of changes in internal structure on graphitized and graphitic cathode blocks used in aluminium reduction cells. Research has focused on sub-surface chemical and electrochemical wear. Previous work on graphitized material (published in Light Metals 2005) showed increasing the total porosity leads to uneven wear with pitting due to particle detachment becoming a noticeable feature of the surface topography. Results also showed that wear is dependant on the combined interaction of multiple variables, in particular the interaction between current density, bath chemistry and porosity. This paper focuses on recent work with a graphitic material. Compared to the graphitized materials, the graphitic cathodes show even wear phenomena with less surface pitting; this is due to the reduction in porosity in the graphitic material. The effect of variables such as current density, bath chemistry and porosity will also be addressed.

8:55 AM

A Study on the Property Changes of Cathode Blocks during Aluminum Smelting: *Fengqin Liu*[1]; Yexiang Liu[2]; [1]Zhengzhou Research Institute of Chalco; [2]Central South University

The properties of the waste cathode samples from the reduction cells with different working life have been studied in this paper. The chemical and mineralogical compositions of the waste cathodes and their physical

properties and microstructures have been analysed by SEM and XRD etc. The study results show that the volume density, compressive strength of the cathode samples are enhanced, and their conductivity is even increased by 30-50% after undergoing aluminum reduction process. It is found from SEM and XRD analysis that there appears a large amount of graphite in the waste cathodes and some cryolite, fluorides and metal aluminum penetrate into the gaps in the cathodes. It is suggested that the cathode blocks with higher strength and electrical conductivity and lower porosity and permeation property should be used in order to prolong the cathode working life.

9:20 AM

Wetting and Cryolite Bath Penetration in Graphitized Cathode Materials: Nikolai Shurov[1]; Nina Kulik[1]; Leonid Sitnikov[1]; Larissa Babushkina[1]; Victor Stepanov[1]; Yurii Zaikov[1]; Andrei Khramov[1]; Vyacheslav Malkov[1]; Alexander Gusev[2]; *Anton Frolov*[2]; [1]Institute of High Temperature Electrochemistry; [2]RUSAL

The special electrochemical cell was used in order to measure angles of wetting on different materials depending on cathode current density. Wetting of all samples was found to improve with cathode current rise. The dynamics of contact angles decrease depends on a type of material and the open porosity value. The investigation of bath penetration was carried out at 960°C under argon atmosphere in a special closed container made of the same cathode materials. Penetration depth was found to depend on open porosity. At the same conditions impregnated materials have lower penetration depth. The distribution of electrolyte components depending on distance from interface boundary has been investigated by local microspectral analysis method. The conclusions on different elements absorption mechanism were made on the basis of electrolyte components concentration in pores.

9:45 AM

Sodium Penetration into Carbon-Based Cathodes during Aluminum Electrolysis: *Jilai Xue*[1]; Qingsheng Liu[1]; Jun Zhu[1]; [1]University of Science and Technology Beijing

Sodium penetration into carbon-based cathodes during aluminum electrolysis was investigated in a laboratory cell. The sodium penetrations into the cathode specimens were determined against electrolysis time, electrolyte composition and current density. The results show the sodium penetration varies as various additives present in the carbon-based cathodes.

10:10 AM Break

10:25 AM

SiC in Aluminium Electrolysis Cells: An Update: *Rudolf P. Pawlek*[1]; [1]TS+C Technology Information Services and Consulting

The physical and chemical properties, the bond types and the resistance to bath, alumnium, carbon dioxide and air attack of SiC are reviewed, outlining especially the use of non-oxide bonded SiC refractories as sidewall materials for aluminium electrolysis cells. This type of SiC sidewall lining material is particularly suitable for the operation of modern electrolysis cells equipped with point feeders for automatic alumina feeding to the bath.

10:50 AM

A New Test Method for Evaluating Si3N4-SiC Bricks' Corrosion Resistance to Aluminum Electrolyte and Oxygen: *Xiaozhou Cao*[1]; Bingliang Gao[1]; Zhaowen Wang[1]; Xianwei Hu[1]; Zhuxian Qiu[1]; [1]Northeastern University

In the paper a new test method was applied to evaluate the bath resistance and oxygen resistance of silicon nitrite bonded silicon carbide bricks that were used for sidelining in aluminum electrolysis pots. In test, carbon dioxide gas was input to the molten electrolyte in which part of sample was immersed in order to simulating the factual operating conditions of cells. Based on the method the bath resistance and oxygen resistance of refractory bricks from several Chinese refractory factories were tested. The method was also used to evaluate the bath resistance of different parts of one brick. The test result showed that the inside of the brick had worse bath resistance than the outside, which may be caused by lower content of Si3N4 and higher content of impurities in the inner part of the brick.

11:15 AM

Test Method for Resistance of SiC Materials to Cryolite: *Junguo Zhao*[1]; Zhiping Zhang[1]; Wenwu Wang[1]; Guohua Liu[1]; [1]Luoyang Institute of Refractories Research

To obtain a stable and reliable test method for determination of the resistance of SiC materials to cryolite. This 'Rotating-CO2-Cryolite' test method has been designed by LIRR. The test specimens are being rotated in molten bath and CO2 and then placed in the test furnace to simulate the actual working conditions in reduction cells (pots). The influences of test temperature, holding time, CO2 flow rate and atmosphere inside the furnace (CO2, air and argon gas) on the cryolite resistance of Si3N4-SiC materials have been studied. The initial study shows that the optimized test parameters are 1000°C, holding time 24h, CO2 flow rate 1.0 L /min and rotating speed 30 rpm.

Cast Shop Technology: Casting, Solidification and Cast Defects

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

Wednesday AM — Room: 7C
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Steve Cockcroft, University of British Columbia

8:30 AM

Heat Fluxes at Metal-Mold Interface during Casting Solidification: *Adrian S. Sabau*[1]; [1]Oak Ridge National Laboratory

In this study, the heat transfer at the metal-mold interfaces is investigated using a sensor for the direct measurement of heat flux. Casting experiments were conducted using graphite molds for aluminum alloy A356. Several casting experiments were performed using a graphite coating and a boron nitride coating. The temperature of the mold surface was provided by the heat flux sensor while the temperature of the casting surface was measured using a thermocouple. Results for the heat transfer coefficients were obtained based on measured heat flux and temperatures. Four stages were clearly identified for the variation in time of the heat flux. Values of the heat transfer coefficient were in good agreement with data from previous studies.

9:00 AM

An Integrated Approach to Control Hot Tearing in Sheet Ingot DC Casting: *Benoit Commet*[1]; André Larouche[2]; [1]Alcan CRV; [2]Alcan ARDC

Among other defects, hot tearing is responsible for an important proportion of production losses. This paper presents an integrated approach to tackle this problem. The approach relies on fundamental understanding, experimental casting, mathematical modelling and application on particular situations. Simple, but promising hot tearing criteria are described. Criteria evaluate cracking sensitivities of alloys and provide an estimation of the castability in new product conception. Presentation of industrial cases is made. Also, realistic process conditions of application of the criteria are presented. Tuning of casting practices and/or development of new technology using this approach are highlighted. Various examples show that a rigorous approach integrating physical and numerical modelling provide an efficient tool to improve DC-cast slab quality and to reduce ingot defects increasing production recovery.

9:30 AM

Comparison of Experiments with Computer Simulations of Constant Unidirectional Melt Flow along Solidification Front: *Andrey Turchin*[1]; Dmitry G. Eskin[1]; Laurens Katgerman[2]; [1]Netherlands Institute for Metals Research; [2]Delft University of Technology

Effects of melt flow on structure formation during solidification were studied experimentally using a specially designed setup consisting of an electromagnetic pump and a melt transfer system with a built-in chill with the aim to correlate the structure and process parameters under conditions

of constant melt flow. In the present paper, CFD simulations of melt flow and solidification are applied as a tool to interpret the structure development under conditions of linear flow. Different geometries of the chill are tested in order to achieve laminar flow conditions in the widest possible flow velocity range. The computational results are validated against experimentally measured temperatures.The experimental and computational results show that flow parameters such as velocity and temperature affect the grain morphology, size and growth direction in a binary Al–Cu alloy.

10:00 AM

Studies in the Casting of AA6111 Strip on a Horizontal, Single Belt, Strip Casting Simulator: *Roderick I. L. Guthrie*[1]; [1]McGill University

Strips of AA6111 automotive alloy were cast on a horizontal single belt strip casting simulator. Chill substrates of pure copper and carbon steel were sprayed with various coatings for improving the strips' surface quality. The microstructures of the strip samples were studied by microscope and Scanning Electron Microscopy (SEM). The effects of casting parameters, including casting temperature and strip thickness, on interfacial heat fluxes, cooling rates and microstructures, were investigated. Very fine equiaxed grains were obtained for both 1.2mm and 2.5mm thick strips thanks to the rapid extraction of heat, and effective grain refinement procedures.

10:30 AM Break

10:45 AM

Billet Quality Assessments and Investigation of Fine Oxides in 6000 Series VDC Cast Product: Malcolm J. Couper[1]; *Barbara Rinderer*[1]; Ben Cumerford[2]; [1]Comalco Aluminium Ltd; [2]University of Queensland

The assessment of homogenised billet quality is routinely carried out for random samples from a range of billet diameters and alloys produced in the casthouse. The results of the assessments are tracked to confirm quality and process control. The laboratory evaluation includes ultrasonic and dye penetrant testing for inclusions and defects, hardness, macro etching for grain size and chill zone, as well as microstructural examination of intermetallics at the surface mid-radius and centre of the slices. The presence of fine oxides within the butt region of the billet have been investigated to determine their number, size and distiribution. Factors influencing the occurence of these oxides are discussed.

11:15 AM

Remelt Ingot Mould Heat Flow and Deformation: *John Francis Grandfield*[1]; [1]CSIRO

A study of mould heat transfer, air gap formation and mould deformation has been made with pure aluminum cast in a 22.5 kg ingot mould. A rig was build it which casting machine conditions were replicated for a single ingot allowing thermocouples to be placed in the ingot and the mould. Displacement transducers were used to measure the ingot and mould displacements. Thermal and stress modeling were conducted. The heat flow across the ingot/mould interface was found to be controlled by the formation of an air gap between the ingot and the mould. The air gap dimension is in turn a function of mould distortion and ingot contraction.

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

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

Wednesday AM Room: 206A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jian Li, Natural Resources Canada; Donato Firrao, Politecnico Di Torino

8:30 AM

Blunt Notch Specimens Fracture Behavior in Quenched and Tempered Tool Steels with and without Surface Treatment: *Donato Firrao*[1]; Daniele Ugues[1]; [1]Politecnico Di Torino

A comparison of fracture modes on impact tested quenched and tempered blunt notch die steel samples with varying notch root radii are described. Fracture mechanisms occurring on such samples is compared with that of nitrided specimens. On the basis of a previously developed model, different zones of the fracture surface are identified: (I) a "stress free zone", where multiple cracking formation at notch root provides a stress release in subsurface layers and actually creates a virtual larger notch radius; (II) a zone where the crack propagates along logarithmic spirals of a slip line field; (III) a zone where the crack finally rapidly propagates along the reduced section. The metallurgical structure of the surface layer is correlated with the aforementioned zones, considering the case of quench and tempered tool steels, where only mechanical working affected structures can be present, and of surface treated tool steel, where compound and diffusion layers exist.

8:55 AM

Characterization of Surface-Treated and Cold-Worked Nickel-Base Superalloys: *Hyojin Song*[1]; Peter B. Nagy[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati

The intentional introduction of near-surface compressive residual stresses, using method like laser shock peening (LSP), is well known for enhancing the resistance to fatigue crack nucleation and growth of turbine engine parts. Recently, nondestructive quantitative eddy current conductivity measurements have been successfully utilized to measure the state and degree of residual stress in shot-peened and cold worked nickel-base superalloys. However, the difficulty in separating the macro residual stress (elastic) and cold work (plastic) contributions to the conductivity has militated against the achievement of a complete analysis of the measured frequency-dependent apparent eddy current conductivity (AECC) results, and, furthermore, there is a lack of basic understanding of how intrinsic and extrinsic factors like microstructural changes brought about by surface and/or cold-work treatments affect the conductivity. The present study combines measurements of electrical resistivity/conductivity with detailed characterization of microstructure, residual stress distributions and quantitative analysis of surface-treated and cold worked Ni-base superalloys.

9:20 AM

Effect of Retrogression and Re-Aging Treatment on Stress Corrosion Cracking Resistance of 7075 Aluminum Alloy: *Murat Baydogan*[1]; Hüseyin Çimenoglu[1]; Sabri Kayali[1]; Jahan Rasty[2]; [1]Istanbul Technical University; [2]Texas Tech University

Effect of retrogression and reaging (RRA) treatment on hardness and stress corrosion cracking resistance of 7075 aluminum alloys was investigated. As received 7075 alloys in T6 tempered condition were retrogressed at 170, 220, 240 and 380°C for different durations between 15 s and 60 min. Subsequent re-aging was made at the T6 ageing condition. Treated materials exhibited higher hardness and better stress corrosion cracking resistance than T6 temper in a wide range of retrogression temperature and time. The highest hardness was obtained after retrogression at 170°C

for 210 s followed by re-aging, corresponding to 20% hardness improvement. Improvements in stress corrosion cracking resistance of the alloy by as much as 85% was also observed. Stress corrosion cracking resistance of the alloy was also improved dramatically. In this paper, improvements in hardness and stress corrosion cracking resistance are discussed on the basis of the microstructural changes during RRA treatment.

9:45 AM
Microsctructural Characterization of Mg Based Alloys: Apóstolos Jean Sideris Junior[1]; Ana Lucia Diegues Skury[1]; Guerold Sergevitch Bobrocnitchii[1]; *Sergio Neves Monteiro*[1]; [1]UENF

It known that Mg can act as a catalyst/solvent in the high pressure-high temperature diamond synthesis. However, above pressure levels of the order of 7 GPa, and temperatures from 1700-1800°C, it was not possible yet to use Mg for the industrial diamond production. A possible alternative in the use of MgNi based alloys. Moreover, owing to the great Mg reactivity, it was consider, so far, a difficult task to deal with such alloys. The present work presents successful results regarding the application of MgNi alloys, with different composition, at high pressure conditions. The behavior of the alloys and their microstrutured characterization is analysed.

10:10 AM Break

10:20 AM
Texture Evolution and Monotonic and Cyclic Response of Interstitial Free (IF) Steel after Severe Plastic Deformation at Room Temperature: *Guney Guven Yapici*[1]; Steve Sutter[1]; Ibrahim Karaman[1]; Hans J. Maier[2]; [1]Texas A&M University; [2]University of Paderborn

Present work investigates the mechanical behavior and texture evolution of severely deformed IF steel using equal channel angular extrusion. ECAE is applied by extruding bulk billets through two perpendicular channels of equal cross section achieving a simple shear deformation in a thin layer at the crossing plane of the channels. 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.

10:45 AM
The Characterization of the Intermetallic Fe-Al Layer of Steel-Aluminum Weldings: *Michael Potesser*[1]; Thomas Schoeberl[1]; Helmut Antrekowitsch[1]; Juergen Bruckner[2]; [1]University of Leoben; [2]Fronius International GmbH

One of the main targets in the automotive industry and supplying industry is the weight reduction of the vehicle for decrease the fuel consumption. For that reason combinations of different kinds of materials and connections for example steel and aluminium are developed or are already successfully field-tested. A determining factor concerning the quality assessment of the welded joint during hybrid welding is the characterisation of the intermetallic Fe-Al layer regarding strength, hardness, morphology and mainly the different phases in which Fe occurs together with the aluminium (FeAl, Fe2Al5, FeAl3.). A combination of conventional light microscopy, SEM - EDX - analysis , the usage of an atomic force microscope including a nanointender and theoretical thermodynamic calculations turned out to be highly effective. Based on the attained results the intermetallic FeAl layer, influenced by silicon, manganese and zinc, can be predicted.

11:10 AM
Microstructure of Nb-Si-Ti-Al-Cr-X Alloys for High Temperature Aeroengine Applications: *Hyojin Song*[1]; Raghvendra Tewari[1]; Amit Chatterjee[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati; [2]Rolls-Royce Corporation

Advanced intermetallic materials, in particular the refractory Nb-based silicides, possess a good combination of properties like high strength at elevated temperatures, high stiffness, low density, etc., which make them potential candidate materials for high temperature applications. However, the binary material exhibit low room temperature ductility and poor oxidation at elevated temperature. A multi element approach, which produces these silicides in equilibrium with the soft β-matrix, appears to provide a solution to the aforementioned problems. The present paper reports microstructures of as-cast and heat treated Nb-Si-Ti-Cr-Al based multi component system. The microstructural investigation has revealed the presence of four distinct phases, namely, the matrix β phase, the $Cr_2(Nb,Ti)$ Laves phase, which had dissolved and re-precipitated during various heat treatments, and Nb- and Ti- 5-3 silicides, in these alloys

11:35 AM
Local Thermal Property Analysis by Scanning Thermal Microscopy of Ultrafine-Grained Surface Layer in Copper and Titanium Produced by Surface Mechanical Attrition Treatment: *Fuan Guo*[1]; Nathalie Trannoy[1]; [1]Suzhou Institute for Nonferrous Metal Research

Ultrafine-grained surface layers were obtained by surface mechanical attrition treatment (SMAT) in copper and titanium samples, the thermal properties of the deformed layers were then characterized using a scanning thermal microscopy that allows thermal conductivity to be mapped down to the submicrometer scale. It is found that the microstructures obtained by SMAT show different thermal conductivities that strongly depend on the grain size: the thermal conductivity of the nanostructured surface layer decreases substantially if compared with that of the coarse-grained matrix of the sample. A theoretical approach, based on this investigation, was used to calculate the heat flows from the probe tip to the sample and then estimate the thermal conductivities at different scanning positions. Experimental results and theoretical calculation demonstrate that the SThM analyses open a new way for the thermal property and microstructural analysis of ultrafine-grained microstructures.

Computational Thermodynamics and Phase Transformations: Phase Field Models I

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

Wednesday AM — Room: 210A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jeffrey J. Hoyt, Sandia National Laboratories; Nikolas Provatas, McMaster University

8:30 AM Invited
Phase Field Simulations of Microstructural Development: Topology and Topological Singularities: R. Mendoza[1]; S. Hao[1]; K. Thornton[2]; *P. W. Voorhees*[1]; [1]Northwestern University; [2]University of Michigan

The evolution of microstructures using three-dimensional reconstructions and computations is investigated. Three-dimensional phase field calculations are employed along with three-dimensional reconstructions of microstructure to follow the evolution of a topologically complex dendritic microstructure during coarsening in two-phase mixtures, and grain structures in Ti alloys. By using the experimentally measured microstructures as initial conditions in the phase field calculations it is possible to compare quantitatively the experimentally measured microstructure with the calculations. For systems undergoing coarsening, phase field calculations are used to compute the average time rate of change of a given pair of principle interfacial curvatures at a point on the surface. The calculations also show the important role topological singularities play in the evolution of dendritic structures during coarsening. The results of calculations of grain growth that employ experimentally measured grain structures will also be presented.

9:00 AM Invited

Mixed Stress: An Approach to Mixing Fluids and Solids in Phase Field Models: *Adam C. Powell*[1]; [1]Massachusetts Institute of Technology

The Mixed Stress method combines elastic stress in a solid with viscous stress in a fluid in order to model phase transformations involving solids which move in response to fluid flow around them. The local weighting of the elastic and viscous stresses is a function of the local phase: in the fluid, only the viscous stress applies; in the solid, only the elastic stress (unless the solid is viscoelastic); in between, an interpolation function combines the two to produce smooth variation across a diffuse fluid-solid interface. This method is not optimal for all fluid-solid phase transformations; for example, liquid metals near their melting point behave more like very viscous fluids than elastic solids. This talk will therefore focus on application of mixed stress to other phenomena, such as electrochemistry and polymer crystallization, and discuss avenues for further development and application.

9:30 AM

Three-Dimensional Simulation and Characterization of Coarsening in Complex Microstructures Following Order-Disorder Transformation: *Yongwoo Kwon*[1]; Katsuyo Thornton[2]; Peter W. Voorhees[1]; [1]Northwestern University; [2]University of Michigan, Ann Arbor

Order-disorder transformation is observed in a variety of materials ranging from polymers to metals. This process frequently produces a complicated and highly interconnected 3D interfacial morphology possessing both positive and negative curvatures. Such a microstructure cannot be fully described by information obtained from its two-dimensional cross-sections. Characterizing the morphology of these structures in three-dimensions has proven challenging. We employ analyses of the three-dimensional curvature and the genus in two-phase microstructures obtained by large-scale phase-field simulations using the Allen-Cahn equation. The simulations are conducted in a high-performance parallel computing environment. We investigate the evolution of microstructures using quantifiable measures such as the interfacial shape distribution (the probability of finding a patch of interface with a given pair of principal curvatures) and topological quantities like the genus of the microstructures. We will discuss quantitatively the microstructural evolution and the presence of scaling using these characteristics.

9:50 AM

Phase Field in Polymeric Membrane Formation: *Bo Zhou*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology

A ternary phase field model is applied to simulate the polymeric membrane formation by phase inversion techniques. A ternary Cahn-Hilliard formulation incorporating a Flory-Huggins homogeneous free energy function is used. The Water/DMF/PVDF ternary system with a two-layer polymer-solvent/nonsolvent initial condition and with a mass-transfer boundary condition is used to simulate actual membrane fabrication conditions. 2D and 3D simulation results show the membrane morphology evolution during the spinodal decomposition. The simulated final morphologies in 2D show an asymmetric structure of membranes with a top layer, while 3D simulation results show a more symmetric structure. Simulations with different initial compositions show membrane morphology changes from isolated droplets to bi-continuous patterns. The effects of concentration-dependent polymer mobility are studied in both 2D and 3D. Different formula of variable mobility are adopted. In addition, the Navier-Stokes equations are coupled with this ternary Cahn-Hilliard to model hydrodaymic effects in 2D and 3D.

10:10 AM Break

10:30 AM Invited

Confronting Atomistic and Continuum Models of Solid-Liquid Interfaces: *Alain S. Karma*[1]; Kuo-An Wu[1]; Mark D. Asta[1]; Jeffrey J. Hoyt[2]; [1]Northeastern University; [2]Sandia National Laboratories

Major advances in both atomistic and continuum modeling of solid-liquid interfaces have been made over the last few years. On the one hand, the advent of the capillary fluctuation method has yielded an accurate determination of energetic and kinetic interface properties for a wide range of potentials. On the other, the phase-field crystal method inspired from density functional theory has emerged as a potentially powerful continuum simulation tool that enables simulations on much larger time scale than atomistic simulations, and naturally describes anisotropic interfacial properties related to crystal structure The phase-field crystal method is also closely related to older Ginzburg-Landau theories of solid-liquid interfaces based on order parameters that are the amplitudes of density waves corresponding to principle reciprocal lattice vectors. This talk explores the quantitative relationship between atomistic simulations, phase-field crystal models, and Ginzburg-Landau theories in the context of a detailed comparison of solid-liquid interface structure and excess free-energy for body-centered-cubic forming systems.Hopes and perils of respective continuum approaches are emphasized.

11:00 AM Invited

Phase Field Crystal Modeling: *Ken Elder*[1]; [1]Oakland University

Over the past several years a relatively simple phase field model has been developed to study elastic and plastic deformations in crystal growth phenomena. This simple model was used to study many interesting phenomena including epitaxial growth, yield strength in nanocrystals, grain growth, reconstructive phase transitions and ductile fracture. In this talk I would like to discuss recent developments in this modeling technique such as the incorporation of specific atomic potentials, extensions to three dimensions and binary systems.

11:30 AM

Finding the Critical Nucleus of Martensite with Phase Field Method: *Chen Shen*[1]; Ju Li[1]; Yunzhi Wang[1]; [1]Ohio State University

A mechanistic description of nucleation of martensite was proposed more than two decades ago by Olson and Morris, where a martensitic nucleus is formed from dissociation of a group of pre-existing lattice dislocations and the nucleation barrier is evaluated in a classical nucleation framework. This picture is adopted in a new phase field model originated from a treatment for dislocations at microscopic length scales. The model includes both dilatational and shear strain components and uses *ab initio* generalized stacking fault energy for quantitative calculations. The activation energy and morphology of a critical nucleus is sought in a non-classical nucleation scheme by finding the saddle point of the total energy consisting of a self-consistently described stacking fault energy and elastic energy.

11:50 AM

Analysis of Nucleation and Interfacial Mobility for Polygonal Ferrite Formation Using Phase Field Simulations: *Maria Mecozzi*[1]; Matthias Militzer[2]; Jilt Sietsma[3]; Sybrand van der Zwaag[3]; [1]Netherlands Institute for Metals Research; [2]University of British Columbia; [3]TU Delft

2D and 3D phase field simulations of the austenite (g) to ferrite (a) transformation during continuous cooling at different cooling rates were performed for a Fe-0.10 C- 0.49 Mn (wt%) steel. The initial austenitic microstructure and the nuclei density are input data based on experimental observations. Ferrite nuclei are assumed to form continuously over a temperature range of dT. The model considers carbon diffusion and employs an effective interfacial mobility with an activation energy of 140kJ/mol and a pre-exponential factor, m0, that, together with dT, is used as fitting parameter to optimise the agreement between the predicted and measured ferrite fraction. The best fitting combinations (m0, dT) have then been concluded by considering the ferrite grain size distribution that depends on dT as selection criterion. The model provides a reasonable description of the experimental data when dT and m0 are increased with cooling rate.

12:10 PM

A Novel Approach to the Calculation of Energy and Lattice Resistance to Sliding of Small-Angle Twist Boundaries: *Chen Shen*[1]; Ju Li[1]; Yunzhi Wang[1]; [1]Ohio State University

Grain boundary properties including grain boundary energy and sliding under stresses have been extensively studied with atomistic models such as molecular dynamics. At small angles the simulation becomes increasingly expensive with the size of the periodic cell. A recently developed phase field model for dislocations at microscopic length scales is extended to study twist grain boundaries via atomic disregistry fields in the boundary plane, which at small twist angles give rise to well defined dislocation networks. The model makes use of first principles γ-surface for quantitative study. It is first validated by comparing with Peierls model the calculated core structures of straight dislocations, and then applied to

simulating dislocation core structures of small-angle twist boundaries and to calculate their energies. Following the Peierls-Nabarro scheme the lattice friction for grain boundary sliding is calculated from the Peierls stress of constituent screw dislocations.

Deformation and Fracture from Nano to Macro: A Symposium Honoring W. W. Gerberich's 70th Birthday: Macroscopic Mechanical Behavior

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

Wednesday AM — Room: 214D
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Kumar V. Jata, U.S. Air Force

8:30 AM Invited
Fatigue Crack Initiation in Supergrains in Waspaloy at 20°C: *David L. Davidson*[1]; R. Tryon[2]; Michael E. Oja[3]; K. S. Ravi Chandran[3]; [1]Consultant; [2]VEXTEC; [3]University of Utah

Orientation microscopy (EBSP) was used to measure the Euler angles for grains that initiated fatigue cracks and for grains surrounding the initiating grain. It was hypothesized that cracks initiated preferencially in clusters of grains having similar orientations so that slip could be more easily transmitted across grain boundaries, thus making it appear as if these grain clusters were effectively one large grain (supergrain). Analysis of the results supports this hypothesis. Supergrain criteria were not satisfied in two non-crack regions used as controls. Grains in an area where a crack initiated from a void did not satisfy the supergrain criteria. A basis for predicting the sites for crack initiation in clean alloys has been established.

8:50 AM
Elevated Temperature Low-Cycle-Fatigue Behavior of HAYNES® 188® Superalloy: *Yulin Lu*[1]; L. J. Chen[1]; G. Y. Wang[1]; M. L. Benson[1]; P. K. Liaw[1]; S. A. Thompson[2]; J. W. Blust[2]; P. F. Browning[2]; A. K. Bhattacharya[2]; J. M. Aurrecoechea[2]; D. L. Klarstrom[3]; [1]University of Tennessee; [2]Solar Turbines, Inc.; [3]Haynes International, Inc.

Total strain controlled low cycle fatigue (LCF) tests with and without hold times were performed at temperatures ranging from 816 to 982°C in laboratory air on a cobalt-based superalloy, HAYNES 188. The influence of hold time on the cyclic-stress response and fatigue life was studied. Fracture surfaces and metallographic sections were evaluated in terms of the crack initiation and propagation modes, i.e., transgranular or intergranular. The introduction of a hold time led to a decrease in fatigue life. An increase in temperature and/or the introduction of a hold time decreased the stress hardening rate and increased the softening rate. Within the two phases of the fatigue process, crack initiation was more severely influenced by the change of hold time and/or temperature.

9:05 AM
Low-Cycle Fatigue Behavior of an As-Extruded AM50 Magnesium Alloy: Lijia Chen[1]; Chunyan Wang[1]; Wei Wu[1]; Zheng Liu[1]; Grigoreta M. Stoica[2]; *Peter K. Liaw*[2]; [1]Shenyang University of Technology; [2]University of Tennessee

The low-cycle fatigue behavior of an as-extruded AM50 magnesium alloy has been investigated. The cyclic stress response of the alloy strongly depends on the imposed strain amplitude. It is also noted that at the higher total strain amplitudes, the alloy exhibits a pronounced anisotropic behavior in the direction of tension and compression, where the width of the hysteresis loop in the tensile direction is greater than that in the compressive direction. At the total strain amplitude of 1.5%, a serrated flow can be observed in the compressive direction of the hysteresis loop. It means that the dynamic strain aging takes place during fatigue deformation. The relation between elastic and plastic strain amplitudes with reversals to failure shows a monotonic linear behavior and can be well described by the Basquin and Coffin-Manson equations, respectively. In addition, crack initiation and propagation modes are suggested, based on SEM observations on the fracture surfaces.

9:20 AM
Remarkable Dynamic Mechanical Properties of a "Trimodal" Al 5083/B4C Composite: Haitao Zhang[1]; Shailendra P. Joshi[1]; *K. T. Ramesh*[1]; Jichun Ye[2]; Julie M. Schoenung[2]; Ernest S. C. Chin[3]; [1]Johns Hopkins University; [2]University of California, Davis; [3]Army Research Laboratory

We have examined the mechanical behavior of a "tri-modal" Al5083/B4C composite in compression at both low and high strain rates. The material is tri-modal in that it incorporates (through cryomilling) a boron carbide particulate reinforcement within a nanostructured Al 5083 matrix, which is subsequently blended with unmilled (coarse grained) Al 5083. The composite powder is consolidated via cold isostatic pressing (CIP) and hot extrusion. The mechanical properties are evaluated through a combination of low strain rate testing with a servohydraulic testing machine and high strain rate testing using a compression Kolsky bar. The resulting mechanical properties are remarkable for an aluminum-based material, in that very high strengths and substantial ductility are both observed at both low and high strain rates. Very little rate sensitivity is observed. The primary failure mode in compression appears to be through shear band development. We discuss the deformation mechanisms that may produce the observed behavior.

9:35 AM
The Elastic and Plastic Strain Formation Near the Crack Tip of Stainless Steel During Fatigue by Neutron Diffraction: *Yinan Sun*[1]; R. Barabash[2]; H. Choo[1]; P. K. Liaw[1]; Y. Lu[1]; K. An[2]; F. Tang[2]; C. Hubbard[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

The deformation in the vicinity of the crack tip during fatigue tests was studied with neutron diffraction. Formations of large dislocation densities together with high residual stresses were observed. The inhomogeneous plastic deformation was determined. The anisotropic line broadening and lattice strain were observed at different distances from the crack tip. The dislocation density and arrangement were studied from the linewidth and profile behavior. Lattice strains and stresses were analyzed by the Rietveld-refinement technique. The comparison of the results and line-profile analyses facilitate the understanding of the change of dislocation densities and strains in the plastic zone. The dislocation density was found to decrease with the distance from the crack tip.

9:50 AM
Tensile Damage Evolution and Fracture Mechanisms of Nicalon/CAS Composites: *Jeongguk Kim*[1]; Peter K. Liaw[2]; [1]Korea Railroad Research Institute; [2]University of Tennessee

Infrared (IR) thermography was employed to study the tensile fracture behavior of Nicalon fiber reinforced calcium aluminosilicate (CAS) glass-ceramic matrix composites (Nicalon/CAS) with two different types of samples; cross-ply and unidirectional specimens. During tensile testing, an IR camera was used for in-situ monitoring of progressive damages of Nicalon/CAS samples in terms of the temperature evolution. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate fracture mechanisms of Nicalon/CAS composites. In this investigation, a thermographic NDE technique was used to facilitate a better understanding of fracture mechanisms of Nicalon/CAS composites during tensile testing.

10:05 AM
Structural Health Monitoring Research for Thermal Protection, Cryo and Hot Structures with Emphasis on Materials Aspects: *Kumar V. Jata*[1]; [1]U.S. Air Force

One of the major goals of our current research at the Air Force Research Laboratory is to develop structural health monitoring capability for real time interrogation of material and damage state awareness in thermal protection, cryo and hot structures. This talk will discuss various proposed material concepts and potential failure modes in these structures. Sensing methodologies required to perform real time interrogation of material and damage state awareness will be discussed using a few examples.

10:20 AM Break

10:40 AM Invited

Marine Forensics and Shipwreck Preservation: USS Arizona and RMS Titanic: *Tim Foecke*[1]; Jennifer Hooper-McCarty[1]; [1]National Institute of Standards and Technology

An important historical legacy lies with historically significant shipwrecks scattered around the globe. In conjunction with the National Park Service, Navy and NOAA, the Metallurgy Division of NIST provides technical assistance and finite element models of wrecks currently under preservation. This talk will detail work over the last 9 years investigating the sinking and deterioration of the RMS Titanic in the North Atlantic and the past 3 years work on the USS Arizona, a war memorial at Pearl Harbor in Hawaii. Results presented will include corrosion and bioencrustation studies, geological analyses, metallurgical studies and model predictions of wreck stability and predicted lifetimes.

11:00 AM

Fracture Toughness of a Zr-2.5Nb Tube with Temperature: *Young Suk Kim*[1]; [1]Korea Atomic Energy Research Institute

Fracture toughness tests were conducted on curved compact tension (CCT) specimens taken from a cold-worked and stress relieved Zr-2.5Nb tube at temperatures ranging from RT to 300°C. The CCT specimens were subjected to electrolytic charging of hydrogen to 200 ppm at a maximum. The as-received Zr-2.5Nb with no additional charging of hydrogen had an increased fracture toughness resistance, dJ/da with an increasing temperature with the maximum dJ/da around at 180°C, which corresponds to the g to d hydride transformation temperature. The Zr-2.5Nb tube with hydrogen of 100 to 200 ppm had a ductile-to-brittle transition at around 150 to 180°C and became as ductile as the as-received one at temperatures in excess of 180°C. The ductile-to-brittle transition behavior for the Zr-2.5Nb tube with hydrogen concentration and heat treatment conditions is discussed in association with the g to d hydride phase transition.

11:15 AM

Fracture Morphology Study of Hastelloy®2000®: *Rejanah V. Steward*[1]; Chang Kyoung Choi[1]; Gongyao Wang[1]; Peter K. Liaw[1]; Raymond A. Buchanan[1]; Dwaine Leroy Klarstrom[1]; [1]University of Tennessee

Hastelloy® C2000® Alloy is a commercially designed superalloy manufactured to function in reducing and oxidizing corrosive solutions. Its industrial applications have tremendous potentials in automotive, structural, aviation, and storage components. Albeit its good reducing and oxidizing traits in extremely aggressive media are attractive features of its chemistry, changes in the mechanical properties are believed to be insignificant due to its strong propensity to passivate under corrosive conditions. The ductility behavior and corrosion properties of C2000® are superior to that of stainless steels. The objective of this study is to thoroughly examine the fracture morphology of C2000® and understand the damage evolution from incipient crack-initiation to final fracture after subjection to fatigue in air and 3.5 wt. % NaCl solutions. Electron microscopy and in-situ macro-visualization were used to monitor crack initiation and propagation behavior of C2000®. The fatigue life was drastically reduced under maximum stress conditions and decreasing frequencies.

11:30 AM

Nondestructive Evaluation and Tension Behavior of Nicalon/SiC Composites: *Jeongguk Kim*[1]; Peter K. Liaw[2]; [1]Korea Railroad Research Institute; [2]University of Tennessee

The tension behavior of Nicalon fiber reinforced silicon carbide matrix composites (Nicalon/SiC) was investigated with the aid of nondestructive evaluation (NDE) techniques. The NDE techniques include ultrasonic testing (UT), X-ray computed tomography (CT), and infrared (IR) thermography. UT C-scans were developed to investigate defect distributions and to detect variations in the internal flaws. X-ray CT was used to characterize the type of defects and the location of flaws in composites to compare with UT C-scan results. IR thermography was conducted to generate thermal diffusivity maps for the samples. This paper also investigated the feasibility of using NDE techniques as integrity assessment means for Nicalon/SiC composites. IR thermography was also employed to measure temperature evolution during tensile testing. The tensile testing results showed quite reasonable agreement with previous NDE results. Microstructural characterization was performed using SEM to investigate failure mechanisms of Nicalon/SiC composites, and the results were compared with NDE data.

11:45 AM

An Overview of the Effects of Dispersed Particles on the Creep of Granular Ice: *Ian Baker*[1]; Min Song[1]; David M. Cole[2]; [1]Dartmouth College; [2]U.S. Army Cold Regions Research and Engineering Laboratory

Over 20 yrs ago, Professor Gerberich studied the effects of both crystal size and solid inclusions on the creep strain rate and activation energy for creep of ice [RW Baker and WW Gerberich, J. Glaciology 24 (1979), 179]. In this paper, we present an overview of the current understanding of the effects of inclusions on the creep of ice, and outline our recent findings on the effects of silt-sized particles upon the compressive creep rate and activation energy for creep of polycrystalline ice at temperatures from -20°C to -2°C. We have examined the situations when the particles are distributed only along the grain boundaries and when the particles are dispersed throughout the material. How the particles affect the dislocation densities present (as determined from cyclic loading tests), dynamic recrystallization and texture evolution will also be presented. Supported by NSF Office of Polar Programs, Arctic Natural Sciences Program OPP 011737.

12:00 PM

Microstructural Control of Ti-Al-Nb-W-B Alloys: *Lan Huang*[1]; Peter K. Liaw[1]; Chain T. Liu[1]; [1]University of Tennessee

TiAl alloys have been considered as promising candidates for structural-materials applications at around 800°C. But TiAl alloys are quite brittle at room temperature and have relatively low fracture toughnesses. In this work, new TiAl alloys containing W, B, and Nb have been developed. Fine uniform microstructures, with the colony size smaller than 50 um, can be conveniently developed after HIPing the as-cast alloys without any hot deformation process. Microstructural evolution of the alloys at temperatures ranging from 900°C to 1,310°C, were investigated. The a-phase transus temperature, Ta, has been determined. Different microstructures can be developed with the addition of alloying elements and related heat treatments. The effects of boron and tungsten on the microstructural evolution of the TiAl alloys were analyzed. The research is supported by the Fossil Energy Materials Program, with Dr. R. Judkins and Dr. J. Zollar as program managers.

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

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

Wednesday AM	Room: 216
March 15, 2006	Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Masahiro Endo, Fukuoka University; K. S. Ravi Chandran, University of Utah

8:30 AM Invited

Effects of Volume Fraction of Alumina Short Fiber on High Cycle Fatigue Property in Al-MMCs and Mg-MMCs: *Yasuo Ochi*[1]; Kiyotaka Masaki[1]; Takashi Matsumura[1]; Mitsushi Wadasako[2]; [1]University of Electro-Communications; [2]Nitias Company, Ltd

Rotating bending fatigue tests in high cycle region were carried out on alumina short fiber reinforced aluminum alloy composites (Al-MMCs) and magnesium alloy composites (Mg-MMCs). The matrix materials of an aluminum alloy A6061 and a magnesium alloy AZ91D, and both MMCs with three kinds of volume fraction of 10%, 18% and 25% of alumina short fiber were prepared. It was found that the fatigue strength increased with an increase of volume fraction of alumina short fiber in both MMCs, and also, the fatigue strength in Al-MMCs at elevated temperature was

improved by reinforcement of alumina short fibers. The crack initiation sites were large size alumina short fibers, cluster of short fibers and large size alumina particles in both MMCs. The crack propagation rates of MMCs decreased with an incease of volume fraction of alumina short fibers.

8:55 AM

Fatigue Crack Growth of Particle Reinforced Metal Matrix Composites: *Nikhilesh Chawla*[1]; V. V. Ganesh[1]; [1]Arizona State University

The fatigue crack growth (FCG) behavior of SiC particle reinforced 2080 Aluminum alloy was examined. The influence of matrix microstructure, particle volume fraction and size, and R-ratio on the FCG behavior of MMCs will be discussed. A fundamental relationship exists between ΔK_{th} and Kmax, which is influenced by the interaction between the fatigue crack, the SiC particles, and the size of the plastic zone ahead at the crack tip. In particular, the SiC particles induce significantly different damage mode than in the unreinforced alloy, particularly under compressive loading. The evolution of damage during fatigue crack growth will be demonstrated from in situ optical measurements of crack growth and the analysis of fracture morphology. The effects of particle morphology and distribution (homogenous and clustered) on crack growth were studied using a continuum-based approach. Predictions from this analysis correlated well with experimental observations of crack growth in these systems.

9:20 AM

The Role of SiC Particle Distribution Heterogeneity in the Very High Cycle Fatigue Behavior of Discontinuously-Reinforced Aluminum Metal Matrix Composites: *James K. Huang*[1]; Jonathan Edward Spowart[2]; J. Wayne Jones[1]; [1]University of Michigan; [2]U.S. Air Force

The role of microstructure heterogeneity on fatigue behavior of two extruded 2009/SiC/15p-T4 DRA composites has been examined in the very high cycle fatigue (VHCF) regime where 10^9 = Nf = 10^7 cycles. Ultrasonic fatigue was used to achieve the very high cycle counts. Both composites were carefully processed using identical powder blending and consolidation methods. One material was extruded to a 400:1 ratio and the other was extruded to a 14:1 ratio. Processing yielded materials with nearly identical compositions: a very homogeneous particle distribution with minimal clustering was produced in the 400:1 extrusion and a more heterogeneous of SiC particles was produced in the 14:1 extrusion. Crack initiation in the homogeneous, high extrusion ratio material was observed almost exclusively at AlFeCu inclusions while initiation at particle clusters was frequently observed in the material subjected to the lower extrusion. In all cases, fatigue lives at a given stress level exhibited very minimal scatter and subsurface crack initiation was observed in all cases. The role of microstructure variations in the fatigue behavior will be described.

9:45 AM

Fatigue of Titanium Alloys and Composites: *Winston O. Soboyejo*[1]; [1]Princeton University

This paper presents an overview of the fatigue of titanium alloys and composites. These include near $\alpha + \beta$ and β titanium alloys and composites reinforced with whiskers and fibers. The underlying mechanisms of fatigue crack growth and crack-tip shielding are elucidated using a combination of experimental techniques and theoretical models. The studies suggest that the effects of stress ratio can be explained largely as a result of crack closure phenomena. In particular, the role of roughness-induced closure is shown to be important, as identified in the original work of Art McEvily. The shielding contributions from roughness-induced crack closure are then quantified using fracture mechanics models. Subsequently, the mechanisms of fatigue are reviewed for crack growth in in-situ TiB whisker-reinforced and SiC fiber-reinforced composites. The crack/microstructure interactions observed in these composites are presented along with their effects on fatigue crack growth rates and fatigue life.

10:10 AM Break

10:25 AM Invited

Growth of Small Cracks and Prediction of Lifetime in High Temperature Alloys: *Luc Rémy*[1]; [1]Ecole des Mines de Paris

The lifetime to initiate an engineering crack is usually predicted from of S-N curves in high cycle fatigue or Coffin-Manson curves for low cycle fatigue. Furthermore, the engineering life to initiate an engineering crack could be mostly spent in the growth of small cracks. This paper intends to show a few examples, which have been investigated in our laboratory for various high temperature applications involving superalloys, stainless steels or CMMs. The examples provided include small crack initiation from PM superalloys, and small crack growth under large scale yielding in stainless steels and CMMs. A damage model based on the propagation of microcracks originating at casting defects has been developed. The model gives satisfactory life predictions on various single crystal superalloys for isothermal LCF tests with or without dwell, for variable temperature TMF tests with dwell period, as well as for thermal transient tests on a wedge-shaped structure.

10:50 AM

Fatigue Crack Growth from Small to Long Cracks in VHCF with Surface and Internal "Fish-Eye" Failures for Ferrito-Perlitic Low Carbon Steel SAE 8620: *Israel Marines Garcia*[1]; Paul C. Paris[1]; Hiroshi Tada[1]; Claude Bathias[2]; [1]Washington University in St Louis; [2]CNAM-ITMA

In recent years, the importance of knowing the materials behavior under Very High Cycle Fatigue (VHCF), has been point out by many research laboratories around the world. An important failure phenomenon has been seen between 10^6 and 10^8 cycles, where, the failure initiation switched location from the specimen surface to an interior "fish-eye". As used before, the Paris-Hertzberg-McClintock crack growth rate law, will demonstrate that crack growth is not of significant portion of total life if over 10^7 cycles for a low carbon steel SAE 8620, with ferrito-perlitic microstructure. In this discussion, the presence of closure will be explored for a range of transition sizes from small to long crack as well as various load ratios. Crack initiating at both surface and internal sites will also be considered. Finally, the K at final failure for internal failure will be estimated to see its impact.

11:15 AM

Fatigue Crack Propagation in Cold Drawn Steel: *Jesús Toribio*[1]; Beatriz González[1]; Juan Carlos Matos[1]; [1]University of Salamanca

This paper deals with the influence of the manufacturing process on the fatigue behaviour of pearlitic steels with different degrees of cold drawing. The fatigue crack growth rate (da/dN) is related to the stress intensity range (ΔK) by means a compliance method to evaluate the crack depth a in the samples at any instant during the tests. The analysis is focussed on the region II (Paris) of the fatigue behaviour in which $da/dN=C(\Delta K)m$, measuring the constants (C and m) for the different degrees of drawing. From the engineering point of view, the manufacturing process by cold drawing improves the fatigue behaviour of the steels, since the fatigue crack growth rate decreases as the strain hardening level in the material increases. In particular, the coefficient m (slope of the Paris laws) remains almost constant and independent of the drawing degree, whereas the constant C decreases as the drawing degree rises.

11:40 AM

In-Situ Studies of Local Stresses and Textures in Bulk Nanocrystalline Metals under Large Plastic Deformation Obtained by the Four-Points Bending Device: *Yandong Wang*[1]; G. Fan[2]; H. Q. Li[2]; Y. Ren[3]; H. Choo[2]; P. K. Liaw[2]; L. Zuo[2]; [1]Northeastern University; [2]University of Tennessee; [3]Argonne National Laboratory

Of those various mechanisms to control the plastic deformation in bulk nanocrystalline metals (BNMs), the diffusion-assisted grain-boundary sliding and/or diffusion-assisted grain rotation are the common ones, instead of the dislocation-motion-mediated grain rotation in the coarse-grained materials. However, it is an open question whether the deformation mechanism will change during the large plastic deformation (over a 10% strain) in the BNMs. Stimulated by the recent experimental finding on the appearance of strain-hardening in the gold nanowires over a large deformation [Wu, et. al., Nature Materials 4, 525-529(2005)], we think that the above mechanisms are not unique in BNMs. In this experiment, we used a four-point bending device, instead of the usual tensile frame, for studying the distributions of various stresses and grain orientation as a function of the depth from the bending tensile surface of a nano crystalline Ni-Fe alloy. Thus, at least over a 10% tensile strain was produced on the bottom side of the bended BNM samples. A micro-beam high-energy X-ray was

used to map the lattice strain/orientation distributions with a resolution of 5μm along the compression axial. The type I, II and III stresses, and grain-orientation distribution function (ODF) were determined and modeled for elucidating the detailed deformation mechanisms of BNMs during the large plastic deformation. The present work is supported by the National Science Foundation International Materials Institutes (IMI) Program with Dr. Carmen Huber as the Program Director and the National Natural Science Foundation of China (Grant No. 50471026).

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

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

Wednesday AM Room: 215
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jaraslav Polak, Institute of Physics of Metals; Yasuo Ochi, University of Electro-Communic

8:30 AM Invited

Microstructurally Induced Fatigue Variability and Life Prediction of Turbine Engine Materials: *Sushant K. Jha*[1]; Michael J. Caton[2]; James M. Larsen[2]; Reji John[2]; Andrew Henry Rosenberger[2]; [1]Universal Technology Corporation; [2]U.S. Air Force Research Laboratory

A more physically based life prediction coupled with a real-time damage-state monitoring may be the key to removing some of the uncertainties in the current approach to life management of fracture critical turbine engine components and therefore, increasing their utilization potential. In this paper, we examine the factors contributing to the variability in fatigue lifetimes of turbine disk materials including a+b titanium alloys and nickel-base superalloys. The effect of microstructure, surface residual stress, and the loading variables on the scale and the nature of fatigue variability will be discussed. A worst-case life prediction methodology applicable to the materials in this study will be presented and its physical basis will be discussed. The implications of this approach for the assessment of the role of microstructure and loading variables in the worst-case lifetime distribution and reducing the uncertainty in life prediction will also be addressed.

8:55 AM

Orientation Imaging Microscopy of Fatigue Crack Nucleation and Growth in Waspaloy: *Michael E. Oja*[1]; K. S. Ravi Chandran[1]; Robert G. Tryon[2]; [1]University of Utah; [2]VEXTEC

The effects of grain orientation on fatigue crack nucleation and growth in a polycrystalline nickel-base superalloy, known as Waspaloy, were investigated. Specimens were from a used forged compressor disk from an aircraft engine. Fatigue tests were conducted at 85-95% of the yield stress of the material, at R = 0.1. Multiple nucleations of cracks of the order of a few grain diameters and propagation of the dominant crack were documented. Orientation Imaging Microscopy (OIM) was used to investigate orientations of grains in the cracks' nucleation and adjacent areas. Cracks nucleated in either larger grains or in groups of similarly-oriented grains, collectively called as a "supergrain" due to the closeness of Schmid factors between the grains. The "supergrain" effect was seen in 8 of the 10 cracks investigated. The nature of small crack growth, crack path and the benefits of applying OIM to the study of fatigue cracks are discussed.

9:20 AM

Propagation of Small Surface Crack in Nickel Base Superalloy ME3: *Yong Gao*[1]; Mukul Kumar[1]; Robert Ritchie[1]; [1]University of California

High-cycle fatigue (HCF), involving the premature initiation and/or rapid propagation of cracks to failure due to high-frequency cyclic loading, has been identified as one of the leading causes of turbine engine failures in aircraft. Under HCF conditions, small crack propagation is of great importance for nickel-base superalloys used as turbine engine components. In this paper, details of small surface fatigue crack propagation in the surface stress (75~95% yield stress) of a nickel base superalloy ME3 and the effect of grain-boundary-engineering processing on small fatigue crack behavior have been studied using Optical Microscopy (OM), Scanning Electron Microscopy (SEM), Focused Ion Beam (FIB), and Electron Backscattered Diffraction (EBSD) techniques.

10:10 AM Break

10:25 AM Invited

Simulation-Based Strategies for Microstructure-Sensitive Fatigue Modeling: *David L. Mcdowell*[1]; [1]Georgia Institute of Technology

Efforts to develop microstructure-sensitive fatigue life estimation methods for alloy systems must consider various factors that are not presently addressed by conventional fatigue design tools such as the strain-life curve, the stress-life curve, the modified Goodman diagram, fatigue limit concepts, or by traditional linear elastic fracture mechanics (LEFM) approaches. In this work, relations between remote loading conditions and microstructure-scale plasticity and crack behavior are considered as a function of stress amplitude, stress state and microstructure. Based on multiscale modeling that employs computational micromechanics, influences of microstructure-scale heterogeneities are characterized in terms of cyclic microplastic strain, shakedown, percolation limits for microplasticity through polycrystals and two phase microstructures, fatigue at inclusions (micronotches), and driving forces for microstructurally small cracks. Treatment of grain boundary strength relevant to the distribution of grain boundary type is also explored. Some applications involving materials design and material prognosis are briefly described.

10:50 AM

Microstructure Variability and the Very Long Life Fatigue Behaviors of Rene' 88 DT: *J. Miao*[1]; Tresa M. Pollock[1]; J. W. Jones[1]; [1]University of Michigan, Ann Arbor

Elevated temperature ultrasonic fatigue techniques have been used to examine the fatigue behaviors of the Ni-base superalloy Rene' 88 DT in the lifetime regime of 10^5 to 10^9 cycles at 593°C for a load-ratio of 0.05. The results obtained from ultrasonic fatigue test and conventional fatigue test are comparable and fatigue crack initiation location changes from surface to interior at longer lifetimes/lower stresses. At low loading stress, fatigue cracks are found to initiate from large grains. The correlation between microstructure, fatigue crack initiation behavior and fatigue lifetime has been examined. These observations are used to describe a mechanism for very long life fatigue crack initiation in this alloy.

11:15 AM

Life Prediction of Fretting Fatigue with Advanced Surface Treatments: *Patrick Golden*[1]; Michael Shepard[1]; [1]Air Force Research Laboratory

Fretting fatigue tests were performed on dovetail specimens with and without advanced surface treatments. Laser shock processing and low plasticity burnishing have been shown to produce deep compressive residual stresses with relatively little cold work. Testing showed these advanced surface treatments improved fretting fatigue strength by approximately 50%. In addition to advanced surface treatments, several specimens were also coated with diamond like carbon applied through a non-line-of-sight process capable of coating small dovetail slots in an engine disk. Testing with this coating also significantly improved fretting fatigue strength due to low friction. This work presents a mechanics based lifing analysis of these tests that takes into account the local plasticity and the redistribution of residual stresses due to the contact loading. Local changes in the coefficient of friction due to coatings and wear were also carefully considered. Comparisons were made using different fracture mechanics software tools.

General Abstracts: Light Metals Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Magnesium Committee, TMS: Reactive Metals Committee, TMS: Recycling Committee
Program Organizers: Jim McNeil, Novelis Inc; Neale R. Neelameggham, US Magnesium LLC

Wednesday AM Room: 7D
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Neale R. Neelameggham, US Magnesium LLC; Sivaraman Guruswamy, University of Utah

8:30 AM
Microstructural Evolution in Titanium 5%Mo-5%V-5%Al-3%Cr upon Aging: *Roque Panza-Giosa*[1]; Zhirui Wang[1]; David Embury[1]; [1]McMaster University

This paper presents optical, SEM and TEM characterization of the microstucture of Ti-5553 after solution heat treatment and four aging cycles. The response to ageing was established based on changes in mechanical properties and hardness with ageing time. The four aging times selected represent inflection points in the ageing time/hardness curve. The relationship between microstucture and mechanical properties resulting from these ageing cycles is explored in light of decomposition of Beta phase during ageing.

8:55 AM
Titanium Production through Electrolysis of Consuming TiCxOy Anode: *Hongmin Zhu*[1]; Shuqiang Jiao[1]; Xiaotong Hu[1]; [1]Beijing University of Science and Technology

Titanium oxide can be reduced by carbon forming titanium carbide or oxygen doped titanium carbide (TiCxOy). It was found that the product sintered at temperatures higher than 1573K is electronic conductor like a metal. A series of researches has been performed on the possibility of titanium electrolysis using carbon doped titanium oxide (TiCxOy) as an anode. Carbon monoxide CO was monitored at the anode during the electrolysis when the potential was kept at a certain value. The product on the cathode was investigated by scanning electrode microscopy (SEM) with EDS and X-ray diffraction (XRD) technique. High purity titanium, with the oxygen content of less than 300 ppm, was obtained, and the current coefficient was higher than 90%.

9:20 AM
High Grade Titania from Rutile: *David Freeman*[1]; Graham Sparrow[1]; [1]CSIRO Minerals

One aim of the Light Metal Flagship initiative in CSIRO is to cut the cost of production of titanium metal in half. Several processes being developed for the production of titanium metal use a titania feedstock. It is considered that the removal of impurities during the preparation of the titania, rather than during the processing of the titanium metal, is likely to be a less expensive processing option and so the preparation of a high grade titania material from titaniferous feedstocks may provide a significant economic advantage. Simple processing involving physical separations of ilmenite concentrates, synthetic rutile and rutile concentrates using electrostatic and magnetic techniques gave products containing 97-98% TiO2. A product with 98.5 wt% TiO2 was obtained from commercial rutile concentrates by physical separations, heating with a flux and leaching the heated product. Potential processing conditions are discussed.

9:45 AM
Lithium Peroxide Production: *Javad Khosravi*[1]; Ralph Harris[1]; [1]McGill University

Lithium peroxide can be utilized as final product in space projects as an oxygen source or as an intermediate product for production of pure lithium metal by vacuum reduction. Different procedures for production of lithium peroxide were reviewed and the manufacture of lithium peroxide by leaching with organic solvents and utilizing hydrogen peroxide as a reactant was experimentally studied in an attempt to establish the mechanisms and kinetics of the process. The solubility of lithium hydroxide monohydrate as a precursor material in the organic mediums was measured. The conversion rate of lithium hydroxide monohydrate into lithium peroxide was seen to be high and vary as a function of the test parameters, notably the kind of organic solvent and oxidant. The effects of temperature and time on conversion also were studied.

10:10 AM
A Global View on Aluminium Recycling: *Marlen Bertram*[1]; [1]Organisation of European Aluminium Refiners and Remelters and European Aluminium Association

In 2004, almost 15 million tonnes of recycled aluminium were used globally compared to 30.2 million tonnes of primary aluminium. Aluminium scrap is utilised all over the world, but the solid recycling industry is centred in the traditional recycling areas such as Europe, North America and Japan. Recently fast growing recycling activities have been recognised in China and other North Asian countries. The talk will give an overview of the global aluminium flow based on latest studies of the Global Aluminium Recycling Committee and will show the development of aluminium recycling in the different regions of the world. It will also describe how the aluminium industry is improving the transparency of global scrap flows, analysing week points in the aluminium value chain and forecasting scrap availability. A further subject of the presentation is the importance of aluminium recycling in the context of sustainable development.

General Abstracts: Materials Processing and Manufacturing Division: Powder Processing

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

Wednesday AM Room: 211
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Hyungsik Chung, Ajou University

8:30 AM
Mechanical Alloying and Reactive Processing of Ni3Al Intermetallics: *K. Morsi*[1]; Satyajit Shinde[1]; E. A. Olevsky[1]; [1]San Diego State University

The current paper investigates the effect of mechanical alloying (MA) of mixtures of aluminum and nickel (with different nickel particle sizes) on the dispersion and cold compaction of the composite powders. The concepts of the theory of plasticity of porous bodies are also employed to assess the influence of MA on the yield stress of the composite powder. The influence of processing parameters on the combustion synthesis of optimised compositions is also discussed

8:50 AM
Processing and Properties of Armstrong Process Ti and Ti6Al4V Powders: *Aditya Sathaye*[1]; Adam Benish[2]; Philip Nash[1]; [1]Illinois Institute of Technology; [2]International Titanium Powder

Titanium and Titanium alloys have desirable properties for applications in the transportation and biomedical fields. Reduction in the price of feedstock and processing would widen the market for titanium products. In this paper, we report on the processing and properties of Grade 4 CP Titanium powder and pre-alloyed Ti6Al4V powders synthesized by the Armstrong process developed by ITP. A conventional cold-press and sinter process was selected for consolidation of the powders. Pre-processing of the powder by ball or jet milling was necessary to improve the apparent density. The effect of using blended elemental powders was also investi-

gated. The sintering kinetics for the different powder combinations were determined by dilatometry. The effect of sintering parameters and heat treatment on the mechanical properties will be reported.

9:10 AM
Development of a Cu-W Composite by a Powder Metallurgical Technique: *Debajyoti Maitra*[1]; Ravinder Kumar Dube[2]; Ajit Kumar Roy[1]; [1]University of Nevada; [2]Indian Institute of Technology Kanpur

A powder metallurgical route has been proposed for making tungsten dispersed copper composite strips used as electric contact materials. Composition of 80Cu-20W has been considered in this study. The route consists of high energy milling of a mixture of Cu2O and WO3 powders in the required ratio in a high-energy ball mill which is then co-reduced in hydrogen at a suitable temperature. It is then compacted and sintered at suitable temperature. The strips are then subsequently hot rolled. The resulting grain size of co-reduced powder was found to be ~25 nm. 93% theoretical density of the Cu-20%W composite strip was achieved. The results indicate that this composite was capable of maintaining appreciably high electrical conductivity, strength and microhardness. The overall data indicate that the enhanced metallurgical and physical properties may be attributed to the fine dispersion of W in Cu due to the combined effect of milling and co-reduction.

9:30 AM
Rapid Prototyping of Biofouling Resistant ABS: *Mario H. Castro-Cedeno*[1]; [1]Rochester Institute of Technology

A surface coating of silver nanoparticles added to ABS rapid prototyping material was sucessful in increasing the biofouling resistance of the parts produced. Surfaces treated with silver-ion nanoparticles have been shown to resist the growth of bacteria and mold and are not hazardous to humans. By treating the material used for rapid prototyping with silver-ion particles, we can produce prototype parts or small production runs of parts that can be used in environments where biofouling is a problem.

9:50 AM Break

10:10 AM
Preparation of Ag Powders by Mechanochemical Reaction in AgCl-Cu System: *Jaeryeong Lee*[1]; Ikkyu Lee[1]; Jonggwan Ahn[1]; Dongjin Kim[1]; Jae-Chun Lee[1]; Hun S. Chung[1]; [1]Korea Institute of GeoScience and Mineral Resources

Silver powders have been widely used as for conductive paints, inks and paste to making films due to good thermal/electrical conductivity, anti-oxidation and catalysis. In accordance with these purposes, its specific morphology is strongly desired. Especially, plate-shaped Ag powders are widely used as fillers for isotropic conductive adhesives. For this reason, many processes have been researched to prepare the Ag powder. Among them, mechanical grinding is the most common and popular process for the production of Ag powders with a shape of plate, although there are two problems due to the intensive grinding; one is the impurity caused by wearing of pot and balls, and the other is the defect of crystal structure. In the present work, a noble technique was proposed. Plate-shaped Ag powders were prepared by mechanochemical reaction in AgCl-Cu system, and the effect of additives was investigated on morphological and structural properties of the Ag powders.

10:30 AM
Effects of Alloying Elements on the Sintered Properties of Mixed Elemental Aluminum Powder Alloys: *Hyungsik Chung*[1]; Jae-Hwan Ahn[1]; Moon-Tae Kim[1]; [1]Ajou University

The effects of Cu, Mg and Zn on the sintering of elemental aluminum powder mixtures were studied. In binary aluminum mixtures, copper was most effective for raising sintered density and properties. Higher strength could be obtained with Al-6 to 10% Cu than those of commercial powder blends. A small amount of magnesium was also effective for improving the sintering to some extent. Too much magnesium over 1%, however, resulted in coarse pores after sintering due to the transient nature of the liquid phase formed, and lowered the overall sintered properties. The Al-Zn also formed a transient liquid phase during sintering but yielded much lower sintered properties than Al-Cu. The addition of Mg or Zn to Al-Cu altered the sintered structure significantly and produced some improvements of the sintered properties. Zn addition, however, was much more effective for raising the sintered properties than Mg.

10:50 AM
Measurement Method for Cell Gap and Twist Angle of Reflective LCD: *Zou-Ni Wan*[1]; Chia-Fu Chang[1]; Si-Wen Wan[1]; [1]Kun Shan University of Technology

A polarization insensitive, electrically tunable Gooch-Tarry's first minimum condition is demonstrated. When the light passes through the TN medium an even number of times. Furthermore, some properties of this method are discussed and an analysis by theoretical calculation is undertaken. We proposed a cell gap measurement method to determine the cell thickness by simulation values of the transmitted light for arbitrary wavelength regions.

11:10 AM
Morphological Characterization of Diamond Particles Synthesized at High Pressure and High Temperature: *Ana Lucia Diegues Skury*[1]; Sergio Neves Monteiro[1]; [1]UENF

The performance of synthetic diamond used in wear resistant tools or abrasive pastes depends on many factors such as the mechanical strength, cristalinity, size and morphology of the particles. Diamond powders synthezised in different solvent/catalyst systems at high pressure and high temperature conditions, contains crystals that could be separated into groups of distinct sizes and defect morphology. These groups can be differing by their mechanical strength. In general the strength of diamond particle is affected by its surface condition, shape, defect concentration and other factors. In this work results on the morphological characterization of diamond particles, obtained at 4.3 GPa and 1200C from a mixture of graphite and NiMn alloy, are presented.

General Abstracts: Structural Materials Division: Microstructure and Properties of Materials I

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

Wednesday AM — Room: 218
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Donna Ballard, U.S. Air Force

8:30 AM
Cu-SiC_p Composites with Interpenetrating Network Structure for Thermal Management and Electrical Contact Applications: *Aditya Putrevu*[1]; V. V. Bhanu Prasad[2]; Veeredhi Vasudeva Rao[3]; [1]University of Texas; [2]Defense Metallurgical Research Laboratory; [3]Sreenidhi Institute of Science and Technology

Copper matrix composites reinforced with varying content of particulate SiC (0-30% by volume) were processed by vacuum hot pressing and press-sinter routes. No reaction products were detected in any of the composites. Density and porosity of the composites reduced considerably with increasing SiC content and there is an evolution of an interpenetrating network structure with increasing SiC content. Significant strengthening of the composites took place with increasing reinforcement content with an increase in hardness, strength and elastic modulus. Coefficient of thermal expansion of the composites reduced with SiC content. Inspite of lower thermal and electrical conductivity of SiC compared to copper, the conductivity of the composites was considerable. Superior mechanical

properties, lower density, cheaper SiC reinforcement without foregoing much of the conductivity makes Cu-SiC_p composites potential materials for thermal management and electrical contact applications.

8:55 AM
Numerical Modeling of Stress Concentration on Clustered Porosity: *Yi Cheung Lok*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology

The effect of stress concentration on a single spherical cavity is wellunderstood. This study models numerically the stress concentration on a cluster of cavities and compared with that on a single cavity. The effect of the number of cavities, their relative location and the distance between cavities on stress concentration will be discussed. The result will be used to validate the applicability of Saint Venant's principle and can possibly act as a guideline for evaluating clustered porosity.

9:20 AM
On the Sensitivity and Uniqueness Issues in Determining the Elasto-Plastic Properties of Materials through Indentation: A Comparative Analysis: *Hongzhi Lan*[1]; T. A. Venkatesh[1]; [1]Tulane University

Instrumented indentation as a technique for extracting the fundamental mechanical properties of materials has recently received considerable attention. Issues concerning the uniqueness and robustness of the extracted properties to variations in experimentally measured quantities have been recognized as being important. In the present study: (i) A uniform framework for assessing the uniqueness and sensitivity of the forward and reverse analyzes, for all the principal methods that have been developed so far, will be identified. (ii) Within a broad range of homogeneous, isotropic, power-law hardening materials, domains where the indentation method is applicable for an unambiguous identification of elasto-plastic properties will be distinguished. (iii) The differences in the nature of the sensitivity of the indentation analyzes, as a function of the nature of the indentation method, will be characterized. (iv) Guidelines for selecting appropriate methods for accurate and robust determination of elasto-plastic properties will be provided.

9:45 AM
In-Situ Internal Strain Measurement in Polytetrafluoroethylene by Neutron Diffraction: *Eric N. Brown*[1]; Philip J. Rae[1]; Dana M. Dattelbaum[1]; George T. Gray[1]; Donald W. Brown[1]; [1]Los Alamos National Laboratory

Measurements of internal strain development during in-situ deformation of polytetrafluoroethylene (PTFE) have been performed utilizing neutron diffraction measurements with the SMARTS diffractometer at Los Alamos National Laboratory. While PTFE is known to be semi-crystalline in nature, the effect of mechanical deformation on this structure has received limited attention. The chemical structure of PTFE, (C2F4)n, makes it ideally suited for investigation by neutron methods as it is free of the H that results in limited penetration depths and poor diffraction acquisition in most polymers. Internal strains have been measured in tension and compression for well pedigreed PTFE 7C with accurate temperature control to ensure the phase IV crystalline structure. Six primary diffraction peaks have been identified. For compression the (210) and (300) helical lattice orientations exhibit the largest strains (~0.5% internal strain at 60% bulk true strain) and closely parallel the bulk imposed stress.

10:10 AM Break

10:25 AM
Shock and Recovery of Polytetrafluoroethylene through the Phase II-III Transition: *Eric N. Brown*[1]; Philip J. Rae[1]; Carl P. Trujillo[1]; Neil K. Bourne[2]; George T. Gray[1]; [1]Los Alamos National Laboratory; [2]University of Manchester

Polytetrafluoroethylene (PTFE) is semi-crystalline in nature with its linear chains forming complicated temperature and pressure dependent phases. Experimental studies on pressure-induced phase transitions using shock-loading techniques and the resulting changes in crystalline structure are presented. Disks of pedigreed PTFE 7C have been shock loaded in momentum trapped assemblies using a 80 mm gas launcher, and recovered in a density graded polymer network. Experiments were performed with impact pressures from 0.4 to 0.85 GPa to investigate the material response above and below the phase II to phase III crystalline transition. Recovered samples were planar with residual strains of less than 5%. Changes in crystalline structure of the recovered materials were quantified using dynamic scanning calorimetry (DSC) and density, both indicating decreased crystallinity with increased impact pressure.

10:50 AM
Characterization of Grain Boundary Liquation in Inconel 718 Subjected to GTAW: *Shenavia Wilkerson Howell*[1]; Viola L. Acoff[1]; [1]University of Alabama

Inconel 718 is a high-strength precipitation hardenable nickel-based superalloy that is used for many high temperature applications. During welding of this alloy, the area immediately adjacent to the fusion zone is subjected to partial melting along the grain boundaries. This phenomenon is termed liquation. Extensive studies have shown that constitutional liquation of grain boundary precipitates results in intergranular microfissuring in the weld heat-affected zone. In the present study, bead-on-plate welds were made on specimens using gas tungsten arc welding (GTAW). The resulting welds were characterized using electron backscattered diffraction patterns and transmission electron microscopy. The liquation of grain boundaries is a heterogeneous process. This means that some grain boundaries liquate while others do not. Therefore, the purpose of this study is to investigate the crystallographic orientations of grain boundaries in the liquated region, and the conditions under which liquation occurs.

11:15 AM
Annealing Effects on the Grain Growth of ZR-702: Brian James Sarrail[1]; Shake Babakhanyan[1]; Charles Schrupp[1]; Richard Clark[1]; Omar Es-Said[1]; *John Ogren*[1]; [1]Loyola Marymount University

Zirconium is a hard, shiny, grayish white metal. With its superior corrosion resistance capabilities, it has increasingly become the material of choice in the fabrication of chemical processing equipment and nuclear reactors. This project determined the intermediate annealing step that will avoid the growth of very large crystallites in the metal during the final material annealing step. In addition, the effect of cold work on grain growth was analyzed. The amount of cold work was varied from 0% to 15%, and a T-5 radius bend test also used. These samples were then annealed in air and the microstructure was examined. No significant grain growth was found to occur.

11:40 AM
Influences of Different Copper Coatings on Microstructure of C/Cu/Al Composites: *Zhuokun Cao*[1]; Guangchun Yao[1]; [1]Northeastern University of China

Different copper coatings whose thickness varies between 0.5μm to 2μm were deposited on carbon fibers using both electroless and electroplating method. The fibers with copper coating were chopped and fabricated composites with melting aluminum. Influences of different copper coatings on distribution and interface bonding condition of carbon fibers in aluminum matrix were studied by scanning electron microscope (SEM). The role of the copper layer on the microstructure in the system was also discussed. The results indicate that 0.7μm thick copper coating deposited using electroplating technique performs the best effects in the matrix and that copper layer reacts with aluminum matrix, results to the formation of intermetallic compound CuAl2 which can protect the fibers at a high temperature and help to improve the mechanical properties of the composites.

12:05 PM
The Effect of Thermal Exposure on the Mechanical Properties of 2099-T6 Die Forgings, 2099-T83 Extrusions, 7075-T7651 Plate, 7085-T7452 Die Forgings, 7085-T7651 Plate, and 2397-T87 Plate Aluminum Alloys: Joseph Jabra[1]; J. Lai[1]; E. Lee[1]; M. Setiawan[1]; *Eui W. Lee*[2]; Jeffrey J. Witters[3]; Nabil Abourialy[2]; Michael Romios[1]; John R. Ogren[1]; Omar S. Es-Said[1]; [1]Loyola Marymount University; [2]Naval Air Systems Command; [3]Alcoa Aerospace Applications Engineering

Aluminum alloys 2099-T6 die forgings, 2099-T83 extrusions, 7075-T7651 plate, 7085-T7452 die forgings, 7085-T7651 plate, and 2397-T87 plate were thermally exposed at temperatures of 180°C (350°F), 230°C (450°F), and 290°C (550°F) for 0.1, 0.5, 2, 10, 100, and 1000 hours. The purpose of this study was to determine the effect of thermal exposure on the mechanical properties and electrical conductivity of these alloys. The data show that higher temperatures and longer exposure times generally

resulted in decreased strength and hardness and increased percent elongation and electrical conductivity.

Hume Rothery Symposium: Multi-Component Alloy Thermodynamics: Alloy Design and Properties

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

Wednesday AM Room: 202A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Fan Zhang, CompuTherm LLC

8:30 AM Invited
Phase Diagram and Design of Cobalt-Base Alloy Systems: *Kiyohito Ishida*[1]; [1]Tohoku University

Cobalt-base alloys are widely used in various applications such as magnetic materials, corrosion and heat-resistant alloys, wear-resistant alloys, prosthetic alloys in medical parts etc., where the phase diagrams play a key role in material development. This paper presents recent progress on the phase diagrams and design for Co-base alloys, focusing on magnetic recording media, ferromagnetic Co-Ni-Al-base shape memory alloys and Co-base superalloys. The calculated miscibility gap between ferromagnetic and paramagnetic states in the Co-X(X: Cr, Mo, W) systems was found to agree well with the experimental results, strongly implying that the magnetic induced phase separation is responsible for the compositional heterogeneity in Co-base magnetic recording media. New ferromagnetic Co-Ni-Al shape memory alloys were also developed based on phase diagrams. The introduction of γ(Al structure) to the β(B2) matrix was found to drastically improve the ductility. Finally, the possibility of potent strengthening of Co-base superalloys upon precipitation of γ'(L12) is presented.

9:00 AM Invited
Thermodynamic Modelling of Multi-Component Oxide Systems: *Ling Zhang*[1]; Shouyi Sun[1]; Sharif Jahanshahi[1]; [1]CSIRO Minerals

The cell model originally proposed by Kapoor and Frohberg for simple silicate melts was later extended to multi-component liquid slags by Gaye and Welfringer. CSIRO has extended the application of this model to describing the behaviour of numerous oxide species commonly found in metallurgical slags. The developed database includes the following species; SiO2, Al2O3, Cr2O3, TiO2, Ti2O3, Fe2O3, FeO, CaO, MgO, MnO, CrO, PbO, NiO, CoO, ZnO, Na2O and Cu2O in the liquid slag and oxide solid solutions. Recent enhancements to the model will be discussed with particular focus on the behaviour of Al2O3 containing ternary and higher order systems. As a structurally based model, the cell model calculates parameters related to the degree of polymerisation in silicate melts. These parameters have been used in a model for calculating the viscosity of silicate melts. Examples on viscosity of silicate slags with alumina and other oxides will also be presented.

9:30 AM Invited
Application of Thermodynamic Modeling Tool to Commercial Titanium Alloys: *Fan Zhang*[1]; Shuanglin Chen[1]; [1]CompuTherm LLC

Materials are developed and improved by adjusting both the alloy chemistry and the processing conditions to achieve desired microstructures and properties. Traditionally, these improvements have been made by a slow and labor-intensive series of experiments. Today, thermodynamic modeling has become an essential tool in understanding the effect of alloy chemistry on the final microstructure of a material. Implementation of such a tool to improve material processing via parameter optimization has resulted in significant cost savings through the elimination of shop/laboratory trials and tests. In this study, a thermodynamic modeling tool developed at CompuTherm is being utilized to predict properties of commercial titanium alloys, such as beta transus, phase proportions, phase chemistries and so on. This tool includes Pandat, software for multi-component phase equilibrium calculations, and PanTitanium, a thermodynamic database for titanium alloys. Model predictions are compared to experimental results for number of alpha-beta alloys.

10:00 AM Break

10:20 AM
Determination of Thermodynamic Properties and Calculation of Viscosity and Phase Diagrams of Various Ternary Lead-Free Solder Materials: Adolf Mikula[1]; *Sabine Knott*[1]; Zuoan Li[1]; Peter Terzieff[1]; [1]University of Vienna

The target of our thermodynamic investigations was to determine a complete set of partial and integral quantities of ternary systems as a function of temperature and molar weight. Surface tension and viscosity directly affects the solder fluidity characteristic and the ability to wet a substrate surface. The thermodynamic measurements and calculations were done in the framework of the European COST 531 action. Little information about the thermodynamic properties and viscosity data of the ternary Ag-Bi-Sn, Ag-Cu-Sn and Bi-In-Sn systems are known. We carried out thermodynamic measurements of these systems in the liquid state using two different methods: calorimetric and an EMF method with a liquid electrolyte. The integral Gibbs free energy of the system at 1000 K was calculated by Gibbs-Duhem integration. The results of the thermodynamic investigations were used do calculate the viscosity and the phase diagrams. The results of these measurements and the calculations will be presented.

10:45 AM
Experimental Studies and Scheil Simulations of Microstructure in Nb-Ti-Hf-Si Alloys: *Bernard P. Bewlay*[1]; Ying Yang[2]; Laurent Cretegny[1]; Y. Austin Chang[3]; [1]General Electric; [2]CompuTherm LLC; [3]University of Wisconsin

Nb-silicide based in-situ composites are promising materials for future high-temperature structural applications. Nb-silicide composites are typically alloyed with Hf, Ti, Cr and Al to provide a balance of mechanical and environmental properties. Solidification processing can play a critical role in the control of the final microstructure of these composites. In this study, thermodynamic modeling of the Nb-Ti-Hf-Si system was performed using the CALPHAD approach; in conjunction with this approach, Scheil and global-equilibrium simulations of solidification paths were then preformed based on the optimized thermodynamic description for the Nb-Hf-Ti-Si system. These simulations can serve as a guide in the selection of alloy compositions for microstructure optimization, and a range of examples will be described. Experimental observations for selected directionally solidified alloys will be compared with simulation results. These experimental results are also used in the validation of the thermodynamic descriptions.

11:10 AM
Multicomponent Alloy Thermal Physical Property Prediction Coupled Computational Thermodynamics with Back Diffusion Consideration: *Jianzheng Guo*[1]; Mark T. Samonds[1]; [1]ESI U.S. R&D

Simulation technologies are applied extensively in casting industries to understand the aspects of heat transfer and fluid transport phenomena and their relationships to the microstructure and the formation of defects. It is critical to have accurate thermo-physical properties as input for reliable simulations of the complex solidification and solid phase transformation processes. The thermo-physical properties can be calculated with the help of thermodynamic calculations of phase stability at given temperatures and compositions. A comprehensive multicomponent alloy solidification model, coupled with a Gibbs free energy minimization engine and thermodynamic databases, has been developed. The thermal physical properties can be calculated. A back-diffusion model is integrated so that the solidification conditions, such as cooling rate, can be taken into account.

Lead Free Solder Implementation: Reliability, Alloy Development, and New Technology: Electromigration

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

Wednesday AM Room: 214A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Sinn-Wen Chen, National Tsing Hua University; Kwang-Lung Lin, National Cheng Kung University

8:30 AM
Effect of UBM Consumption on Failure Mechanism in Flip Chip Solder Joints during Current Stressing: *Yen-Liang Lin*[1]; C. Robert Kao[1]; Yi-Shao Lai[1]; [1]National Central University

The electromigration failure mechanism in flip chip solder joints through the rapid depletion of UBM was investigated. The solder used was 63Sn37Pb and the joints had a nominal diameter of 125 um. The UBM on the chip had a Cu/Ni(V)/Al metallization, and the surface finish on the substrate side was Au/Ni. Our results showed that Ni UBM adhered well after 300 hours at 150°C when no electric current was applied. However, under current stressing, a rapid consumption of Ni UBM occurred and the joints failed due to UBM exhaustion and de-adhesion. In other words, electric current accelerated the Ni UBM consumption, and eventually resulted in the failure of the solder joints. In this presentation, the effect of UBM consumption on flip chip solder joint failure will be discussed.

8:50 AM
Effect of Electromigration on Mechanical Property of Lead-Free Solder Joints Studied by Nano-Indentation Continuous Stiffness Measurement: *Fei Ren*[1]; King-Ning Tu[1]; Luhua Xu[2]; John H. L. Pang[2]; Zhong Chen[2]; [1]University of California, Los Angeles; [2]Nanyang Technological University

Tensile test structures of a solder ball connected by two Cu wires of 300 to 500μm diameter were prepared and tested. The ultimate tensile strength decreases with longer time or higher current density of electromigration. To study the effect of electromigration on mechanical property of lead-free SnAgCu solder joints, a nano-indentation continuous stiffness measurement (CSM) was used. In electromigration at 100ºC, the applied current density was from 1.5×103 A/cm2 to 1×104 A/cm2 and the time from 3 to 144 hrs. An array of 50 nm indentations was created by the nano-indenter from the cathode area, across the bulk of the solder, to the anode area. The change of Young's modulus and hardness from the cathode to the anode was analyzed from the CSM. Combined with the microstructure study of intermetallic compound formation at the interfaces, the effect of electromigration on mechanical property of the solder joint will be discussed.

9:10 AM
Electromigration Effect on Intermetallic Growth and Young's Modulus in SAC/ENIG Solder Joint: *Luhua Xu*[1]; John H. L. Pang[1]; Fei Ren[2]; K. N. Tu[2]; [1]Nanyang Technological University; [2]University of California, Los Angeles

The impact of electromigration on growth of interfacial IMC in ENIG/SAC/ENIG single ball solder joint was studied. When solder joint was subjected to a current density of 5000A/cm2 at 125C, IMC layer growth on anode interface is faster than cathode interface, and both are faster than isothermal aging. Young's modulus and hardness were measured by nanoindentation CSM (continuous stiffness measurement) on these interfacial IMCs after 86 hrs EM test. Different value was observed on anode and cathode sides. Young's modulus for the IMC at anode side is around 180-190 GPa, while the value at cathode side is only 130-140 GPa. EDS Line Scan was conducted at interface from Ni(P) substrate to SAC solder, showing that the copper concentration at anode IMC is about 25-35%, while less than 10% at cathode side IMC. This indicates composition and crystal structure of IMC at anode and cathode is affected by EM.

9:30 AM
Electrochemical Migration of the Sn-9Zn-0~4Ag Reflowed on Cu/Ni/Au Substrate: *Jing Chie Lin*[1]; Yi-Fang Hong[1]; Sheng-Long Lee[1]; [1]National Central University

The electrochemical migration of the lead-free solders Sn-9Zn-0~4Ag reflowed on Cu/Ni/Au substrate was investigated. A term tcm is defined as the duration from null to a sudden rise of current to evaluate the reliability of the system. Reliability of the solders Sn-9Zn-0~4Ag decreases with increasing the silver content from 0 to 4 wt%. Failure is caused by a connection of the dendrites that extend from cathode to anode. Chemical analysis of the dendrites by XPS depicts that the concentration ratio of Zn/Sn decreases with migration time. Investigation of anodic potentiodynamic polarization benefits the delineation of the electrochemical migration. The solders contain higher silver content shifts their open circuit potential (OCP) to noble. Anodic current is greater for the solder containing higher silver concentration at any potential higher than OCP. Galvanic corrosion induced by Ag and AgZn3 leads to this failure.

9:50 AM
Electromigration in Cu Column Flip Chip Joints: *Jae-Woong Nah*[1]; Jong-Ook Suh[1]; King-Ning Tu[1]; Vempati Srinivasa Rao[2]; Seung Wook Yoon[2]; Vaidyanathan Kripesh[2]; [1]University of California, Los Angeles; [2]Institute of Microelectronics

When size of solder bumps decreases for fine pitch,stand-off height between chips and substrates also decreases. The low stand-off height is of concern in mechanical fatigue life. Also, as size of solder bumps decreases, current density through the joint increases and electromigration reliability becomes an issue. Therefore, new packaging concept is required for flip chip with fine pitch, high stand-off height, and high current density capability simultaneously. In this study, we introduce a new flip chip technology by using Cu columns which has 100-um pitch and 60-um stand-off heights. We investigate electromigration in solder part of the Cu column flip chip joints. When we compare electromigration resistance between conventional solder flip chip joints and Cu column flip chip joints, we found that the latter has better electromigration resistance than the former. The reason will be explained. The effect of Cu columns on current distribution has been confirmed by simulation.

10:10 AM Break

10:25 AM
Electromigration Study Using Kelvin Bump Structure: *Yuan-Wei Chang*[1]; Shih-Wei Liang[1]; Chih Chen[1]; [1]National Chiao Tung University

Electromigration of flip-chip solder joints has been studied extensively in recent years. Voids formed at the solder in the vicinity of the entrance point of the Al trance for solder joints with thin film UBM. However, the nucleation and propagation of the voids is still not clear. For Al and Cu interconnects, void nucleation and propagation during electromigration is monitored by resistance change. But the bump resistance is quite small compared with the resistance of the metallization traces. Therefore, daisy-chain structure cannot detect the slight changes in microstructure in the solder joint. In this study, we designed and fabricated Kelvin bump probe, and used it to monitor the bump resistance change during electromigration successfully. Three-dimensional (3D) finite element modeling was also performed to simulate the bump resistance increase due to void formation. This approach facilitates the systemic study of failure mechanism due to electromigration in flip-chip solder joints.

10:45 AM
Electromigration Study of Pd-SnAgCu Solder-Pd Structure Using V-Groove Lines: *Annie T. Huang*[1]; King-Ning Tu[1]; [1]University of California

The use of Pb-free solders is gradually replacing the Pb-containing solders due to environmental concern. However, high temperature Pb-free solder with melting point around 300°C has not yet been developed. Because the trend of increasing I/O density in interconnect technology, size of flip chip solder joints has to be reduced. It is anticipated that interme-

tallic compound (IMC) will gradually replace the bulk part or the entire bump of a solder joint in the near future as the solder joint size decreased. The element Pd was reported to have ultra-fast IMC formation with Sn-containing solders. Fast IMC formation between Pd and Sn may function as high temperature solder joint due to the higher melting point of IMC. In this talk, we report the fast formation of IMC between Pd electrode and SnAgCu solder, and electromigration behavior across Pd-SnAgCu solder-Pd lines in v-grooves on Si wafers.

11:05 AM

Mechanism of Void Formation in Flip-Chip Solder Joints: *Shih-Wei Liang*[1]; Yuan-Wei Chang[1]; Tung-Liang Shao[1]; Chih Chen[1]; [1]National Chiao Tung University

Redistribution of current density and temperature due to void formation in flip-chip solder joints during electromigration was investigated using three-dimensional thermo-electrical coupled modeling, in which the current density and temperature redistribution were simulated at different stages of void growth for solder joints with thin-film UBM. After the void formation near the entrance point of Al trace, the current may drift through the UBM layer to the periphery of the solder joint, leading to the void formation in the periphery of the solder joint, which was low current density region before the void formation. This proposed mechanism successfully explained our experimental results. In addition, the temperature change due to the void formation will be presented in this meeting.

11:25 AM

Prevention of EM-Induced Cu Dissolution by an Effective Ni Barrier Layer: *Yu-Hsiang Hsiao*[1]; Cheng-Yi Liu[1]; [1]National Central University

EM(Electromigration)-induced dissolution of Cu UBM in eutectic SnPb has been reported, which is a crucial issue for the flip-chip solder bump. By next few years, Sn-rich Pb-free solders would replace SnPb solder in C4 bumps. Those promising Sn-rich Pb-free solders are known to have higher Cu solubility than that of SnPb solder. Therefore, much serious EM-induced Cu dissolution would be expected for Sn-rich Pb-free solders. In this study, a Ni barrier layer was deposited on the Cu metal bond pad to retard EM-induced Cu dissolution. Our preliminary results show that the Ni thin layer, indeed, can retard EM-induced Cu dissolution. In this talk, we will also report EM behaviors of three different Pb-free solders, ie.e, Sn, Sn0.7Cu, Sn3.0Cu on Ni/Cu substrates.

11:45 AM

The Combined Effects of Electromigration Flux and Cross-Interaction Flux in SnAg Flip-Chip Solder Joints: *Chiaming Tsai*[1]; Yi-Shao Lai[2]; C. R. Kao[1]; [1]National Central University; [2]Advanced Semiconductor Engineering, Inc.

In a solder joint with the Cu layer on one side and the Ni layer on the other, there is a net Cu flux from the Cu side to the Ni side, which is known as the cross-interaction flux. When an current passes through a solder joint, the current could produce the EM(electromigration) flux. In this study, the combined effects of EM flux and cross-interaction flux in flip-chip solder joints was studied. The UBM had a Ti/Cu/Ni triple-layer structure, and the surface finish on the board side was SOP Cu. When the direction of electron flow was from board side to chip side, Cu cross-interaction flux and Cu EM flux were in the same direction, but when the direction of electron flow was from chip to board, these two fluxes were in the opposite direction. Effects generated from such configuration were reported.

12:05 PM

Heat Effect and Impact Resistance during Electromigration on Cu-Sn Interconnections: *Tae-Kyu Lee*[1]; J. W. Morris, Jr.[1]; Fay Hua[2]; [1]University of California, Berkeley; [2]Intel Corporation

The influence of electron current on the diffusion of Sn and Cu in simply designed Cu-Sn-Cu diffusion couples was investigated. The diffusion couples were designed to permit in situ studies of the progress of diffusion. Initial tests were done in various temperature in air with a current density over 1x10^4A/cm^2. The results showed Cu movement into Sn in the direction of the electron current with accompanying grain boundary sliding of the Sn grains. A continuous intermetallic compound formation was observed at the anode side, simultaniously with an intermetallic dissipation at the cathode side. Temperature measurements at the interconnection using thermo-couples and IR(Infra Red) camera showed a continuous temperature increase during current stressing. A series of results on impact resistivity of the Cu-Sn interconnections during current stressing were also achieved by using a modified Micro-Charpy impact testing method.

Magnesium Technology 2006: Thermodynamics and Fundamental Research

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

Wednesday AM — Room: 6B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Zi-Kui Liu, Pennsylvania State University; Rainer Schmid-Fetzer, Clausthal University of Technology

8:30 AM

Finite-Temperature Thermodynamic Properties of Intermetallics in the Mg-Ca-Sn System via First-Principles Methods: *Raymundo Arroyave*[1]; Munekazu Ohno[2]; Zi-Kui Liu[1]; Rainer Schmid-Fetzer[2]; [1]Pennsylvania State University; [2]Clausthal University of Technology

In recent years, interest in the use of Magnesium alloys in automotive applications has increased significantly due to economic, political and environmental factors. Accordingly, a considerable effort has been put into the development of a complete thermodynamic database for Mg alloys, based on the CALPHAD approach. This methodology requires the existence of experimental data to determine the values of the parameters in the models describing each phase. In this work, first-principles methods are used to calculate the total free energies of most intermetallic phases in the Mg-Ca-Sn system so they can be eventually used in CALPHAD models of this ternary system. Vibrational and electronic degrees of freedom are taken into account, as well as the contributions due to lattice thermal expansion. To illustrate how one can use the finite-temperature, first-principles thermodynamic properties in CALPHAD modeling, the Mg-Ca binary system is re-modeled. The results are then compared with previous CALPHAD models.

8:50 AM

Investigation of the Liquidus Surface in the Mg-Rich Corner of the Mg-In-Ce Ternary System: *Elvi C. Dalgard*[1]; Dmytro Kevorkov[1]; Mihriban O. Pekguleryuz[1]; [1]McGill University

The liquidus surface in the ternary Mg–In-Ce system was studied in the composition range 1 to 3% In and 0.1 to 1.5% Ce. Thermal analysis was used to determine the liquidus points in order to construct polythermal sections of the ternary phase diagram. Metallography and x-ray techniques were used to examine phases present at the compositions studied. A triple point presumed to represent a peritectic was discovered at 580°C. In addition, FACTSage was used to confirm thermodynamic agreement of the new data with existing literature on the system.

9:10 AM

Linking Meso- and Macroscale Simulations: Crystal Plasticity of HCP Metals and Plastic Potentials: *Dirk Steglich*[1]; [1]GKSS Research Center

Due to its hexagonal crystallographic structure, magnesium alloys show a strong tension-compression asymmetry, which have to be accounted for by advanced material models. The untypical mechanical behaviour of magnesium can be explained and reproduced with the help of a viscoplastic model for crystal plasticity. The study of single crystal specimens subjected to channel die compression tests reveals the active slip and twinning systems. In order to describe the mesoscopic behaviour of a polycrystal, texture is incorporated into polycrystalline RVEs and mechanical properties of extruded bars and rolled plates can be predicted. In order to extend the modelling potential, a phenomenological yield surface accounting for anisotropy and tension/compression asymmetry has been developed and implemented in a finite element code and compared with crystal plasticity calculations. This allows for a direct comparison of simulation

strategies on different length scales and thus for a calibration of model parameters.

9:30 AM

Study of Phase Equilibria in Magnesium-Cerium Binary System via the Diffusion Couple Technique: *Xin Zhang*[1]; Dmytro Kevorkov[1]; Mihriban Pekguleryuz[1]; [1]McGill University

A liquid-solid diffusion couple method was implemented to investigate the magnesium-cerium binary system. Pure Mg and Ce contact was vacuum-encapsulated in quartz tube and the Mg and Ce inter-diffused at 400°C. All the four single-phase zones corresponding to the Mg-Ce phase diagram were observed. The solid solubility ranges of the phases at magnesium-rich side were investigated by means of SEM line-scan. The phase identification was carried out by electron microprobe analysis (EMPA), X-ray diffraction and SEM. In order to investigate the composition range of the intermetallic compounds and clarify data in existing phase diagrams, alloys containing target phases were prepared, cast and annealed at a temperature range of 400-550°C. The room-temperature and elevated-temperature (quenched) microstructures of these alloys were observed and the lattice parameters of the intermetallics were determined via XRD.

9:50 AM

Study of Thermal Evolution of the Mg Solid Solution in the Mg-Al-Li-Zn System: *Dmytro Kevorkov*[1]; Carl Fuerst[2]; Mihriban Pekguleryuz[1]; [1]McGill University; [2]General Motors R&D

The modification of commercial AZ alloys with Li requires precise knowledge concerning solubility ranges in the Mg-Al-Li-Zn solid solution. The study of solubility range variations with temperature (thermal evolution) is extremely important for the prediction of solidification behavior in multicomponent alloys. The Mg solid solution in the Mg-Al-Li-Zn system has a complex shape and has never been studied in detail. The two, three and four phase samples were annealed at the temperatures of 250, 300, 350 and 400°C, and quenched in liquid nitrogen. The crystal structures of intermetallic phases and the maximum solubilities of elements in Mg were determined by powder x-ray diffraction (Rietveld analysis). Alloy microstructures were studied with SEM and the compositions of the intermetallic phases were determined by quantitative EDX. The shapes of the Mg-Al-Li-Zn solid solution were constructed at various temperatures and their thermal evolution was analyzed.

10:30 AM Break

10:10 AM

The Effect of Solute Elements on the Lattice Parameters of Binary Solid Solutions of Magnesium: *Ana Maria Becerra*[1]; Dmytro Kevorkov[1]; Carlton D. Fuerst[2]; Mihriban O. Pekguleryuz[1]; [1]McGill University; [2]General Motors of Canada

The influence of solute elements on the lattice parameters of magnesium has been studied. Binary solid solutions of Mg-Ce, Mg-In, Mg-Zn, and Mg-Li alloys were cast in copper moulds and homogenized at temperatures between 375°C and 400°C. Filings were then taken under protective atmosphere, encapsulated under argon and then annealed at 275°C to remove residual stress. Lattice parameter measurements were carried out with a Philips PW 1710 powder diffractometer with Cu-a radiation. The experimental XRD results were refined and analyzed using the Rietveld method. Alloys were also characterized using optical and scanning electron microscopy. The effects of solutes on lattice parameters were explained on the basis of atom size differences, and the change in electron overlap of magnesium due solute additions.

10:50 AM

Thermodynamic Modeling of the Mg-Sn-Zn-Al System and Its Application to Mg Alloy Design: *In-Ho Jung*[1]; Woo Jin Park[1]; Sangho Ahn[1]; Daehoon Kang[2]; Nack J. Kim[2]; [1]Research Institute of Industrial Science and Technology; [2]POSTECH

Recently Mg-Sn-Zn-Al alloy system has been investigated actively in order to develop new magnesium alloy which satisfies a stable structure and good mechanical properties at high temperatures. In the present study, available thermodynamic and phase diagram data of the Mg-Sn-Zn-Al quaternary system have been critically evaluated and all reliable data have been simultaneously optimized to obtain one set of model parameter for the Gibbs energy of the liquid and all solid phases as functions of composition and temperature. The optimized database was applied to new alloy design. All calculations were performed using FactSage thermochemical software.

11:10 AM

Dissolution Kinetics and Recovery of Manganese Additions into Liquid Magnesium: Zhi Li[1]; *Stavros A. Argyropoulos*[1]; Timothy J. Kosto[2]; [1]University of Toronto; [2]Milward Alloys Inc

This paper will focus primarily on the dissolution of various Manganese compacts in liquid magnesium. A unique experimental procedure has been developed, using cylindrical compacts with various Manganese compositions. The various temperatures of the addition and magnesium bath, as well as the apparent weight of the addition were measured simultaneously during the immersion of the addition into the magnesium bath. The exothermicity level of these additions during their dissolution in liquid magnesium was determined. The relation of the exothermicity level with the dissolution kinetics was also examined. In addition, SEM and EDX work was conducted for additions, which were withdrawn from the Magnesium bath. Finally, the recovery of these additions was estimated by taking samples from the Magnesium bath and conducting ICP analyses.

11:30 AM

Two-Stage Thermodynamic Modeling of a Thixoforming Process: *Rainer Schmid-Fetzer*[1]; Mile B. Djurdjevic[1]; [1]Clausthal University of Technology

A two-stage thermodynamic calculation is proposed as a tool to analyze the alloy related aspects in semisolid forming processes. The two process stages are identified as (i) equilibration in semisolid state, and (ii) subsequent fast solidification of the material. For the example of Mg-Al-Zn-Ca alloys the results and limitations of this modeling approach are given. The wealth of information provided, such as process temperature and sensitivity, freezing ranges and the detailed constitution of the processed material, is suggested to assist in alloy development. This work is supported by the German Research Foundation (DFG) in the Priority Programme “DFG-SPP 1168: InnoMagTec”.

11:50 AM

Tensile Properties and Microstructures of the Mg-13Li-9Al-1Zn Alloy Prepared in Air by Electrolytic Diffusing Method: *Meng-Chang Lin*[1]; Jun-Yen Uan[1]; [1]National Chung Hsing University

The Mg-Li-Al-Zn alloy was successfully produced in air by electrolytic diffusing method at 500°C. In electrolysis cell, Mg-9Al-1Zn (AZ91) alloy plate with 3mm in thickness was used as cathode and graphite was as anode. The mixtures of 55wt%KCl - 45wt%LiCl were employed as the electrolyte. The microstructure was investigated by optical microscopy, scanning electron microscopy, X-ray diffraction and transmission electron microscopy. After electrolysis experiments, the Mg-13Li-9Al-1Zn alloy plate can be obtained, the hexagonal-closed-pack AZ91 plate (3mm in thickness) was converted to the mixture of a-Mg and BCC Mg phase (thickness changed to 4.6mm). The Mg-13Li-9Al-1Zn alloy plate could be cold rolled easily into 1.5mm strip. After heat treatment, the Mg-13Li-9Al-1Zn alloy strip exhibits yield strength ~ 148MPa. The elongation can reach ~ 22%, which is much better than AZ91D alloy.

Magnesium Technology 2006: Wrought Alloys and Forming Processes 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

Wednesday AM Room: 6A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Karl U. Kainer, GKSS Research Center; Ravi Verma, General Motors Corporation

8:30 AM
Development of CaO Added Wrought Mg Alloy for Cleaner Production: *Jin-Kyu Lee*[1]; Hyung-Ho Jo[1]; Shae K. Kim[1]; [1]Korea Institute of Industrial Technology

Magnesium alloys are attractive for structural components in transportation industries. At present, magnesium components for structural applications are mainly produced by the die casting process. It is, however, commonly acknowledged that structural components made from wrought alloys through a metal forming process have great advantage in terms of obtainable strength and toughness. In addition, most of the wrought magnesium alloys are heat treatable and thus allow their strengths to be further improved or their mechanical performance to be tailored by adjusting the parameters of heat treatment. The wrought process route of melting, ingot casting/continuous casting, forming and heat treatment is carried out under protective cover gas to prevent oxidation and ignition. In order to eliminate cover gas during forming process, the aim of present study is to investigate the effect of CaO additions on surface oxidation of extrudate without protective cover gas and properties of CaO added AZ31 alloys.

8:55 AM
Development of Thixoextrusion Process for AZ31 Magnesium Wrought Alloy: *Young-Ok Yoon*[1]; Hoon Cho[1]; Hyung-Ho Jo[1]; Shae K. Kim[1]; [1]Korea Institute of Industrial Technology

Magnesium and magnesium alloys are used in a wide variety of structural applications. Recent developments in the field of magnesium wrought alloys are opening up diverse industrial applications, especially, in the field of extrusion process. Various extrusion processes such as rapid cooling extrusion, isothermal velocity extrusion, alternative section extrusion and thixoextrusion, have been developed for improving extrudability of AZ31 magnesium wrought alloy. Especially, thixoextrusion process has advantages of high productivity, reduced extrusion pressure and homogeneous internal microstructure compared with conventional extrusion processes. The aim of this study is to investigate feasibility of thixoextrusion for AZ31 magnesium wrought alloy through simple partial remelting. Microstructural evolution on globular solid particle size and distribution with respect to isothermal holding temperature and time will be discussed.

9:20 AM
Drawing Formability of Magnesium Alloy (AZ31) Sheet with Conical Cup Test: Hee Taek Lim[1]; Jeong Hun Kang[1]; Seong Ho Cho[1]; *Jeong Whan Han*[1]; [1]Inha University

The drawing forming has considerable potential because of its competitive productivity and performance. Though die-casting process has been widely used in magnesium forming, unavoidable poor surface qualities are major obstacles for applying to electronic housing components. In the present study, a conical cup test was made to evaluate drawing formability of magnesium alloy (AZ31) sheet. Sheets fabricated by extrusion and rolling were examined and effects of punch speed and temperature were investigated as major process parameter. Drawing Formability was evaluated by using CCV (conical cup value) designated in the standard, which is the arithmetical mean of maximum and minimum diameter. In order to investigate microstructure, the examined specimens were sectioned in the middle vertically and observed microstructure locally. It was found that the CCV increase with the increase of punch speed. This means high punch speed has poor drawing formability. The CCV reaches to a minimum value at 573K.

9:45 AM
Effect of Extrusion Temperature on Formability and Tensile Properties of AZ31B Extrusions: *Weinan Ding*[1]; Jiang Li[1]; Dongchang Jiang[1]; Changbao Ren[1]; [1]SinoMaG-GRM

This study investigated the extrusion formability and mechanical properties of AZ31B extrusions at various extrusion temperatures from 2000C to 4500C. A vertical direct chill casting equipment was set up in GRM for semi-continue casting ϕ110 mm AZ31B billets and a horizontal 630-ton extrusion press was used to extrude AZ31B extrusions.Extrusion die, holding tube and F95x350 billets were all heated to specified temperature before extrusion. Microstructure and tensile properties of ϕ25 mm round bars extruded with different extrusion temperatures were investigated. Extrusion experiments indicated that the lowest extrusion formable temperatures are variable with different section profiles and extrusion ratios. Extrusion surface tearing defects become worse with extrusion temperature increasing. The proper extrusion temperature range is optimized by the consideration of metal formability and extrusion surface quality. The strength and ductility of extrusions were obviously improved in respect with billet material. Extrusion temperatures significantly influence metal recrystallization performance and tensile properties.

10:10 AM Break

10:30 AM
Effect of Strain Rate on Deformation Behavior of a Mg-9Li-2Zn Alloy Sheet at Room Temperature: Hong Bin Li[1]; Guang-Chun Yao[1]; *Yi Han Liu*[1]; [1]Northeastern University

A two-phase Mg-9Li-2Zn alloy sheet is made by cold-rolling at room temperature and the formability of it at room temperature is investigated in this study. Uniaxial tension tests are carried out for various strain rates between 0.5mm/min and 250mm/min, and the microstructural changes during the tests are observed. The sheet has high formability at comparatively low strain rates. Maximum elongation amounts to 40%. However, ductility decreases with the increase in strain rate. Even at room temperature, the stress is also sensitive to the strain rate. There are many large dimples at comparatively low strain rates, and small dimples occur at high strain rates, it shows fine sub-grains come into being.

10:55 AM
High-Speed Heavy Rolling of Magnesium Alloy Sheets: *Hiroshi Utsunomiya*[1]; Tetsuo Sakai[1]; Satoshi Minamiguchi[1]; Hiroaki Koh[1]; [1]Osaka University

As Mg alloys are well known difficult-to-work alloys, they are mostly processed by die-casting or thixo-moulding. The lower deformability is due hcp crystal structure and inactiveness of basal slip at low temperature (<500K). Sheets are produced by rolling. Rolls are often heated and multi-pass low reduction rolling with intermediate annealing are frequently employed to suppress edge cracks or fracture. The authors propose a high-speed heavy-reduction rolling for manufacturing of Mg alloy sheets. Commercial AZ31B sheets were processed at high-speed (~2000m/min) between RT and 623K. It is found that the thickness can be reduced 60% by one-pass operation even at room temperature without fracture. The sheets processed at 473K showed the finest recrystallized microstructure with a mean grain size of 2.2 microns. The fine-grained sheets show superior mechanical properties than conventional sheets. It has been concluded that the high-speed heavy rolling is promising for industrial manufacturing of Mg sheets.

11:20 AM
Hot Rolling of AM50 for Sheet Production: Jürgen Göken[1]; *Norbert Hort*[2]; Kurt Steinhoff[2]; Karl Ulrich Kainer[2]; [1]University of Kassel; [2]GKSS Research Center

In the past magnesium cast alloys have been successfully developed and are fairly good introduced to vehicles even in power train applications with service temperatures up to 150°C and more. But there is still a lack of information's regarding the use of magnesium alloys as wrought materials especially in sheet applications. This concerns both the availability of alloys and the processing parameters of a chosen wrought process. In the present work AM50 has been used to produce sheet by hot

rolling. Preheated material has been deformed in several steps until the sheet reached a final thickness of 0.3 mm. Further deformation was applied on the sheets by deep drawing to obtain information's of the materials behavior after a secondary deformation process. The paper describes the rolling and deep drawing experiments and the microstructure obtained in the different steps of processing with a special regard to recrystallization behavior.

Materials Design Approaches and Experiences II: Steels and Titanium 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

Wednesday AM Room: 202B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Timothy P. Gabb, NASA Glenn Research Center; Yunzhi Wang, Ohio State University

8:30 AM Invited
Computational Systems Design of Hierarchically Structured Materials: *Gregory B. Olson*[1]; [1]Northwestern University and QuesTek Innovations LLC

A systems approach integrates processing/structure/property/performance relations in the conceptual design of multilevel-structured materials. Using examples of high performance alloys, numerical implementation of materials science principles provides a hierarchy of computational models defining subsystem design parameters which are integrated via computational thermodynamics in the comprehensive design of materials as interactive systems. Recent initiatives integrate materials science with quantum physics and applied mechanics, and address the acceleration of the full materials design, development and qualification cycle. Principal barriers to the adoption of the new paradigm of science-based materials engineering are primarily cultural, and will require substantial reform of undergraduate materials education.

9:00 AM Invited
The Application of Computational Thermodynamics to Materials Design: *John M. Vitek*[1]; Ron L. Klueh[1]; [1]Oak Ridge National Laboratory

Computational thermodynamics provides the basis for identifying phase stability in multi-component systems, such as ferrous and nickel-based alloys, by considering the free energy of phases as a function of composition and temperature. This tool can be used effectively to model the influence of composition and heat treatment on the phases that form during solidification, thermal processing and service exposure. Consequently, it can lead to improved alloy design by tailoring alloy compositions to meet specific needs. This presentation will describe several examples in which calculations provided guidance in terms of identifying alloy modifications or heat treatment schedules to optimize material performance. This research was sponsored by the Laboratory Technology Research Program of the Office of Science, U. S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:30 AM
Carbon Diffusion and Phase Transformations during Gas Carburizing of High Alloyed Stainless Steels: Experimental Study and Theoretical Modeling: *Thierry Turpin*[1]; Jacky Dulcy[2]; Michel Gantois[2]; [1]Aubert and Duval; [2]Ecole des Mines de Nancy

Gas carburizing of high alloyed stainless steels increases surface hardness, as well as the overall mechanical characteristics of the surface. The growth of chromium rich carbides during carbon transfer into the steel causes precipitation hardening in the surface, but decreases the chromium content in solid solution. In order to maintain a good corrosion resistance in the carburized layer, the stainless steel composition and the carburizing process need to be optimised. To limit the experimental work a methodology using software for modeling the thermodynamic and kinetic properties in order to simulate carbon diffusion and phase transformations during gas carburizing is presented. Experimental carbon profiles were determined by WDS-EPMA analyses, while carbide compositions were measured by EDS_X analyses. A good agreement between calculated and experimental values was observed for the Fe-13Cr-5Co-3Ni-2Mo-0.07C and the Fe-12Cr-2Ni-2Mo-0.12C (wt pct) martensitic stainless steels at 955°C and 980°C.

10:00 AM Break

10:20 AM Invited
Use of Modeling Tools for Forging and Heat Treat Process Design: *David U. Furrer*[1]; Gangshu Shen[1]; Vikas Saraf[1]; [1]Ladish Company Inc

Modeling and simulation has been advancing at a rapid rate. The use of deformation and thermal simulations, along with microstructure evolution modeling tools is now advancing to the point where virtual process design is possible from component configuration to final component microstructure and properties. Combined simulation tools are being used for a variety of materials, including steel, titanium and nickel-base superalloy forgings. The future will continue to demand more rapid process design and increased component capabilities, which will require further advancements in the developmentand use of computer-based simulation tools.

10:50 AM Invited
Design Tools for Predicting Microstructure/Property Relationships in Ti Alloys: *Hamish L. Fraser*[1]; [1]Ohio State University

There has been a significant effort aimed at integrating computational methods with critical experiment to produce design tools for structural materials. This paper describes the work that has been focused on the development of such tools for prediction of the interrelationship between microstructure and properties for Ti alloys. This has been done by a number of researchers within our group who will be gratefully acknowledged during the presentation. The methods for quantification of microstructure will be detailed, including new ways in which 3-D representations of microstructure may be determined. These data together with measurements of mechanical properties are used to populate databases that become the basis for the development of neural network models capable of predicting the desired interrelationships. The ways in which these neural networks are used to perform virtual experiments, leading to increased mechanistic understanding, and to develop design tools which operate on PC computers will be demonstrated.

11:20 AM
Phase Field Modeling of Microstructural Evolution in Ti-6Al-4V: *Ning Ma*[1]; Yunzhi Wang[1]; [1]Ohio State University

Phase transformation and microstructural evolution in commercial titanium alloys are extremely complex. In this presentation we discuss our recent effort in integrating thermodynamic modeling and phase field simulation to develop computational tools for quantitative prediction of phase equilibrium and spatiotemporal evolution of microstructures during thermal processing. The models account explicitly for precipitate morphology, spatial arrangement and anisotropy. The models developed were validated against experimental observations using optical metallography, SEM, and TEM, with microstructural features quantified by a set of rigorous procedures based on stereology. The rendering of the predictive capabilities of the phase field models as fast-acting design tools through the development of constitutive equations is also demonstrated.

11:40 AM
Neural Networks for Mechanical Property Predictions – Example of Fracture Toughness in Ti Alloys: *Sujoy Kar*[1]; Thomas Searles[2]; Jaimie Tiley[3]; Brian Welk[1]; Joshua Tuggle[1]; Santhosh Koduri[1]; Gopal Babu Viswanathan[1]; Hamish L. Fraser[1]; [1]Ohio State University; [2]Rolls-Royce Corporation; [3]Air Force Materials Laboratory

The accelerated insertion of titanium alloys in component application requires the development of predictive capabilities for property-microstructure relationships. This paper discusses the development of Neural Network (NN) Models based on Bayesian statistics to predict the fracture toughness at room temperature. The microstructural data are obtained by stereological procedures. These data along with the measurements of frac-

ture toughness constitute the database. This database is used to train and test the NN to predict the fracture toughness. In addition, the trained NN models are successfully used to identify the functional dependence of fracture toughness on individual microstructural features via virtual experiments, which in turn can assist in the development of physically based models. Comparative fractography analysis is performed to understand the fracture behavior of extreme fracture toughness samples. Development of web based design tool for online prediction of fracture toughness using NN model is demonstrated.

Materials in Clean Power Systems: Applications, Corrosion, and Protection: Interconnection and Sealing in Fuel Cells II

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

Wednesday AM Room: 212B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Jeffrey W. Fergus, Auburn University; Anthony Petric, McMaster University

8:30 AM Invited
Spinel Coatings for Solid Oxide Fuel Cell Interconnects: *Anthony Petric*[1]; Ping Wei[1]; M. Reza Bateni[1]; [1]McMaster University

Chromia scale-forming alloys have adequate corrosion protection as SOFC interconnects but the electrical resistance becomes a problem with time. In addition, volatile chromia species can poison the cathode reaction. One possible solution is to apply a compatible coating to the interconnect surface. We surveyed the electrical and thermal properties of spinel compounds. Spinels based on Al2O3 and Cr2O3 exhibit low values of conductivity and thermal expansion. CuFe2O4, CuMn2O4 and Co2MnO4 were found to have the most compatible properties for interconnect coatings. Our measurements showed that conductivities range from 10 to 100 S/cm at 800°C and thermal expansion coefficients are 10-12 ppm/K. Spinel coatings up to 50 um thick were grown by electroplating the metals individually onto UNS430 ferritic stainless steel, followed by oxidation at 750°C for 1 day. The coatings were dense and showed good adhesion to the substrate. Testing of the performance of these coatings at fuel cell conditions is underway.

9:00 AM
Development of (Mn,Co)3O4 Protection Layers on Ferritic Stainless Steels for SOFC Interconnect Applications: *Zhenguo Gary Yang*[1]; Gordon Xia[1]; Gary Maupin[1]; Shari Li[1]; Prabhakar Singh[1]; Jeff Stevenson[1]; [1]Pacific Northwest National Laboratory

Due to their excellent oxidation resistance and thermal expansion match to the other stack components, stainless steels are among the most promising candidate materials for interconnect applications in intermediate-temperature planar SOFC stacks. For this particular application, however, there remain some issues in their long-term surface stability and the electrical resistance arising from the oxide scale growth, as well as chromia evaporation and potential poisoning of cells. Application of (Mn,Co)3O4 spinel protection layers on the ferritic stainless steels appears to be a promising solution to overcome these issues. To optimize the spinel protection layers for maximum performance, the spinel materials were systematically studied; different fabrication approaches and parameters have also been investigated. This paper will present details of this work and give an overview on the materials and different approaches of fabricating the spinel protection layers on ferritic stainless steel SOFC interconnects.

9:25 AM
Dual Atmosphere Tolerance of Ag-CuO Based Air Braze: *Jin Yong Kim*[1]; John S. Hardy[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

Silver-based reactive air braze (RAB) is an effective alternative for hermetic sealing of high termperature devices such as solid oxid fuel cells (SOFCs). Since the seal in the SOFCs (especially the cell-to-frame seal) is exposed to the dual atmosphere such as hydrogen on one side and oxygen on the other side, the dual atmosphere tolerance is one of the critical requirements for this application. Exposure to the dual atmosphere can initiate degradation such as hydgrogen embrittlement after long term operation. We will discuss the microstructure after dual atmosphere exposure and apparent mechanisms of the degradation.

9:50 AM
Enabling Inexpensive Metallic Alloys as SOFC Interconnects: An Investigation into Hybrid Coating Technologies to Deposit Nanocomposite Functional Coatings on Ferritic Stainless Steels: *Paul Gannon*[1]; Max C. Deibert[1]; Richard Smith[1]; Asghar Kayani[1]; Z. Gary Yang[2]; Jeffery Stevenson[2]; Steven Visco[3]; Craig Jacobson[3]; H. Kurokawa[3]; Stephen Sophie[4]; [1]Montana State University; [2]Pacific Northwest National Laboratory; [3]Lawrence Berkeley National Laboratory; [4]NASA-Glenn Research Center

Reduced operating temperatures (600-800°C) of Solid Oxide Fuel Cells (SOFCs) may enable the use of inexpensive ferritic steel interconnects. Due to the demanding SOFC operating environment, protective coatings are gaining attention to increase long-term interconnect stability. In this study, large area filtered arc deposition (LAFAD) and hybrid filtered arc-assisted EBPVD (FA-EBPVD) technologies were used for deposition of two-segment coatings with Ti-Cr-Co-Al-Y-O-N based bottom sublayer and Mn-Co-O top layer. Coatings were deposited on ferritic steel and subsequently annealed in air for various time intervals. Surface oxidation was investigated using RBS and SEM/EDS analyses. Cr-volatilization was evaluated using a transpiration apparatus and ICP-MS analysis of the resultant condensate. Area Specific Resistance was studied as a function of time using the four-point technique. The oxidation behavior, Cr volatilization rate, and electrical conductivity of the coated and uncoated samples are reported. Transport mechanisms for various oxidizing species and coating diffusion barrier properties are discussed.

10:15 AM Break

10:30 AM Invited
Evaluation of Fe-Ni Alloys for Reduced-Temperature SOFC Interconnect Application: *Jiahong Zhu*[1]; S. J. Geng[1]; M. P. Brady[2]; I. G. Wright[2]; X. D. Zhou[3]; H. U. Anderson[3]; [1]Tennessee Technological University; [2]Oak Ridge National Laboratory; [3]University of Missouri-Rolla

Current standard solid oxide fuel cell (SOFC) interconnect materials, ferritic Fe-Cr alloys, have the Cr volatility problem, which leads to the poisoning of the cathode and subsequent degradation in SOFC performance over long-term stack operation. A new generation of Fe-Ni base alloys which are either free of Cr or have low Cr content is under development to mitigate the Cr volatility problem. This talk will summarize our recent alloy design efforts on Cr-free or low-Cr Fe-Ni alloys with different Ni levels as interconnect materials for reduced-temperature SOFC. Evaluation of these alloys includes their coefficient of thermal expansion (CTE), oxidation resistance, oxide scale area specific resistance (ASR), and interaction/compatibility with cathode materials. The promises of the Fe-Ni alloys as SOFC interconnect will be highlighted and alloy design strategies to address the remaining issues related to these alloys will be discussed.

11:00 AM
Investigation of Clad Materials for Use in PEMFC Bipolar Plates: *K. Scott Weil*[1]; Z. Gary Yang[1]; Gordon Xia[1]; Jin Yong Kim[1]; [1]Pacific Northwest National Laboratory

We are currently investigating clad materials for potential use as bipolar plates in PEMFCs. In our present approach the thin metal laminate sheet consists of a middle filler layer sandwiched between two transition metal alloy layers that can be thermally nitrided or carbided to form a conductive, yet corrosion-resistant (i.e. passivating) barrier layer. Ideally the material selected for the filler, which will form the thickest of the three layers, is chosen based primarily on material cost, formability, durability, and thermal conductivity. The cladding material, on the other hand, is selected based on the ease of nitriding or carbiding, the corrosion resistance and electrical transport properties of the resulting nitride or carbide,

formability, and cost. In this way, the bipolar plate can be tailored to take advantage of the merits of each material, while minimizing material and processing costs.

11:25 AM
Nitrided Stainless Steels for PEM Fuel Cell Bipolar Plates: *Bing Yang*[1]; Michael P. Brady[1]; David Young[2]; Peter Tortorelli[1]; Heli Wang[3]; John Turner[3]; [1]Oak Ridge National Laboratory; [2]University of New South Wales; [3]National Renewable Energy Laboratory

This paper will overview efforts to form electrically conductive and corrosion resistant Cr-nitride surfaces on commercial and developmental stainless steel alloys for use as proton exchange membrane fuel cell (PEMFC) bipolar plates. Emphasis will be placed on the details of the nitridation reaction for both austenitic and ferritic alloy substrates, and the possible role of oxygen impurities in the nitriding environment in aiding establishment of the external nitride layer. Results of interfacial contact resistance measurements and polarization screenings in simulated PEMFC environments will also be presented.

Materials Processing Fundamentals: Powders and Composites

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

Wednesday AM — Room: 203A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Princewill N. Anyalebechi, Grand Valley State University

8:30 AM
Effects of Temperature and Precursors on Preparation of Fe-TiC Composite from Ilmenite: *Sutham Niyomwas*[1]; [1]Prince of Songkla University

The Fe-TiC composite powder was synthesized in situ by carbothermal reduction of ilmenite. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of the reacting species. The effects of The reaction temperature (1400°C - 1600°C) and milling time of precursors on the Fe-TiC conversion and powder morphologies were investigated using XRD and SEM method. The synthesized products showed that composite with iron matrix and titanium carbide as a reinforced phase was formed.

8:55 AM
Production of Titanium Powder Directly from Titanium Ore by Preform Reduction Process (PRP): *Haiyan Zheng*[1]; Toru H. Okabe[1]; [1]Univeristy of Tokyo

The preform reduction process (PRP) based on the calciothermic reduction of titanium ore was investigated. To develop a new process for producing metallic titanium powder directly from titanium ore, thermodynamic analyses for removing iron from the ore by selective chlorination were conducted before the experimental work. Titanium feed preform was fabricated at room temperature by casting slurry, which is a mixture of titanium ore (rutile TiO2 with impurities, e.g., iron), flux (CaCl2), and binder. The fabricated preform was calcined at elevated temperatures and iron was removed by selective chlorination. The obtained preform was then reduced using metallic calcium vapor as the reductant, and the reduced preform was subject to leaching to remove CaO, Ca, and other impurities. When de-ironized rutile ore mixed with CaCl2 flux was reduced, metallic titanium powder with 99% purity was obtained. Thus, the PRP is feasible to produce titanium powder directly from titanium ore.

9:20 AM
EMF Studies on Nb-Al Intermetallics: *Pratheesh George*[1]; Ramana G. Reddy[1]; [1]University of Alabama

Niobium-Aluminum alloys are of great interest due to its high temperature properties. Nb is also used as an alloying element with Ti-Al alloys to improve properties like oxidation resistance. In this investigation, Gibbs energy of mixing of $NbAl_3$ was determined using EMF method in the temperature range of 933 to 1098 K. The construction and design of the cell used for EMF measurement was also discussed. The cell reaction is as follows: Pt /Al, Ca_2 AlF $_7$ $\|CaF_2\|$Al (Nb-Al alloy), Ca_2AlF_7/Pt. Experimental results showed that a linear relationship between EMF and temperature was obtained. The partial Gibbs energy of formation was calculated using the Nernst equation. The thermodynamic properties like activity, activity coefficient and partial Gibbs energy of mixing was determined in the temperature range of 933 to 1098 K for $NbAl_3$. These experimentally determined thermodynamic properties of Nb-Al alloys were compared with other aluminides.

9:45 AM
In Situ Synthesis of Al-TiC-Al2O3 and Al-SiC-Al2O3 Composites: *Napisporn Memongkol*[1]; Sutham Niyomwas[1]; [1]Prince of Songkla University

Syntheses of Al-TiC-Al2O3 and Al-SiC-Al2O3 composites were obtained in situ by combustion assisted synthesis (CAS) of mixtures of Al powder, TiO2 powder and activated carbon powders and Al powder, SiO2 powder and activated carbon powders. The reactions were carried out in a horizontal tube furnace under flow of inert gas. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of the reacting species. The synthesized products were mixtures of aluminum matrix reinforced with titanium carbide and alumina and aluminum matrix reinforced with silicon carbide and alumina. The effect of the quantity of the matrix powder (Al powder) was investigated using XRD and SEM method. The synthesized products showed those composites with aluminum matrix and titanium carbide and alumina as reinforced phases and aluminum matrix and silicon carbide and alumina as reinforced phase were formed.

10:10 AM Break

10:25 AM
Ti Product from the Reaction of TiCl4 Gas with Droplets of Molten Mg: Akio Fuwa[1]; *Masashi Tsumura*[1]; [1]Waseda University

Presently, sponge titanium metal product is produced through Kroll process where TiCl4 reactant gas is reacted and reduced with molten Mg reactant in a batch reactor. This reduction reaction may be limited taking place at these reactants interfacial area, so that higher reduction rate may be limited as well. This research investigates the reduction reaction of TiCl4 with molten Mg metal droplets, and titanium product situation and morphology. Molten Mg droplets have larger interfacial area so that the higher reaction rate can be expected. Here, molten Mg droplets are continuously formed by flowing Mg through a nozzle with small hole at a heated, holding crucible, introduced in a reactor and reacted with TiCl4 gas, fed continuously into the reactor. The reduction reactions are studied with the following experimental parameters; temperature, droplet size, Mg/TiCl4 feed ratio, and etc.

10:50 AM
Titanium Subchloride Synthesis by Reaction of Titanium with TiCl4: *Osamu Takeda*[1]; Toru H. Okabe[1]; [1]University of Tokyo

In order to establish a new semi-continuous/high-speed titanium production process based on the magnesiothermic reduction of titanium subchlorides, a novel synthetic process of titanium subchlorides (TiClx, x = 2, 3) by reacting titanium metal with TiCl4 was investigated. Titanium metal powder placed on a molybdenum tray was heated to 1273 K in a stainless steel reactor filled with argon gas, and TiCl4 liquid was supplied into the reactor by a pump at a rate of 0.12~0.64 g/min. Deposits recovered from the trays after the experiment were identified to be TiCl2. Under certain conditions, a trace of TiCl3 was also observed in the TiCl2 deposit. Currently, a more efficient method for titanium subchloride production using a molten salt medium is under investigation. Some results on the reduction of synthesized TiCl2 by magnesium will also be shown.

11:15 AM
Study of Decomposition Regularities for a Zn-Containing Volatile Complex Used in ZnO Film Synthesis: Emma Rubenovna Arakelova[1]; *Fridrikh Akopovich Grigoryan*[1]; Vergine Gagikovna Parvanyan[1]; Goarik Gagikovna Asatryan[1]; [1]Statye Engineering University of Armenia

H2O2 (98%) vapor passed through a reactor at 25 Pa where a ZnO target was placed at 273K-373K. Experiments were carried out on a flow-type vacuum device. H2O2 decomposition on ZnO surface accompanied by formation of a Zn-containing volatile complex which transferred to the gas phase, and ZnO deposited on a substrate at condensation. As the substrate, five sequentially located quartz cylinders were used at 293K-423K (TП). Decomposition of the Zn-containing complex on quartz substrates carried out as a first- order reaction. The complex decomposition constant(Kp) increased with the substrate temperature (TП = 293 K, Kp = 7.05 sec^{-1}; TП = 373 K, Kp = 87 sec^{-1}; TП = 423 K, Kp = 158 sec^{-1}). The complex decomposition constant did not practically vary when the target temperature increased; the complex formation rate increased slightly.

11:40 AM

Preparation of Mono-Dispersed Co3O4 Particles by Homogeneous Precipitation with Urea Followed by Decomposition: *Guo Xueyi*[1]; [1]Central South University

The mono-dispersed Co3O4 precursor particles were prepared by homogeneous precipitation from the high Co2+ concentration with urea, the main factors were investigated on the homogeneous precipitation, which include the Co2+ concentration, the initial pH of the solution, the temperature and the reacted time for the reaction, the use of the urea, the agitation pattern, It is determined that the precursor particles were cobalt hydroxyl-carbonate with fine crystalline. Then, the precursor was decomposed at high temperature and the mono-dispersed Co3O4 particles with 2um in average particle size and special micrograph, fine crystalline were obtained. The effects of the temperature, the time for decomposition were addressed.

12:05 PM

The Formation of Misty Band near the Substrate during Contact Angle Measurement: Wu Linli[1]; Yao Guangchun[1]; *Tianjiao Luo*[1]; Zhang Xiaoming[1]; [1]Northeastern University

During measuring contact angle there was a misty band near the substrate. Hsien-Nan Ho and Shinn-Tyan Wu thought this was because the oxygen atoms on the interface migrate to the droplet surface to form an oxide band near the substrate. But from our experiments misty band appeared before the drop reach to the substrate. Theoretically speaking, the oxygen partial was the same everywhere in the vacuum furnace. So there was no condition to provide enough oxygen forming oxide. This appearance would be explained more reasonably with the optics principle. Most reflect light would be absorbed by the substrate and light cannot reach the observation area, so the misty band formed.

Multicomponent-Multiphase Diffusion Symposium in Honor of Mysore A. Dayananda: Industrial Applications

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

Wednesday AM
March 15, 2006

Room: 203B
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Sungjin Koh, University of Texas at Arlington; John E. Morrall, Ohio State University

8:30 AM

Carbon Diffusion in Steels – An Analysis Based on Direct Integration of the Flux: *Olga Karabelchtchikova*[1]; Richard D. Sisson[1]; [1]Worcester Polytechnic Institute

In the early 1970s Professor Dayananda developed a technique of direct integration of fluxes over distance from the concentration profiles in vapor-solid diffusion couples. This integration allowed determination of concentration dependent atomic mobilities and diffusion coefficients. As part of a project to control and optimize the industrial carburization process in mild and low-alloyed steels, this analysis was applied to several commercial grade carburized steels to determine the effect of alloy composition on their atomic mobility and carbon diffusion in the Austenitic phase. While carbon flux and therefore surface carbon content vary with time during single-stage carburizing even with a fixed carbon potential in the atmosphere, a mass balance at the gas-solid interface must serve as boundary condition. Furthermore, the analysis was applied to determine the characteristic surface mass transfer coefficient and carbon mobilities in several grades of steel. The results were compared with previous determinations and predictions reported in literature.

8:55 AM

Carbon Diffusion in Austenite: A Reassessment: *Irina V. Belova*[1]; Graeme Murch[1]; [1]University of Newcastle

We re-analyse carbon tracer and chemical diffusion in austenite at low carbon compositions using a very general four-frequency model first formulated by McKee. Our re-analysis makes use of the almost exact formalism of Okamura and Allnatt. In the re-analysis of the carbon diffusion and making use of the carbon activity data we confirm the general finding of McKee that the increase of both tracer and chemical diffusivities with carbon composition at 1000 C is largely a result of a much higher rate of rotation of carbon interstitial pairs compared with isolated carbon interstitials. We find however that the carbon atoms move almost twice as fast as a rotating pair than found originally by McKee. We also extrapolate information on the ratios of exchange frequencies to temperatures between 800 C and 1000 C using available experimental data on the carbon chemical diffusion and activity.

9:20 AM

Comparative Studies of Boron Diffusion in Low Carbon Steel, High Strength Alloy Steel and Stainless Steel: *Naruemon Suwattananont*[1]; Roumiana Petrova[1]; Boris Goldenberg[1]; [1]New Jersey Institute of Technology

Comparative studies of boron diffusion in low carbon steel (AISI 1018), high strength alloy steel (AISI 4340) and stainless steel (AISI 304) are reported. The steel samples were boronized by using powder mixture method in temperature range of 600-1000°C for 2, 4, 6, 8 and 10 hours, respectively. Boron diffusion, morphology and types of borides were analyzed by optical microscopy, XRD, SEM and EDS. The growth kinetics of MeB, Me_2B phases was investigated as a function of treatment time and temperature as well as the effect of alloying element concentration on the kinetics process. Diffusion coefficient and activation energy were calculated from the distribution profiles of alloying elements and the thickness of the boride layers. The experimental results show the diffusion coefficient and activation energy of boronizing process are dependent on the concentration of the alloying elements in steel as well as treatment time and temperature.

9:45 AM

Diffusion Phenomena Related to Carburization Resistant Ablation Coatings for Ethylene Pyrolysis Coils: *Alok P.S. Chauhan*[1]; Henry J. White[1]; Weidong Si[2]; [1]State University of New York; [2]Brookhaven National Laboratory

Wholly austenitic heat resistant materials have been the workhorse for the outlet pass of ethylene pyrolysis tubing for many years. Higher operating temperatures (>1100 C) and the presence of both oxidizing (on the outer diameter side) and carburizing (process side) environments have reduce tube life significantly (by 4-6 years). We will present the diffusion related phenomena (carburization and oxidation related events in the material at 1100 C; conversion of the inherent Cr_2O_3 surface protective coating to $Cr_{23}C_6$ and Cr_7C_3; paramagnetic to ferromagnetic transformation; etc.) associated with our efforts to reduce catalytic coke formation (i.e. increase tube life) by coating the inner diameter of ethylene pyrolysis tubing using pulsed deposition carburization resistant coatings. Diffusion controls the structure, properties, processing, and performance of the material. Thus, a complete understanding of diffusion related phenomena will result in a coating/substrate system with limited deleterious microstructures, excellent coating/substrate adhesion, and improved performance.

10:10 AM Break

10:30 AM
Assessment of Superalloy-Dependent Lifetime of a NiCoCrAlY Coating Based on Understanding of Interdiffusion in NiAl vs. Superalloys Diffusion Couples: *Emmanuel Perez*[1]; Travis Patterson[1]; Yong-Ho Sohn[1]; [1]University of Central Florida

Superalloy-dependent lifetime of a NiCoCrAlY coating was examined with several solid-to-solid diffusion couples consisting of NiAl vs. various commercial superalloys (i.e., CM247, GTD-111, IN-939, IN-718, and Waspalloy). The couples were assembled, encapsulated in Ar with quartz capsule, and annealed at 1050C for 96 hours. Concentration profiles measured by EPMA in single-phase NiAl region were employed to determine interdiffusion fluxes and effective interdiffusion coefficients of individual components in single-phase NiAl region. The interdiffusion fluxes and effective interdiffusion coefficients determined experimentally in single-phase NiAl region were employed to predict effective interdiffusion coefficients in multi-phase superalloy region. Microstructural and concentration stability of a NiCoCrAlY coating as a function of superalloys are presented based on effective interdiffusion coefficients predicted from diffusion couple studies. This work was financially supported by CAREER award from National Science Foundation (DMR-0238356).

10:55 AM
Copper Diffusion into Silicon Substrates through TaN and Ta/TaN Multilayers Barriers: Florent Bernard[1]; *Nicole Fréty*[1]; Joël Sarradin[1]; Jean-Claude Tedenac[1]; [1]Université de Montpellier II

The progress of the ultra large scale integration (ULSI) device technology is strongly dependent on the development of new wiring materials exhibiting a lower electrical resistivity and a higher resistance to electromigration compared with those of aluminium-based alloys. Copper has then become the most attractive material. However the copper diffusion into the silicon-based substrates during the device fabrication leads to a degradation of the reliability of the ULSI devices. Various barrier layers, such as tungsten and titanium nitride, have then been developed to prevent the copper diffusion, the thickness of which being of about 100 nm. This thickness needed to be reduced, novel barrier layers have to be developed. The aim of this project was to study the potentiality of thin TaN and Ta/TaN multilayer barriers in the diffusion mechanisms of copper into silicon substrates. The diffusion mechanisms were determined from microstructural characterizations (XRD, SEM, AFM) associated to SIMS analyses.

11:20 AM
Diffusion Path and Interdiffusion Structure in the Flip-Chip Eutectic Sn-Pb Solder Bump with Ni/Cu UBM during Reflow and after Aging: Guh-Yaw Jang[1]; Li-Yin Hsiao[1]; Su-Yueh Tsai[2]; *Jenq-Gong Duh*[1]; Mysore A. Dayananda[3]; [1]National Tsing Hua University; [2]Ming Hsin University of Science and Technology; [3]Purdue University

In the current flip-chip technology, Ni-based under-bump metallurgy (UBM) is widely used, and the formation and growth of intermetallic compounds (IMC) between solders and UBM play a critical role in the solder joint reliability. In this study, solder joints of eutectic Pb-Sn/Ni-UBM were employed to investigate the IMC formation during reflows at 220°C and after aging at 150°C. The interfacial microstructures were revealed with FE-SEM through special etching technique. The quantitative analysis and elemental redistribution were obtained by EPMA, and FE-EPMA. Various types of IMC, such as $(Cu,Ni)_6Sn_5$, $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_3Sn$ were observed. With the aid of X-ray color mapping technique, the measured compositions were mapped onto the ternary isotherm of Sn-Ni-Cu. Series of diffusion paths were then constructed, which should help to interpret the phase transformation among related IMCs.

11:45 AM
Interdiffusion Coefficients Extraction from Multicomponent Diffusion Couples: *Liang Jiang*[1]; Jing Lu[1]; Srikanth Akkaram[1]; [1]GE Global Research

The behavior of multicomponent diffusion can be complex due to the inter-dependence of diffusion among multiple elements. Interdiffusion coefficients are critical to characterize diffusion in multicomponent systems. Multicomponent diffusion couples are typically used to extract the interdiffusion coefficient. In the present study, various procedures for determining interdiffusion coefficients are reviewed and a numerical inverse method is proposed. The numerical inverse method is utilized to extract interdiffusion coefficients from experimental data of various binary and ternary diffusion couples.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials V: Electromigration in Leaded and Lead-Free Solder Joints

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

Wednesday AM Room: 213B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Sinn Wen Chen, National Tsing-Hua University; Katsuaki Suganuma, Osaka University

8:30 AM Invited
Electromigration Study in Flip Chip Pure Sn Solder Joints: *Jae-Woong Nah*[1]; Jong-Ook Suh[1]; King-Ning Tu[1]; Yong-Min Kwon[2]; Kyung-Wook Paik[2]; [1]University of California, Los Angeles; [2]KAIST

Electromigration is serious reliability issue in flip chip technology because design rule in devices requires high current density through solder joints. Many papers have reported that the general failure mode induced by electromigration in flip chip solder joints is the loss of UBM and the interfacial void formation at the cathode contact interface between the interconnect line and the solder bump. However, the effect of electromigration on the change of stress distribution in flip chip solder joints has not been studied. In this study, we have prepared pure Sn flip chip samples and used synchrotron radiation white beam x-ray microdiffraction to measure the stress distribution in the pure Sn samples driven by electromigration. The stress analysis on the basis of the in-situ change of lattice parameters was obtained from the diffracted Laue patterns during electromigration. The electromigration induced failure mechanism in flip chip pure Sn solder joints will be presented.

9:00 AM
Current Direction Effects upon Interfacial Reactions in the Sn-In/Cu and Sn-In/Ni Joints: Sinn-Wen Chen[1]; *Shih-Kang Lin*[1]; [1]National Tsing Hua University

Sn-In alloys are promising low melting point Pb-free solders. Cu and Ni are commonly used substrate materials. At the Sn-In/Cu and Sn-In/Ni joints in electronic products, both electromigration and Joule heating contribute significantly to the interfacial reactions. Sn-20at.%In/Cu and Sn-20at.%In/Ni couples are prepared, and they are reacted at 160 and 140°C with and without the passage of DC currents. It is found that the current directions have a strong effect upon the interfacial reactions. When the electrons are from the solder to the substrate, the reaction products are η-Cu6Sn5 and Ni3Sn4 in the two different couples, respectively. The IMCs' formation and their morphologies are similar to those without passing through of electric currents. However, when the electrons are from the substrate into the solder side, very significant interfacial reactions are observed. It is likely that the liquid phase is formed from interfacial reactions and the substrate is consumed seriously.

9:25 AM
Effect of Aging on Electromigration of Flip-Chip Solder Joints: *Che Cheng Chang*[1]; Chih Chen[1]; [1]National Chiao Tung University

Electromigration has become an important reliability issue in flip chip technology. In this study, effect of aging on electromigration of flip-chip solder joints was investigated. Eutectic Sn63Pb37 solder bumps were aged

at 150°C for different aging times, and then electromigration test were performed. Both solder bumps with thin-film and thick-film under Bump Metallurgy (UBM) were examined. It is found that electromigration life time decreased when the aging time increased for solder bumps with thin-film UBM, whereas the life time increased as the aging time increased for the solder bumps with thick-film UBM. It is speculated that the intermetallic compound layer played critical role during the electromigration test. The reasons that caused the different behaviors will be presented in the conference.

9:50 AM

Electromigration Effects on Intermetallic Growth Trends: *Helen T. Orchard*[1]; A. Lindsay Greer[1]; [1]University of Cambridge

Microelectronic device structures continue to be miniaturised, allowing improvements in performance, but also introducing new reliability issues. Of interest are bimetallic interfaces across devices, such as wire bonds and solder joints. Intermetallic compounds form at these interfaces, and excessive growth can cause device failure. Theoretical work shows that when a direct current is passed through a bimetallic couple, an electromigration effect can occur, resulting in a change in the rate of intermetallic thickening [Orchard and Greer, Appl. Phys. Lett. 86 (2005) 231906, 1-3]. When the electromigration flux augments the diffusion flux the intermetallic thickening is accelerated. If the electromigration flux opposes the diffusion flux the intermetallic thickening is slowed and the intermetallic reaches a limiting thickness. In this paper we compare the predicted growth regimes to experimental observations of intermetallic thickening with a direct current. The model is extended to include multiphase intermetallic compounds and alternating current conditions.

10:15 AM

Electromigration Effects upon Microstructural Evolution and Interfacial Reaction of Eutectic SnBi Solder on Au/Ni Metallization: *Chih-Ming Chen*[1]; Long-Tai Chen[1]; [1]National Chung Hsing University

Eutectic SnBi alloy is one of the most potential candidates for low-temperature lead-free solders. Electromigration is one of the most important reliability concerns in solder systems. This study investigated electromigration effects on microstructural evolution of eutectic SnBi solder and interfacial reaction between eutectic SnBi solder and Au/Ni metallization. Eutectic SnBi solder ball was placed in between two Au/Ni metallization pads, and by conducting a reflow a sandwich-type reaction couple was fabricated. Experiments were conducted by passing through a constant current in the reaction couples. Current density was 5×10^3A/cm^2 and temperature was 70°C. Accumulation of Bi at the anode side was observed, and by measuring the accumulation rate of Bi the apparent effective charge was estimated. Different intermetallic growth and interfacial morphology were also observed at the anode and cathode sides. Current distributions inside the solder and at the joint interface were simulated and correlated with the experimental observations.

10:40 AM Break

10:50 AM

In-Situ Electromigration Studies on Sn-Ag-Cu Solder Joint by Digital Image Speckle Analysis: *John H. L. Pang*[1]; Luhua Xu[1]; [1]Nanyang Technological University

The phenomenon of electromigration in Pb-free Sn-Ag-Cu solder joint specimen subject to high current density was characterized by in-situ test. Digital Image Speckle Analysis (DISA) was used to measure the in-situ micro-deformation of a cross sectioned solder joints, which is subject to electromigration with a current density of 5000 A/cm^2 under a constant temperature of 125°C. The tests were conducted on specimen with different geometry, lapshear specimen and wired specimen. It was recognized in this study that EM can cause strain gradient effects from cathode to anode. High strain concentration was found near pre-existed void. Void growth is anticipated at current crowding regions and this can be measured by in-situ digital image measurements and correlation analysis.

11:15 AM

In-Situ Observation of Pb and Sn Migration during Electromigration-Induced Drift of Eutectic SnPb Lines: *Young-Bae Park*[1]; Min-Seung Yoon[2]; Oh-Han Kim[1]; Min-Ku Ko[2]; Yong-Duk Lee[1]; Ja-Young Jung[1]; Young-Chang Joo[2]; [1]Andong National University; [2]Seoul National University

Flip chip packages with smaller size are likely to have higher current densities through their solder bumps, which has given rise to electrical failure due to solder electromigration. Since the eutectic SnPb solder bump consists of two major elements, it is necessary to take both the migration of Sn and that of Pb into consideration. In this study, we have investigated the distinct migration behavior of both Sn and Pb in the eutectic SnPb solder lines as a function of temperature and current density by using in-situ SEM. It was of interest that an incubation stage for the edge drift was observed during electromigration of the eutectic SnPb solder lines. It was clearly confirmed by in-situ observation that Pb migrated dominantly at 110°C while Sn migrated dominantly at 50°C. The existence of incubation stage for edge drift and the temperature dependence of migration elements will be discussed in detail.

11:40 AM

Redistribution of Pb-Rich Phase during Electromigration in Eutectic SnPb Solder Stripes: *Cheng-Chang Wei*[1]; Chung-Kwang Chou[1]; Chih Chen[1]; [1]National Chiao Tung University

By using Blech structure, we studied the EM behaviors on SnPb solder stripes. The thickness of the solder stripes varied from 3 to 8um and the width of the stripes was 100um. In order to investigate the microstructure evolution in the eutectic SnPb solder, we applied high current density to stripes. It is found that Pb-rich phase ripened and aligned along the direction of electron flow after the current stressing of 9.7x103 A/cm^2 at 80°C for 24 hours. It is very interesting that this phenomena became more significant with the stressing time or current density increased. Simulation results shows that the resistance of the solder stripes may be reduced when the Pb-rich phase aligns along the direction of the electron flow. This reduction of the total resistance provides the driving force for the Pb-rich phase to align along the direction of the electron flow.

12:05 PM

Thermomigration in Composite High Pb - Eutectic SnPb Flip Chip Solder Joints: *Annie T. Huang*[1]; King-Ning Tu[1]; [1]University of California

High current density in the Al interconnect can cause a large joule heating in flip chip solder joints. Because Silicon is a much better thermal conductor than a polymer substrate, temperature across a silicon chip differs only by a few degrees whereas a large temperature change occurs across a polymer substrate side from the bump with current stressing to no-current bumps. Temperature gradient thus exists across the bumps located on the same chip regardless if the bumps have current or not. Composite solder consists of 97Pb3Sn and eutectic SnPb. Because of the difference in the diffusion rate between Sn and Pb, its unique structure can be utilized to study thermomigration based on the redistribution of Sn and Pb. In this talk, we report the thermomigration and failure mode of composite solder bumps without current stressing by a thermal gradient induced by joule heating from the neighboring bumps with current stressing.

Phase Transformations in Magnetic Materials: Magnetic Shape Memory Alloys and Information Storage

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

Wednesday AM Room: 213A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Marc J. DeGraef, Carnegie Mellon University; Lynn Kurihara, Naval Research Laboratory

8:30 AM Invited
Formation and Evolution of Domain Strutures in Ferromagnetic Shape Memory Alloys: Jingxian Zhang[1]; *Long Qing Chen*[1]; [1]Pennsylvania State University

Ferromagnetic shape memory alloys undergo both ferromagnetic and ferroelastic (martensitic) phase transformations. This presentation will discuss a computational model for predicting formation and evolution of both the ferroelastic and ferromagnetic domain structures in ferromagnetic shape memory alloys. It combines the phase-field approach with micromagnetics and microelasticity theory. As an example, the Ni2MnGa ferromagnetic shape memory alloy is considered. The emphasis is on the overall strain response and associated evolution of magnetic domain structure and martensite microstructure under an applied stress or a magnetic field with different initial conditions. The interactions between martensite twins and magnetic domain walls will be discussed. The energetic contributions that are responsible for the experimentally observed reorientation of magnetic domain walls during martensite plate growth will be analyzed. Finally, the effects of defects on the formation of tweed structures as well as on the twin boundary mobility under a stress or magnetic field are investigated.

9:05 AM Invited
Interfacial Phase Formation during Growth of Ferromagnetic $CdCr_2Se_4$ on AlGaAs/GaAs: *Ramasis Goswami*[1]; George Kioseoglou[2]; Aubrey T. Hanbicki[2]; Berend T. Jonker[2]; George Spanos[2]; [1]Geo-Centers Inc.; [2]Naval Research Laboratory

Ferromagnetic semiconductors (FMS) provide an opportunity to control spin dependent behavior and study spin injection and transport in semiconductor heterostructures. A key element of semiconductor-spintronics technology is the quality of heterointerfaces that affect the injection of polarized carriers. This presentation is centered about the overall microstructural evolution, interfacial phase formation, and magnetic properties of ferromagnetic off-stochiometric $CdCr_2Se_4$ thin films deposited on AlGaAs/GaAs LED structures. High Resolution Transmission Electron Microscopy (HRTEM), Z-contrast imaging, Electron Energy Loss Spectroscopy (EELS), and Energy Dispersive Spectroscopy (EDS) were used to characterize the films. Z-contrast imaging revealed that Cr-rich nano-islands ~2 nm thick formed epitaxially at the AlGaAs side of the $CdCr_2Se_4$/AlGaAs interface. Z-contrast imaging and HRTEM studies suggest that the possible interfacial phase is CrAs, which is a half-metallic ferromagnet. Detailed microstructural analysis of the interface and comparison with near stochiometric $CdCr_2Se_4$ on AlGaAs/GaAs (LED), and on ZnSe/AlGaAs/GaAs (LED), will be presented.

9:40 AM
Phase Transformation in Nonstoichiometric $Ni_{49}Mn_{23}Ga_{28}$: *Peng Zhao*[1]; Liyang Dai[1]; James Cullen[1]; Manfred Wuttig[1]; [1]University of Maryland, College Park

The martensitic phase transformation in a nonstoichiometric alloy, $Ni_{49}Mn_{23}Ga_{28}$, with M_s=273 K and T_C=383 K has been studied. The martensitic phase is a tetragonal with a c/a ratio of 1.33 and lattice distortion of about 6.1%, determined by high resolution neutron diffraction. Hence, it shows potential capability to exhibit ferromagnetic shape memory effect (FSME) because the FSME depends largely on the tetragonality. The temperature dependence of magnetization shows a temperature hysteresis of about 10 K, demonstrating that the martensitic phase transformation is reversible. The low field thermal magnetization curve shows that the alloy also undergoes a pre-martensitic transformation at around 333 K, corresponding to the anomalies displayed by the temperature dependence of elastic constants C_{44} and C'. This intermediate, pre-martensitic state is believed to have a micro-modulated structure that develops prior to the transformation of the parent cubic phase to the tetragonal phase.

10:15 AM Invited
Magnetic Nanostructures for Spintronics Applications: *Adekunle Olusola Adeyeye*[1]; Navab Singh[2]; Sarjoosing Goolaup[1]; Chenchen Wang[1]; Debashish Tripathy[1]; Jun Wang[1]; [1]National University of Singapore; [2]Institute of Microelectronics

There has been considerable interest in magnetic nanostructures in the last decade due to advances in nanofabrication and nanocharacterization techniques. From an application point of view, nanomagnets are the building blocks of magnetic random access memory cell and also patterned magnetic media. In this talk, we will present a large area nanofabrication technique for synthesizing ferromagnetic nanostructures using deep ultraviolet lithography at 248 nm exposing wavelength. One unique advantage of this technique is that unlike e-beam lithography, thicker resists can be used to make high aspect ratio nanostructures. Ferromagnetic nanostructures of different shapes and sizes were fabricated. The magnetic properties were characterized using vibrating sample magnetometer, magnetic force microscopy and magnetotransport measurements. We observed that the magnetic properties of the nanostructures are strongly dependent on the size and shape of the nanostructures. We have also used micromagnetic modeling to aid our understanding of the magnetization reversal process in the nanomagnets.

10:50 AM Break

11:00 AM
Micromagnetism in the Ultrathin Limit: *Danilo Pescia*[1]; Oliver Portmann[1]; Andreas Vaterlaus[1]; [1]ETH Zurich

We derive some results concerning the static and dynamic micromagnetic behaviour of magnetic elements in the ultrathin limit. In this limit, a most remarkable logarithmic correction of the magnetostic energy appears. Experimentally observed phenomena such as the quenching of the precessional motion at boundaries and the multi-to-single domain transition in ultrathin elements with perpendicular magnetization are ascribed to this logarithmic singularity. Because of the competition between the very weak but long ranged dipolar interaction and the strong but short ranged exchange interaction, perpendicularly magnetized ultrathin films are unstable against stripe domain formation. This competition completely changes the pattern of the phase transition: the stripe system is forced out of the Landau-Ginzburg-Wilson universality class and becomes a Coulomb frustrated ferromagnet (CFF). Because of its complexity, the phase transition in a CFF is presently a subject of strong controversy. Here we discuss some theoretical and experimental aspects of a CFF.

11:25 AM Invited
Short Range Order, Long Range Order and Phase Miscibility of Nanostructured CoCrPt and FePt Media Films: *Gan Moog Chow*[1]; [1]National University of Singapore

The properties of nanostructured magnetic films are controlled by composition, dopants, structure, microstructure, texture and interfaces. Nanostructure alloying may not necessarily follow conventional phase diagrams that ignore the effects of surfaces and interfaces. The composition of a textured long range order, which may differ from the global average composition, needs to be controlled to provide optimum texture-dependent properties. In this talk, selected examples of some of our work on nanostructured CoCrPt and FePt films for high density magnetic recording are discussed. The effects of seedlayer and underlayer, long range order, short range order, compositions of textured Bragg peak and dopants were investigated using high resolution x-ray scattering, anomalous x-ray scattering, extended x-ray absorption fine structure, transmission electron microscopy and vibrating sample magnetometry. Correlations of magnetic properties with structures, phase miscibility and dopants are addressed.

Point Defects in Materials: Thermodynamics

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

Wednesday AM — Room: 210B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: David J. Srolovitz, Princeton University

8:30 AM Invited

Short-Range Ordering, Clustering, and Early-Stage Precipitation in a Model Ni-Al-Cr Superalloy: *Chantal K. Sudbrack*[1]; Zugang Mao[1]; Ronald D. Noebe[2]; David N. Seidman[1]; [1]Northwestern University; [2]NASA

Short-range ordering (SRO) and clustering in substitutional alloys occurs by solute diffusion, which is mediated by a vacancy mechanism. These processes have been primarily characterized in binary alloys with small-angle scattering in reciprocal-space, as meaningful quantification is difficult for multicomponent alloys (technologically important systems). A radial distribution function analysis of direct-space atom-probe tomography images allows these processes to be studied in detail in a Ni-Al-Cr superalloy, where $L1_2$-ordered $Ni_3(Al_xCr_{1-x})$ precipitates form via a first-order transformation. The fast diffusion of Al leads to the observation of $L1_2$-Ni_3Al SRO domains ($\langle R \rangle$ = 0.6 nm), after the initial quench to room temperature from the single phase regime. Congruent SRO precedes phase separation, where phase separation is established by compositional fluctuations that are small in spatial extent ($\langle R \rangle$ = 0.75 nm) and large in amplitude. The observed pathways are considered in light of classical nucleation theory and compared to lattice kinetic Monte Carlo simulations.

9:00 AM

Effect of a Ternary Addition on Ordering Kinetics in $L1_2$ -Ordered Quasi-Binaries: *Ewa Partyka*[1]; Rafal Kozubski[1]; [1]Institute of Physics Jagiellonian University

Studies of the influence of admixing a ternary element on long-range ordering kinetics in quasi-binary systems with $L1_2$ superstructure were carried out.The intermetallic $Ni_3\ Al_{1-x}\ Fe_x$ system showing a decrease of the ordered phase stability upon alloying with Fe was examined by means of quasi-residual resistometry and positron lifetime spectroscopy. It has been found out that the previously reported decrease of the activation energy for ordering kinetics with increasing Fe concentration is due to a decrease of the vacancy formation energy. It suggests an increase of the "vacancy availability" in the neighbourhood of Fe atoms with an increase of the Fe content and easier diffiusion of Fe atoms than Al atoms within Ni-sublattice. Applied Monte Carlo simulations revealed the atomistic mechanism of the ordering process in $L1_2$ -ordered quasi-binaries which is controlled mainly by the migration of a ternary component within the majority sublattice.

9:20 AM Invited

Thermodynamics of Impurities in Vanadium: *Oliver Delaire*[1]; Tabitha Swan-Wood[1]; Max Guy Kresch[1]; Brent T. Fultz[1]; [1]California Institute of Technology

We investigated the effects of dilute alloying on the lattice dynamics and electronic structure of bcc vanadium. Using inelastic neutron scattering, we have measured the phonon density of states and vibrational entropy of random solid solutions of vanadium with transition metal impurities. Group IVa elements caused a softening of the V phonons, while elements to the right of V induced a gradually increasing stiffening. The stiffening observed for 6% Pt impurities is very large and induces a decrease of vibrational entropy that is larger than the gain in configurational entropy, resulting in a negative entropy of mixing. Using density functional theory, we calculated the electronic structure for all alloys and found that the electronic entropy of alloying follows the same trend as the vibrational part, although it is smaller in magnitude. We discuss the consequences of these findings on the thermodynamics of impurities in vanadium.

9:50 AM

Inelastic Neutron Scattering Studies on the Formation Entropy of Vacancies in FeAl: *Tabitha L. Swan-Wood*[1]; Olivier Delaire[1]; Max Kresch[1]; Brent Fultz[1]; [1]California Institute of Technology

Inelastic neutron scattering spectra of B2 FeAl with quenched vacancies were measured giving the vibrational entropy of vacancy formation. Born von Karman calculations indicate that point defects change the phonon dispersions. The vibrational entropy of vacancy formation is measured to have an upper bound of -0.75 kB/vacancy. Inelastic neutron scattering spectra also measured anharmonic vibrational entropy of B2 FeAl between temperatures 10K to 1323K. We postulate that this anharmonicity is caused by vacancies and Fe anti-site defects in the alloy.

10:10 AM Break

10:25 AM Invited

The Kinetics of Crystallization: Role of Point Defects: *Yinon Ashkenazy*[1]; Robert S. Averback[2]; [1]Hebrew University of Jerusalem; [2]University of Illinois at Urbana Champaign

The common model for crystallization speeds in metals assumes that the velocity of the crystal-liquid interface in metals at deep under-coolings is limited by the rate at which liquid atoms collide with the crystalline interface. We show here by MD simulations that in contrast to current models, crystallization of under-cooled liquids and metallic glasses is controlled by diffusion, in accordance with recent experiments. The big surprise, however, is that the relevant diffusion coefficient appears to be that of self-interstitial atoms in the crystalline state. For the three metals, Fe, Ni and Cu, we find the activation energies of crystallization, from both the liquid and glass states, are precisely those found for the migration energy of self-interstitial atoms. These measurements give new credence to the model attributed to Frenkel that liquids are crystals with high concentrations of point defects, and more specifically to Granato's interstitial theory of liquids.

10:55 AM

Impact of Vacancy Diffusion on the Early Decomposition Stages of Alloys and the Role of Heterophase Interfaces on Coarsening: Zugang Mao[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; Georges P. Martin[1]; *David N. Seidman*[1]; [1]Northwestern University

The kinetic pathway for nucleation and growth is commonly thought to be dictated by the initial supersaturation of solutes in the solid solution undergoing phase separation. We demonstrate that the details of the diffusion mechanism, in the solid solution affect deeply the early stage morphologies of precipitates. Our argument is based on a combined use of atomic-scale observations, with atom-probe tomography (APT) and lattice kinetic Monte Carlo simulation of a Ni(Al,Cr) alloy. By an optimized choice of thermodynamic and kinetic parameters we first reproduce the experimental APT observations. We then modify only the long-range vacancy-solute binding energy, without altering the thermodynamic driving force for phase separation, thereby demonstrating that the microstructural evolution changes from a coagulation to an evaporation-condensation coalescence mechanism. The changes can only be accounted for with non-zero values for the vacancy chemical potential and off-diagonal terms of the Onsager matrix, at variance with classical models.

11:15 AM

Effect of the Antisite Defects on the Temperature Dependence of the Lattice Misfit in γ/γ' Alloys: *Oleg Y. Kontsevoi*[1]; Yuri N. Gornostyrev[1]; Arthur J. Freeman[1]; [1]Northwestern University

The magnitude of the lattice misfit δ between the γ matrix and γ' precipitate phases is one of the key parameters determining the mechanical behavior and microstructure morphology of two-phase high temperature superalloys. The two main contributions to the temperature dependence δ(T) are under intense investigation: (i) the difference in thermal expansion of the two phases, and (ii) the redistribution of alloying components between γ and γ' which creates the substitutional and antisite defects. We explore the role of both contributions for the Ni-Al and Ir-Nb two-phase

alloys based on first principles calculations of the phonon spectra and antisite defects. We demonstrate that the δ(T) behavior depends on the shape of the γ - γ' gap on the phase diagram. The redistribution of the alloy components gives the main contribution to delta(T) for Ni/Ni_3Al, in contrast with Ir/Ir_3Nb where the thermal expansion dominates. Supported by the AFOSR (grant FA9550-04-1-0013).

11:35 AM

Formation of Topologically Close Packed Impurity Locked Phases in Mo and Cr Base Alloys: Nadezhda I. Medvedeva[1]; *Oleg Y. Kontsevoi*[2]; Yuri N. Gornostyrev[2]; Arthur J. Freeman[2]; [1]Institute of Solid State Chemistry; [2]Northwestern University

The application of bcc refractory metals is limited by their room temperature brittleness, which is caused by interstitial impurities (C, N, O) that form brittle carbides, nitrides and oxides. Alloying with rhenium dramatically increases ductility, and the proposed mechanism of this effect connects the increase of the solubility of impurities with the appearance of topologically close packed (TCP) particles. By means of first-principles calculations, we investigated the cumulative effect of both Re alloying and light interstitial impurities on the structural stability of the A15 phase of Mo and Cr, assumed as the the prototype TCP phase. We demonstrate that the metastable A15 M_3Re (M=Mo,Cr) is stabilized by the interstitial impurities. The precipitates of this impurity locked phase may serve as scavengers of interstitials, prevent the formation of brittle phases and grain boundary segregation, and thus improve the ductility of Mo and Cr alloys. Supported by the AFOSR (grant FA9550-04-1-0013).

11:55 AM

Effect of Vacancy on Al-Li Alloy in Earlier Aging Condition: *Gao Yingjun*[1]; [1]Guangxi University

Al-Li alloys are very attractive in the aerospace industry. Binary Al-Li alloys are strengthened by the solute segregation zone and Al3Li particles. Solute segregation zone models are used to calculate the atomic bonding of the solute segregation zone with a vacancy defect of Al-Li alloy in earlier aging condition. Three kinds of atomic bond order are given by bonds of Al-Al, Al-Li and Al-Li-vacancy in the solute segregation zone according to the empirical electron theory (EET) in solid. The strongest covalent bond in the segregation zone with vacancy defect, formed in quenching state of alloy, are the main strengthening reason for alloy in earlier aging condition. These results explain the experimental findings.

Recycling - General Sessions: Aluminum 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 International

Wednesday AM — Room: 8B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Cynthia K. Belt, Aleris International

8:30 AM Introductory Comments

8:35 AM

Emerging Trends in Aluminum Recycling: Reasons and Responses: *Subodh K. Das*[1]; [1]Secat Inc

The growth in aluminum usage in transportation applications, the decline in aluminum beverage can recycling, and the increasing reliance of the domestic fabrication industry on secondary aluminum have combined to create new needs in both the materials design and processing space. This presentation will detail the history and future projections for aluminum recycling, emphasizing the increasing importance of mixed scrap streams in the makeup of secondary aluminum. To most economically utilize these scrap streams, new approaches to developing acceptable materials processed to control properties suitable for an expanded range of applications are needed. How the aluminum enterprise, including industry, academia, and government can work together to meet these important but aggressive targets and transform recycling from strictly an environmental imperative to an economic development opportunity will be discussed.

9:00 AM

Raw Material Usage Strategies under Conditions of Uncertain Alloy Demand: *Gabrielle Gaustad*[1]; Preston Li[1]; Randolph Kirchain[1]; [1]Massachusetts Institute of Technology

Operational uncertainties are the source of much inefficiency in metal alloy production. Past work has shown that recourse model based optimization can identify raw materials acquisition strategies that mute the effects of at least one form of uncertainty - variation in product demand. These models suggest that additional scrap purchase and usage, referred to as purchase hedging, provides financial advantage for remelters across a range of operating conditions. This paper investigates the correlation of production and raw materials characteristics to preferred hedging strategy. In this study, cases are presented involving available scrap materials used in production of both cast and wrought products. Early results indicate that hedging strategy is correlated to the diversity of the production portfolio and the versatility of scrap types.

9:25 AM

Material Selection and the Impact on Recyclability, Green Purchasing and Corporate Social Responsibility – The Manganese Metal Case: *John E. Heinze*[1]; Karen Hagelstein[2]; [1]Environmental Health Research Foundation; [2]Times Limited

Manganese metal is widely used as an alloying agent in the aluminum industry and is available from two distinct production processes. The environmentally preferred process uses sulfur as the catalyst and results in a typical purity of 99.9%. The other process, favored by many Chinese manufacturers, uses selenium as the catalyst with the result that the manganese metal contains up to 0.15% selenium but is produced at a lower cost. This has resulted in 75% of the global market in recent years consisting of selenium-contaminated manganese. A recent case study demonstrates that use of this material has an impact on the recyclability of the aluminum dross and scrap, where recyclers face metal dust exposure issues and may not even be aware of the selenium content that likely triggers hazardous solid waste disposal requirements. This illustrates downstream recycling is an important consideration of green purchasing and corporate social responsibility.

9:50 AM Break

10:05 AM

Recycling Wastes in the Alumina Industry: *N. Ilyoukha*[1]; V. Timofeeva[2]; [1]Aleris International; [2]Academic Ceramic Center

Main wastes of the alumina and aluminum industries are not used at present and are being stored after partial treatment in special places. The paper will discuss the technology of treatment wastes as an additive for the cement industry. The solution of this complex problem will be of great importance for the purification of aerial, earth and waste region in whole. Besides this, as a result of waste treatment there will be special cements: high strength, extensible, gas-water proof, self-stressing needed in the road and runway construction, in building tunnels and pits, and in nuclear wastes burial.

10:30 AM

State of the Art in Recovery of Aluminum from Aluminum Dross: *Lifeng Zhang*[1]; [1]University of Illinois

The paper reviews the current research and development of Aluminum recovery from aluminum dross, including dross types and dross properties, and methods for metal Al recovery from dross, such as reducing metal loss during cooling, separation of metal from dross, and remelting of the metallic fraction. Techniques to separate Al metal from the dross, such as stirring hot dross, crushing and screening cold dross, bubble flotation, and leaching process, are discussed. Several remelting methods are summarized, such as reverberatory furnace, rotary furnaces, salt-free thermal plasma furnaces, and salt-free rotary Arc furnace with graphite electrodes. Fundamentals related to these dross recovery processes are reviewed too, such as thermodynamics and kinetics, lab experiments, and numerical simulation on the fluid flow, electromagnetic field and forces, chemical

reactions, combustion, multiphase flow, heat transfer, radiation, melting, and top surface phenomena. This review provides clear research directions for the Al recovery from Aluminum dross.

10:55 AM Concluding Comments

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

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

Wednesday AM Room: 6D
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Mei Li, Ford Motor Company; David Robert Poirier, University of Arizona

8:30 AM Invited
Modeling of Entrainment Defects: *John Campbell*[1]; [1]General Motors Corporation

The enfolding action of the liquid surface involved in surface turbulence, and sometimes in laminar surface flow, usually enfolds air inside the surface oxide film, creating both bubbles, and unbonded double oxide interfaces in the liquid that act as cracks. To fully understand and predict casting defects, it is essential that such events are included in computer models since such defects are now known to be numerous in liquid Al alloys. For instance up to 85% or more of casting defects are entrainment defects, or defects that are initiated by and grown from entrainment defects. Over the past few years the modeling of such bubbles and bifilm creation events has begun to be tackled with some success. These successes are examined and future challenges for models will be assessed.

8:55 AM Invited
Modeling of Porosity Formation in Aluminum Alloys: *Christoph Beckermann*[1]; Kent D. Carlson[1]; Zhiping Lin[2]; [1]University of Iowa; [2]Caterpillar

A new approach based on microsegregation of gas dissolved in the melt is used to model pore formation during the solidification of aluminum alloy castings. The model predicts the amount and size of the porosity in the solidified casting. Computation of the gas species transport in the melt is coupled with the simulation of the feeding flow and calculation of the pressure and temperature fields. The rate of pore growth is calculated based on the local level of gas supersaturation in the melt. The effects of the dendritic and eutectic microstructure on pore formation are also taken into account. Parametric studies for one-dimensional solidification under an imposed temperature gradient and cooling rate illustrate that the model captures all important phenomena observed in porosity formation in aluminum foundry alloys. Comparisons between predicted porosity distributions and experimental measurements show good correspondence.

9:20 AM Invited
Modeling of Porosity Formation in Multicomponent Alloys in the Presence of Several Dissolved Gases and Volatile Solute Elements: Gael Couturier[1]; *Michel Rappaz*[1]; [1]Ecole Polytechnique Fédérale de Lausanne

Although microporosity is a critical issue for the mechanical properties of cast parts, modeling of such defects is still not yet fully satisfactory. The present contribution addresses the problem of multicomponent alloys and multigas systems including volatile solute elements. The effects of all solute elements on the activity of gas elements (hydrogen in the case of aluminum alloys) are first taken into account, together with the partial vapor pressure of volatile elements such as zinc. After writing an overall mass balance for each element contributing to porosity formation, a linearized system of equations can be solved to relate the increment of porosity to the local pressure. This general formalism has been implemented in a program that calculates the pressures drop in the mushy zone using Darcy's equation and an adaptive refined mesh. Examples related to aluminum alloys will be shown in order to emphasize the various contributions to porosity formation.

9:45 AM Invited
A Porosity Simulation for High Pressure Die Casting: *Chung-Whee M. Kim*[1]; Kimio Kubo[1]; Ken Siersma[1]; [1]EKK Inc.

High pressure die castings have a tendency to have small size porosity defects, which are caused by gas holes and shrinkage. The origin of the gas holes is considered to be mainly hydrogen or nitrogen. Hydrogen evolves from liquid during solidification and nitrogen is a part of the trapped air during filling. This air trapped porosity reduces the quality of the die castings and prevents the use of heat treatment. Shrinkage porosity in the die castings is caused by hot spots just like the other casting processes. A model of the initialization and growth of porosity in the high pressure die castings is proposed. First, trapped air porosity forms during filling. Some of these porosity become large size porosity during solidification. The calculated results of size and distribution of porosity in magnesium die casting plates are compared favorably with the experiments.

10:10 AM Break

10:25 AM
Computational Analysis of Oxide Inclusions in Aluminum Castings: *Gerald P. Backer*[1]; Chung Whee Kim[2]; Ken Siersma[2]; Qigui Wang[1]; [1]General Motors; [2]EKK, Inc

Computer simulations of mold filling and solidification behavior have been used by foundries for years to optimize casting design. However, one aspect of the casting process that has not yet been carefully considered by simulation is the formation of oxides during filling. It is well known that oxides are very detrimental to the microstructure and mechanical properties, as they are often sites for nucleation of shrinkage porosity. In this paper, oxide formation during mold filling is studied computationally and experimentally with 319 and A356 aluminum alloys in both gravity pour and low pressure fill conditions. The simulation results and computational techniques will be discussed.

10:50 AM Invited
Study on Improvement of Casting Design System Based on John Campbell's Casting Design Rules: Henry Hu[1]; Zhaoxia Li[1]; Jean Kor[1]; *Qigui Wang*[2]; Wenying Yang[2]; [1]University of Windsor; [2]GM - Powertrain Engineering HQ

Casting design has a direct influence on the quality and cost of cast components. A full casting design includes design of components, a gating system and casting process parameters. At all times, many efforts have been done to improve quality of castings and at the same time to reduce cost and lead time. However, there are still many problems exist for the casting design process. In this paper, the improvement of casting design system, especially the design of gating system based on John Campbell's casting design rules has been studied. Under the guide of John Campbell's casting design rules, a preferable gating system has been designed. The filling process and the quality of the casting have been simulated using the software Magmasoft\@. The result shows, with the improved gating system, the filling process and the quality of castings have been greatly improved.

11:15 AM
Microporosity Modeling in Aluminum Castings: *Gerald Backer*[1]; Qigui Wang[1]; [1]General Motors Corporation

A new computational method for predicting microporosity in aluminum alloys is described. The method was calibrated against literature data for binary Al-7%Si alloys, and subsequently applied to a chill plate test casting in A356 alloy. The new method allows spherical micropores to nucleate and grow by hydrogen diffusion from a material volume surrounding the pores. This differs from a state of the art interdendritic-flow computational model for calculating porosity that assumes spherical pores

have a diameter given by the secondary dendrite arm spacing. The pore growth method predicts larger pore diameters and a volume fraction of microporosity that is in better agreement with experimental observations than the interdendritic-flow model.

11:40 AM
Study of Micro Defects in Aluminum Casting Using X-Ray Computed Tomography: *Kaname Sasaki*[1]; Harsha Badarinarayan[1]; [1]Hitachi America, Ltd.

Using X-ray Computed Tomography (CT), an aluminum casting specimen was scanned to quantify the amount of the micro defects present. Due to the enormous file size of the X-ray CT raw data, the specimen was limited to 10 x 10 x 180mm in dimension. The pixel resolution was 30micron, and the actual observable defect size was approximately 70 micron. This was done in order to eliminate noise and artifacts that were present in the raw scanned data. The specimen was divided equally into 15 vertical sections. The count and volume of the defects in each section increased from the base to the top section. This was because the casting was in contact with a copper chill at the base and opened to the atmosphere at the top. The defects were more prominent in the top region where a number of small voids (with an approximate diameter 80 micron) were detected.

Solidification Modelling and Microstructure Formation: A Symposium in Honor of Prof. John Hunt: Solidification Defects

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

Wednesday AM Room: 6C
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: A. Dahle, University of Queensland; M. Rappaz, Ecole Polytechnique Fédérale de Lausanne

8:30 AM Invited
Microporosity in Cast Alloys: Contributions to the Understanding of Its Formation and Consequences: *Gérard Robert Lesoult*[1]; [1]Ecole des Mines de Nancy

A few examples of microporosity patterns are selected first on the basis of the significance of their effects on the solidification or post-solidification behaviour and properties of cast products. This first part is completed by a list of the main casting conditions that affect microporosity. The second part summarises the various research programmes that were carried out in Paris and Nancy to study the formation of microporosity in different types of cast alloys: nickel-based alloys in investment casting, spheroidal graphite cast iron, and aluminium-based alloys. The third part of this paper presents the most useful and comprehensive physical ideas that allow understanding of the formation of microporosity for various cast alloys and various casting processes. Emphasis is put on alloy chemistry, hydrostatic pressure in the liquid, microstructure and mechanical state of the solid in the mushy zone. Similarities and differences between microporosity and hot tearing are tentatively drawn.

8:55 AM Invited
Phase-Field Simulation of Microporosity Formation in Solidification: *Christoph Beckermann*[1]; Ying Sun[1]; [1]University of Iowa

Direct numerical simulations on a microscopic scale are presented of porosity formation due to gas evolution during alloy solidification. The gas bubbles nucleate and grow in the supersaturated liquid ahead of the solidification front. Flows are induced by the interaction of the bubbles with the solid and the large density contrast between the bubbles and the melt. A phase-field method is used to model the interface and triple-junction motions in the present three-phase system. The flows and species transport in the gas and liquid phases are solved using a diffuse interface model for two-phase flows with surface tension and phase change. The pore growth, solid-pore interactions, and final shape of the pores are investigated as a function of the initial gas concentration, the nucleation supersaturation, and important solidification parameters. The interactions of the pores with cellular and dendritic microstructures are examined in detail and compared to experimental observations.

9:20 AM
Permeability Gradient Model for Predicting Shrinkage Porosity Formation: Danylo Oryshchyn[1]; *Omer Dogan*[1]; [1]U.S. Department of Energy/Albany Research Center

This model examines porosity formation in castings as a function of the ability of liquid to fill voids in the densest region of the mushy zone. These voids occur as the final interdendritic liquid freezes, shrinking. A minimum liquid flow rate is required to fill voids before final solidification. This minimum flow is accompanied by a pressure drop across the mushy zone. Driving pressure must match or exceed this drop. The permeability gradient model tests the impact of interdendritic channel constriction on liquid flow in a casting. The model examines two regimes: (i) Dendritic solidification: permeability dominated by liquid volume fraction and dendrite arm spacing, and (ii) Eutectic solidification: flow dominated by eutectic viscosity. The cooling eutectic mixture is assumed to behave like a slurry with increasing solid fraction. It is envisioned that this model can be developed into a tool that can be very useful for metal casters.

9:45 AM
Migration of Bubbles in the Mushy Zone: *Qingyou Han*[1]; [1]Oak Ridge National Laboratory

Bubbles usually form during solidification of alloys having a large solubility of gas in the liquid but a negligible solubility of gas in the solid. These bubbles become pores in a solidified casting. Lee and Hunt observed the formation of worm-like bubbles during solidification of aluminum alloys using X-ray. We observed, in transparent organic materials, that this worm-like morphology occurred at certain conditions. More often, bubbles migrate or jump from locations to locations in the mushy zone. It is the migration of bubbles that determine the final distribution and size of the pore in an solidifying casting.

10:10 AM Break

10:25 AM Invited
Dendrite Growth and Stability: Implications for Defect Control in Single Crystal Superalloy Castings: Peter D. Lee[1]; Barbara A. Shollock[1]; *Malcolm McLean*[1]; [1]Imperial College London

The potential of single crystal superalloy castings can only be achieved through routine control of the crystal orientation and avoidance of defects such as stray grains. Relaxation of the quality specifications has design implications, while stringent quality control can reduce yield with obvious economic consequences. Solidification of SX components invariably occurs dendritically; understanding the characteristics of dendrite growth in these complex materials is required for effective quality control. A range of defects observed in SX castings are considered, including: (i)Sensitivity of range of orientations produced to alloy chemistry and solidification rate, (ii)Effect of changes in mould cross-section on dendrite growth and (iii)Origins of stray grains during seeding of specific crystal orientations. The research has involved experimental studies by both controlled/quenched directional solidification and observation of industrial castings in conjunction detailed microstructural characterization. These have been complemented by novel multiscale modeling of solidification processing.

10:50 AM Invited
Solidification in Spray Forming: *Patrick Grant*[1]; [1]University of Oxford

Initially regarded as a rapid solidification process for the manufacture of small near-net shapes, spray forming has found commercial success in the production of specialized billet materials on a semi-continuous basis. In this arrangement, the spray forming solidification conditions comprise two distinct regimes: in-flight rapid solidification of a substantial fraction of the alloy droplets; and much slower solidification of the residual liquid in the billet once droplets/particles are reconstituted at deposition. This paper will focus on the events immediately prior, during and after deposition and show how the resulting solidification conditions lead to the characteristic spray formed microstructure comprising equiaxed grains with

low levels of microsegretation, as well as the possibility for the onset of macrosegregation and porosity. The use of non-intrusive process diagnostics and process modelling to reveal critical process physics will be described, and specific examples will include spray formed Si-Al alloys and ultra lightweight aerospace alloys.

11:15 AM Invited
Quest for a New Hot Tearing Criterion: *Laurens Katgerman*[1]; Dmitry G. Eskin[2]; [1]Delft University of Technology; [2]Netherlands Institute for Metals Research

Hot tearing remains a major problem of casting technology despite decades-long efforts to develop working hot tearing criteria and to implement those into casting process computer simulation. Existing models allow one to calculate the stress–strain situation in a casting (ingot, billet) and to compare it with the chosen hot tearing criterion. In most successful cases, the simulation shows the relative probability of hot tearing and the sensitivity of this probability to such process parameters as casting speed, casting dimensions, and casting recipe. None of the existing criteria, however, cannot give the quantitative answer on whether the hot crack will appear or not and on the extent of hot cracking (position, length, shape). This paper outlines the requirements for a modern hot tearing criterion as well as the future development of hot tearing research in terms of mechanisms of hot crack nucleation and propagation.

11:40 AM
A Tensor Phase Field Approach to Crystallization with Moving Solids: *Adam C. Powell*[1]; Jorge Vieyra[1]; [1]Massachusetts Institute of Technology

The Phase Field method can model crystal solidification in two and three dimensions by representing order parameter and orientation in multiple fields. For example, Kobayashi and Warren use an SO(3) tensor field to describe orientation in three dimensions; this tensor can be thought of as three orthogonal unit vectors representing directions of the local crystalline axes. Here the constraints of orthogonality and unit length are relaxed to allow this tensor to store local elastic strain information in addition to orientation. This information can provide for coupling between mechanics and phase for example giving complex behavior at a crack tip, or can provide the strain field used in the Mixed Stress method for fluid-structure interactions during solidification with moving elastic solids.

12:05 PM
Thermo-Mechanical Effects and Microstructure Formation near the Meniscus during Continuous Casting of Steels: Joydeep Sengupta[1]; *Brian G. Thomas*[1]; [1]University of Illinois

Initial solidification is significantly affected by local changes in heat transfer, fluid flow, pressure forces, and thermal stresses that arise near the liquid steel meniscus during mold oscillation in the continuous casting of steel. These phenomena govern both the mechanical behavior of the initial shell and the sub-surface microstructure. They lead to the formation of periodic oscillation marks and hooks, which are detrimental to the final surface quality. To investigate the formation of these defects, this paper first uses a transient finite-element model (CON2D) to compute temperature, stress development, and the shape of the distorted steel shell during the initial stages of solidification. The model incorporates temperature-dependent properties, phase transformations, thermal shrinkage effects and a creep-type elastic-viscoplastic constitutive equation. The effect of steel grade on the initial shell shape during sudden metal level fluctuations near the meniscus is investigated.

Surfaces and Interfaces in Nanostructured Materials II: Nanoscale Powders, Tubes and Composites

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

Wednesday AM
March 15, 2006
Room: 209
Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Arvind Agarwal, Florida International University

8:30 AM
Advanced FEM-Based Computational Modeling of CNT-Reinforced Damping Composite Materials: *Liya Bochkareva*[1]; Vladimir Kompis[2]; [1]NAS of Belarus; [2]University of Zilina

The focus in this paper is directed toward to the investigation into carbon-based nanoparti-cle/fibre/tube-reinforced composite materials and their advanced computational FEM-based dy-namic/damping characterization. Structural micro to nanomechanical approach has been developed to predict damping behavior of the CNT-reinforced polymer matrix material. The model is based on interface motion to address the damping characteristics of CNT-reinforced composite material. It is worth noting that SWNT can be represented as a shell hollow frame-like structure with a simple nanoscale damping spring characteristics so as to enable multiscale modelling and validation of the assembled computational workbench. Experiments are also performed to investigate the damping characteristics of the CNT-reinforced composite materials and validate the model predictions as a function of a number of various factors, including the magnitude of critical bonding stress, nanotube weight ratio, structure deformation (strain), and CNT properties. Dr. Bochkareva pursues her research work under EU INTAS 2005-2007 fellowship Ref. Nr 04-83-3067.

8:50 AM Invited
Controlling and Modeling the Interphase in Polymer Nanocomposites: *Catherine Brinson*[1]; T. Ramanathan[1]; H. Liu[1]; [1]Northwestern University

Polymeric nanocomposites made by incorporating small amount of nanoscale inclusions into polymer matrices exhibit dramatic changes in thermomechanical properties over the pure polymers. Because the properties of the nanoscale fillers can be extraordinary, even small volume fractions can result in significant changes. Enhancing the effect is the extremely significant role that the interphase plays in these systems. Given the enormous surface to volume ratio for nanoparticles, the interphase volume fraction can dwarf that of the inclusions themselves. In this talk, experimental evidence of the existence of this interphase region are presented. We show that by properly controlled functionalization of the nanoscale inclusions, we can impact the properties of the interphase region and consequently control the properties of the nanocomposites. In conjunction with the experimental results, the viscoelastic behavior of multi-phase polymeric nanocomposites is modeled using a novel hybrid numerical-analytical modeling method that can effectively take into account the existence of the interphase region. This hierarchical modeling approach couples the finite element technique and micromechanical approach and operates at low computational cost. Comparison between experimental and modeling results is reported.

9:30 AM
Effect of Sintering on Thermally Sprayed Carbon Nanotube Reinforced Aluminum Nanocomposite: *Tapas Laha*[1]; Arvind Agarwal[1]; [1]Florida International University

Multi-walled carbon nanotube (MWCNT) reinforced hypereutectic Al-Si nanocomposites were synthesized by high velocity oxyfuel and plasma spray techniques. Post processing sintering of these nanocomposites at 400°C for different periods under inert atmosphere has been carried out for further consolidation. Scanning electron microscopy and quantitative

microscopy has been performed to study the microstructural alteration viz. change in size and volume fraction of pores and primary silicon particles in the nanocomposites. The effect of sintering for prolonged time has been investigated by microhardness testing and nanoindentation technique. The counter effect of grain growth and residual stress on microhardness has been critically studied. Interfacial structure between Al-Si alloy and CNT has been examined by transmission electron microscopy.

9:50 AM Invited
Interfaces and Properties of TiC/A-C:H Nano-Composite Coatings: *Jeff T. H. DeHosson*[1]; Yutao Pei[1]; Damiano Galvan[1]; [1]University of Groningen

TiC/a-C:H nanocomposite coatings, deposited with closed-field unbalanced magnetron sputtering, have been scrutinized with high-resolution transmission electron microscopy focusing on the role of the various heterophase interfaces. These coatings consist of 2-5 nm TiC nanocrystallites embedded in an amorphous hydrocarbon (a-C:H) matrix. The toughening of the nanocomposite coatings has been achieved effectively on two different scales, namely by restraining the formation of columns on a micro-scale and by manipulating the nanostructure on a nanoscale. The hardness (H) and elastic modulus (E) of the coatings are found to increase monotonically with increasing substrate bias whereas the H/E increases with C content. Tribo-tests confirm that the nanocomposite coatings possess superior self-lubrication effects with a coefficient of friction as low as 0.05 in ambient air and below 0.02 in dry air. Physical arguments are presented to explain the toughening mechanism and the ultra-low friction.

10:10 AM
Plasma Sprayed Aluminum Oxide–Carbon Nanotube Nanocomposite Coating: *Kantesh Balani*[1]; Arvind Agarwal[1]; [1]Florida International University

Al2O3 being ceramic is extremely brittle, hence incorporation of carbon nanotube (CNT) as reinforcement is utilized to study the fracture toughness of the composite. Plasma spraying is used to synthesize Al2O3-CNT nanocomposite coating. Phase identification is carried out using X-ray diffraction analysis and Raman Spectroscopy. Scanning electron microscopy has been employed to study dispersion and retention of CNT in the coating after plasma spraying. Preliminary mechanical properties of Al2O3-CNT nanocomposite coating is compared with Al2O3 coating without CNT reinforcement towards evaluating the effect of reinforcement.

10:30 AM
Preparation and Characterization of Alumina-Titania Composite Membranes on Alumina Substrates: Sandra Fernandes Quaresma[1]; Mei Sen[1]; Yang Juan[1]; *José Maria da Fonte Ferreira*[1]; [1]University of Aveiro

High porosity titania-alumina washcoatings were successfully deposited by dip-coating onto alumina substrates for the manufacture of honeycomb type-catalysts via sol-gel method. The thermal behavior of the as-prepared films was investigated by DTA, DTG. The evolution of crystalline phases after heat-treating at different temperatures (650ºC, 800ºC and 950ºC) was determined by XRD. The microstructural and morphology evolution of the membranes was investigated by SEM. It was found that the molar ratio of alumina-titania in the precursor sols plays a determinant role and membranes with high homogeneous distribution of pores were obtained from an alumina-titania molar ratio of 1:11. For this ratio, anatase was the only crystalline phase identified after heat treating up to 650ºC. Higher temperatures gave rise to the appearance other minor phases and to a decrease of porosity.

10:50 AM Invited
Modification of the Stiffness and Q-Factor of Micromechanical Structures Using Carbon Nanotubes: *Srinivas Tadigadapa*[1]; [1]Pennsylvania State University

High frequency mechanical resonators operating in the frequency range of 1GHz and higher are of great interest in RF applications. However, even using the torsional mode of operation, micron sized resonators are typically limited to a maximum frequency of ~1 GHz. Carbon nanotubes (CNT) have been measured to have very high axial modulus of elasticity ~1 TPa. Incorporation of the high modulus CNT into MEMS thin films is expected to improve the elastic properties of deposited thin films. By incorporating CNTs in MEMS structures we have observed an increase in the stiffness of the micromechanical structures. Further, implicit in the miniaturization of mechanical resonators is the maintenance of Q-factor of the resonator necessary for achieving the required frequency stability. We also report the observation of the increase in the quality factor of an AT-cut quartz resonator through deposition of thin layers of SWNTs on its electrodes.

11:30 AM
Behavior of Impurity Elements in Powder Aluminium: *Sergey Lipko*[1]; Vladimir Tauson[2]; Vladlen Akimov[2]; Vecheslav Veselkov[1]; Boris Zelberg[1]; [1]Siberian Research and Design Institute for Aluminium and Electrode Industry JSC; [2]Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences

It is considered the influence of various chemical components on powder aluminium composition, produced using the method of gas dispersion of aluminium melt in nitrogen atmosphere and then annealed in different conditions. Powders with particles of different size 5-7 and about 20 mkm were examined. The presence of W makes better the structure of a film and promotes the formation of less strained nitride coating that was seen during comparative analysis of XPS width, pick no. 1s at half of maximum height after aluminium powder annealing in the presence of W in a sealed test-tube with air. It is anticipated that W has catalytic effect promoting the formation of nitric radicals that interact with particles' surface. W belongs to the group of elements with effect of integral accumulation in fine fractions of spray like Zn, Zr and some others.

The Brandon Symposium: Advanced Materials and Characterization: Small Length-Scales and Microstructures

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

Wednesday AM — Room: 206B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Sheldon Wiederhorn, National Institute of Standards and Technology; Xiaoqing Pan, University of Michigan

8:30 AM Invited
Electron Microscopy: Does it Solve Nano-Materials Problems?: *Jeff T. DeHosson*[1]; [1]University of Groningen

The actual linkage between the microstructure studied by microscopy on one hand and the physical property of a material is almost elusive. The reason is that various physical properties are determined by the collective dynamical behavior of defects rather than by the behavior of an individual static defect. We argue that for a more quantitative evaluation of the structure-property relationship of (nano)structured materials extra emphasis on in-situ measurements is necessary and that will be the topic of this contribution to honor David Brandon. These points will be illustrated with our recent work on nanostructured metal clusters. Various in-situ TEM observations as a function of temperature appeared to be in contrast with classical thermodynamics. In addition, it will be shown that dynamical properties, mechanical as well as magnetic, can be studied with in-situ TEM indentations and with electron holography.

8:55 AM Invited
Predicting Interface-Limited Growth Morphologies with Minimal Experimental Input: *David J. Srolovitz*[1]; Danxu Du[1]; [1]Princeton University

Interesting and useful growth morphologies are routinely observed in covalently and ionically-bound materials. In the limit, where these morphologies are dictated by attachment or reaction kinetics rather than dif-

fusional fields, these morphologies can be predicted from knowledge of the growth velocity as a function of surface normal. Convexification of such a velocity plot yields an asymptotic growth shape known variously as the kinetic Wulff shape or idiomorph. Unfortunately, such velocity plots are rarely known. In this talk, we discuss how to deduce sufficient features of the velocity plot from observations of selected area growth morphologies. We then present an efficient numerical approach that uses such input to predict both asymptotic as well as transient growth morphologies. As an example, we will focus on GaN islands grown using the epitaxial lateral overgrowth technique.

9:20 AM Invited
Superfast Densification by Spark Plasma Sintering of Nanocrystalline Oxide Powders: *Rachman Chaim*[1]; [1]Technion - Israel Institute of Technology

Spark plasma sintering (SPS) is a newly discovered old technique which recently has been used for superfast densification of ceramic powders. Simultaneous application of pulsed DC high current densities and load is the necessary condition for full densification by SPS. Commercial nanocrystalline MgO and yttrium aluminum garnet (YAG) powders were densified to optical transparency using spark plasma sintering at distinctly different homologous temperatures (0.3 for nc-MgO versus 0.7 for nc-YAG). The microstructure and density evolutions versus the SPS parameters were characterized and determined. Although a debate still exists about the exact mechanisms for the enhanced densification rate, an existing hot-isostatic pressing (HIP) model with conventional densification mechanisms was successfully used for the description of the densification of nanocrystalline MgO. Theoretical and experimental results both emphasize the importance of the ultrafine powder character as well as its mechanical and physical properties at high temperatures.

9:45 AM Invited
Tailoring of Multiferroic Nanostructures in Epitaxial Films: *Igor Levin*[1]; [1]National Institute of Standards and Technology

Multiferroic materials which display a coexistence of ferroelectric (FE) and ferromagnetic (FM) responses attract particular interest because of their potential for several novel applications. The present study combined experimental and theoretical work to determine the transferable principles of engineering of self-assembled multiferroic heterophase nanostructures in epitaxial films. The study was conducted on the nanostructures consisting of the epitaxial ferroelectric PbTiO3 and ferrimagnetic CoFe2O4 phases on differently oriented SrTiO3 substrates. Regardless of substrate orientation, the nanostructures consisted of vertical columns of CoFe2O4 dispersed in the PbTiO3 matrix, or vice versa. However, the morphologies of these columns, and their spatial arrangements, exhibited a marked dependence on the substrate orientation. Phase-field modeling of these nanostructures succeeded in reproducing even fine morphological details and revealed that the nanostructure morphologies are determined by the in-plane elastic anisotropy of the films. The implications of these results for a design of multifunctional epitaxial nanostructures will be discussed.

10:10 AM
Nanostructure and Mechanical Properties of Precipitation-Strengthened Al-Sc Alloys with Rare Earth Additions: *Marsha E. Van Dalen*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University

Currently, most precipitation-strengthened aluminum alloys are limited to usage at relatively low temperatures (<460 K), because of the rapid coarsening and/or dissolution of their precipitates. Al-Sc alloys are an exception, because they contain nanometer diameter, coherent Al_3Sc precipitates ($L1_2$ structure) with relatively low coarsening rates. Rare earth (RE) elements (Gd or Yb) are being added to Al-Sc alloys as ternary additions and are found to increase the hardness of the alloys by a factor of three over binary Al-Sc alloys. Transmission electron microscopy and atom-probe tomography are utilized to analyze the morphology and coarsening kinetics of the precipitates. The heterophase interface between a-Al and Al_3 ($Sc_{1-x}Yb_x$) exhibited segregation of Sc. The resulting creep properties of Al-Sc-RE alloys are also presented.

10:25 AM Break

10:35 AM Invited
Some Structure-Property-Function Connections for Healthy and Diseased Human Red Blood Cells: John Mills[1]; Ming Dao[1]; *Subra Suresh*[1]; [1]Massachusetts Institute of Technology

Changes to the biomolecular structure and cytoskeleton network of human red blood cells due to various disease states can result in significant changes in mechanical, chemical and biological responses. In this work, we explore some such connections for human red blood cells where structural changes arise from either infectious or hereditary diseases. We use biorheological measurements and force-displacement responses to characterize progressive changes in mechanical response due to the diseased state. These experiments are supplemented with detailed computational simulations so as to develop mechanistic insights into the changes in properties with specific references to red blood cells and human diseases.

11:00 AM Invited
Characterization of Ceramics Using Luminescence and Piezospectroscopy: *David Clarke*[1]; [1]University of California

Because most ceramic materials are transparent in the visible portion of the spectrum, optical tools, such as luminescence and spectroscopy, can be used in their characterization. In this talk I will describe two principal tools, the characterization of local stresses using piezospectroscopy and the characterization of temperature in doped zirconia coatings using luminescence. Piezospectrocopy involves the determination of strain from the frequency shift, and broadening, of characteristic spectral lines excited by either a laser beam or electron beam. The technique is particularly useful for studying alumina ceramics as chromium, a well-known dopant in alumina, occurs naturally in all alumina ceramics. The basic principles and applications will be described. In the second part of the talk I will describe recent work in doping zirconia, and related ceramics, to facilitate photo-stimulated luminescence and its use in non-contact measurement of temperature in coatings. So far, temperatures up to 1250°C have been measured.

11:25 AM Invited
Twin Quintuplets in CVD Diamond Films: *Dan Shechtman*[1]; [1]Ames Laboratory

Five fold twins have been observed in a large number of crystals, and in nanoparticles in particular. However, these twin quintuplets have been rarely thoroughly studied. Presented here is a crystallographic study of the formation of twin quintuplets in CVD diamond crystals. The quintuplets form during the initial growth process of the film, and at the onset of nucleation crystals with icosahedral shape are often created. The shape of some other CVD diamond crystals is also related to twin quintuplets. Observations by high resolution TEM and surface studies by SEM identify the surface crystallography of the diamond film. Twin quintuplets may consist of four S=3 and one Σ=81 boundaries, but in some cases they consist of only three Σ=3 and two general grain boundaries.

11:50 AM Invited
New Dimensions in Structural Metallurgy: *Srinivasa Ranganathan*[1]; [1]Indian Institute of Science

Metals are characterized by the metallic bond based on free electrons. This leads to a few close packed structures. The rich variety of atomic configurations seen in the larger class of inorganic materials with covalent and ionic bonding has long remained the envy of metallurgists. However, over the past few decades non-equilibrium processing has led to new configurations of quasicrystalline and noncrystalline phases, rivalling in complexity other inorganic structures. These studies have also revealed deep connections between the structure of intermetallics and clathrates and even biological materials with helices. The icosahedral order linking many of these structures will be emphasised.

12:15 PM
Finite Size Effects on Grain Boundary Structures and Interactions with Dislocations: *Emmanuelle A. Marquis*[1]; John C. Hamilton[1]; Douglas L. Medlin[1]; Francois Leonard[1]; [1]Sandia National Laboratories

Grain boundary structures in nanocrystalline materials, which by deviating from their ideal structures in infinitely long bicrystals, may impact

the operating deformation mechanisms. We examine the relaxation of Σ3 {112} grain boundary structures of nanometer length scale, and address the interactions with dislocation focusing on the forces and interactions that drive the emission of extended defect structures from grain boundaries. This issue has become increasingly important in nanostructured materials in which distances between interfaces are sufficiently small that individual interfaces cannot be considered in isolation. Of broader significance, simulations and experiments in the literature have indicated that the emission of extended defects plays a role in the deformation of nanocrystalline materials. The detailed connection of such emission processes to the properties and structure of the emitting interfaces, however, remains unclear. This work points out the relative importance of interfacial and elastic energies in controlling the equilibrium structures of such configurations.

The James Morris Honorary Symposium on Aluminum Wrought Products for Automotive, Packaging, and Other Applications: Continuous Casting and Related Technologies

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

Wednesday AM Room: 207A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Subodh K. Das, Secat Inc; Zhong Li, Aleris International Inc

8:30 AM
Texture Evolution of Continuous Casting AA5052 Aluminum Alloy Sheet during Closed to Equi-Biaxial Stretching: *Xiyu Wen*[1]; Zhengdong Long[1]; Weimin Yin[2]; Tongguang Zhai[1]; Zhong Li[3]; Subodh Das[2]; [1]University of Kentucky; [2]Secat, Inc.; [3]Aleris International, Inc

In this study, texture evolution in a 0.080" gauge hotband of continuous casting AA5052 aluminum alloy sheet after annealed at 750°F for 4 hours was investigated by X-ray through thickness of the sheet. In the three different strain levels from equi-biaxial stretching, the deformed samples were prepared for texture measuring. The major and minor strains were measured. The texture evolution in the different layers through thickness of the three sampless was obtained during the closed to equi-biaxial strain state. It was found that cube component rotates to Goss orientation ((110)<001>) during equi-biaxial deformation. The Goss component gradually changes to Brass orientation ((110)<112>) and penetrates to (110)<hhl> position. The part of Brass component also rotates to S orientation ((213)<364>). In addition, the (110)<111> component was found on the surface.

8:55 AM Invited
Quantitative Analysis of Texture Evolution of Aluminum Alloys during Cold Rolling: A Review: *Wenchang Liu*[1]; Zhong Li[2]; Chi-Sing Man[1]; James G. Morris[1]; [1]University of Kentucky; [2]Aleris International Inc.

The texture evolution of various aluminum alloys during cold rolling was investigated by X-ray diffraction. The rotation paths and stability of the cube and rotated cube orientations were determined based on the variation in the three-dimensional orientation distribution function (ODF) with rolling reduction. Texture volume fractions were calculated by an improved integration method. The relationship between the texture volume fractions and true strain was described quantitatively by mathematical formulae. The effect of alloy composition, initial microstructure and texture, and processing method (CC vs. DC) on the texture evolution during rolling was determined.

9:20 AM
Microstructure, Crystallographic Texture, and Plastic Anisotropy of a Continuous Cast Al-Mn-Mg Alloy Sheet: *Jiantao Liu*[1]; Robert E. Dick[1]; Thomas N. Rouns[1]; Stephen W. Banovic[2]; Richard J. Fields[2]; James G. Morris[3]; [1]Alcoa Technical Center; [2]National Institute of Standard and Technology; [3]University of Kentucky

The microstructure, crystallographic texture, and plastic anisotropy of an continuous cast Al-Mn-Mg alloy sheet was investigated. It was found that the cold rolling reduction and initial heat-treatment of hot rolled sheet had a significant effect on the microstructure and mechanical properties of the sheet, respectively. For the sheet specimens without the initial heat-treatment, a severely elongated grain structure was found in which the texture was dominated by a strong P orientation {011}<566> regardless of the fact that the specimen was completely recrystallized. In contrast, specimens receiving the same cold rolling and annealing conditions but with the initial heat-treatment had an equiaxed grain structure with a Cube orientation {001}<100>. The R values were predicted using Hosford-Backofen model as well as continuum mechanics of texture polycrystals (CMTP) method. The predicted R values were compared with the measured results. The effect of initial heat-treatment on the plastic anisotropy of the specimens was discussed.

9:45 AM Invited
Analysis of Deformed Microstructures in AA5005 and AA6022: *David P. Field*[1]; Reza S. Yassar[1]; [1]Washington State University

Dislocation generation and motion is the primary mechanism of plastic deformation in polycrystalline materials. Strain hardening of these materials is a function of dislocation interactions with one another, and with various additional obstacles or defects in the crystallite. Measurement of the local plastic strain state requires analysis of the dislocation structure. A non-unique correlation between the lattice curvature and plastic strain in polycrystalline aluminum is demonstrated using a variety of microstructural measures. Measurements in aluminum alloys 5005 and 6022 deformed by channel die compression are discussed in the context of structural evolution as measured by lattice curvature and geometrically necessary dislocations. Evolution of the dislocation structures is assumed to be dependent on Taylor factors of individual grains, but little evidence to support this supposition is found. It is shown that non-local factors, such as interactions with neighboring grains, are of importance in describing structural evolution.

10:10 AM Break

10:20 AM
Quantitative Texture Evolution in a Continuous Cast AA5052 Aluminum Alloy during Hot Rolling: *Qiang Zeng*[1]; Tony Zhai[1]; Xiyu Wen[1]; [1]University of Kentucky

In order to investigate the influence of hot rolling on the texture and microstructure evolution in commercial continuous cast Aluminum alloys, a continuous cast (CC) AA5052 slab bitten by the first hot rolling mill at an entry temperature of 465°C and an exit temperature of 370°C was made. The rolling reduction in thickness varied from 8.6 mm to 21.5 mm across the sample along the rolling direction. Textures were measured by XRD at cross-sections perpendicular to the rolling direction in this sample. The evolution of all the texture components during hot rolling was quantified using Johnson-Mehl-Avrami-Kolmogorov type equations in terms of the rolling true strain. Orientation hardening during hot rolling was also calculated based on the texture measurement in the alloy. The microstructure evolution was studied in the sample.

10:45 AM
Thermal Stability of Selected 5000 Series Al Alloys: *Catherine Wong*[1]; Alicia Field[1]; [1]NSWCCD

Due to the propensity of the 5000 series Al alloys containing more than 3% Mg to sensitize it was undertaken to empirically produce the time temperature curve for sensitization of three common alloys. The alloys studied included 5383, 5454 and 5456. They were heated above 100°F and below 400°F for up to 80 days and the degree of sensitization was measured using ASTM G 66 and ASTM G 67. Image analysis was employed to form a relationship between the resulting volume fraction of the beta phase and the weight loss in recrystallized 0.25" plate of these three alloys.

11:10 AM
Heating Rate Effect on Microstructure Evolution during Annealing of Twin Roll Cast AA3105: *Naiyu Sun*[1]; Burton R. Patterson[1]; Jaakko P.

Suni[2]; Eider A. Simielli[2]; Hasso Weiland[2]; Puja Kadolkar[3]; Craig Alan Blue[3]; Gregory B. Thompson[1]; [1]University of Alabama; [2]Alcoa Inc; [3]Oak Ridge National Laboratory

Rapid solidification and cooling of twin roll cast (TRC) AA3105 traps excess solute that precipitates as fine dispersoids during conventional annealing. These particles inhibit recrystallization by Zener pinning of subgrain boundaries, resulting in a coarse, elongated grain structure. Prior studies have shown that a rapid heating rate to the annealing temperature produces a much finer and more equiaxed recrystallized grain structure. The present study examines the effects of three constant heating rates on the resulting precipitation and recrystallization behavior. The two highest rates, 50 and 3°C/s, were achieved in an infrared furnace at the ORNL Materials Processing Lab, while the slowest rate, 0.01°C/s, was obtained in a conventional programmable lab furnace. The time-temperature-transformation diagrams for dispersoid precipitation and recrystallization were determined via conductivity measurements and optical microscopy. The relative positions of these curves were used to explain the effects of heating rate on recrystallization kinetics and grain size.

11:35 AM

Effect of Texture and Second Phase Particle Distribution on Formability of Continuous Cast and Direct Chill Cast 5754 Al-Mg Sheet: Asim Tewari[1]; Shashank Tiwari[1]; *Raja Mishra*[1]; Anil Sachdev[1]; [1]General Motors

Continuous casting (CC) offers an opportunity to decrease the cost of sheet aluminum, however, the formability of CC sheets is slightly lower than sheet made from the Direct Chill cast (DC) process. A systematic study of the texture and microstructure in DC and CC 5754 aluminum sheet was performed using electron back scattered diffraction (EBSD) and digital imaging. Stereological tools were employed to characterize the second phase particles in three dimensions, and unbiased robust values of particle size, particle fraction, interfacial-area and mean free path were estimated. Differences in the size distribution and spatial scatter were also quantified. It is seen that the random components of texture are nearly identical in DC and CC sheet, but individual FCC rolling components differ from one material to the other. It is shown that differences in texture and spatial arrangements of particles correlates with differences in the ductility and fracture properties observed.

12:00 PM

Application of Rapid Infrared Heating for Processing Aluminum Forgings: *Gowreesan Vamadevan*[1]; Frank F. Kraft[1]; Puja Kadolkar[2]; Howard (Rob) Mayer[3]; [1]Ohio University; [2]Oak Ridge National Laboratory; [3]Queen City Forging Company

Infrared (IR) heating has the potential to be used for solutionizing of aluminum forgings with benefits of reduced energy consumption, and improved microstructure and mechanical properties. Standard procedures to take advantage of rapid IR for solutionizing are not currently available. Thus, a primary objective of this work was to determine optimum solutionizing cycles for four aluminum alloys; AA 2014, AA 2618, AA 6061 and AA 7075. A second objective was to demonstrate the mechanical property improvements possible with optimized rapid IR solutionizing cycles. Laboratory experiments on aluminum coupons were performed to establish optimum solutionizing thermal cycles for each alloy. Upset forgings were then produced via conventional production means and "optimized" IR thermal processing. The results of lab and production testing are presented, and the microsturcture and mechanical properties of conventionally heat treated parts and rapid IR treated parts are compared.

The Rohatgi Honorary Symposium on Solidification Processing of Metal Matrix Composites: Modeling and Nanocomposites

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

Wednesday AM — Room: 207B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Daniel B. Miracle, U.S. Air Force; Anil Kumar Gupta, National Physical Laboratory

8:30 AM Invited

Microstructure-Based Modeling of Particle Reinforced Metal Matrix Composites: *Nikhilesh Chawla*[1]; Krishan K. Chawla[2]; [1]Arizona State University; [2]University of Alabama at Birmingham

It is well recognized that microstructure controls the physical and mechanical properties of a material. Several analytical and numerical techniques have been employed extensively to predict and characterize the behavior of multiphase materials. Analytical models provide reasonable predictions for relatively simple configuration of the phases. Numerical models can extend the realm of problems that can be modeled, but they still make some simplifying assumptions about the inherent microstructure of heterogeneous multiphase materials. Microstructure-based modeling of particle reinforced metal matrix composites (MMCs) involves use of microstructural data in finite element modeling, in two and three dimensions, with a view to predict the properties such as elastic, plastic, or thermal behavior of the composite. It can also be used as a tool to understand the mechanical and physical behavior of the composite. We provide examples from important particulate MMCs such as SiC/Al and WC/Co.

8:55 AM

Measurement and Modeling of Damage in Cast Al-Si Alloys: B. Ye[1]; M. Erukullu[1]; Stephen J. Harris[2]; Somnath Ghosh[3]; *Bhaskar S. Majumdar*[1]; [1]New Mexico Tech; [2]Ford Motor Company; [3]Ohio State University

In this presentation, we report on damage mechanisms and ductility prediction of cast A356 and A319 Al-Si alloys. These materials may indeed be considered a subset of cast metal matrix composites, which were pioneered by Professor Rohatgi approximately 4 decades ago. Our observations indicate that although the Al-Si alloys were Sr-modified, the brittle Si-particle shapes have complex 3-D morphological features that are generally not known in such alloys. Tensile specimens were incrementally loaded to different strains, and microstructurally examined to measure particle cracking and matrix damage. In addition, neutron and Raman-shift measurements were conducted to estimate stresses in Si particles. Analytical and computational modeling involved homogenization methods to include particle cluster effects. Results of predicted particle stresses are compared with measured data, and our attempts to predict ductility in such systems are discussed. We acknowledge support from NSF contract CMS 0309519 for conducting this research.

9:20 AM

Numerical Modeling of Interaction of Particles with Solidifying Interfaces: *Mario Roberto Rosenberger*[1]; Eliana Mabel Agaliotis[1]; Carlos Enrique Schvezov[1]; [1]National University of Misiones

The thermal fields of a moving solidification interface towards a spherical particle were dynamically modeled in order to study the deformation of the interface in relation with different thermal properties of particle and matrix. Finite element methods were employed in an axi-symmetric model of the system. The degree of deformation when the particle has different thermal conductivity than the matrix is presented and discussed. The drag force on a particle being pushed by a crystal was calculated with a fluid flow model. The force was calculated for different pushing configurations and the results compared with the values given by the modified Stokes equation; which show that the model value are slightly larger than those

calculated with the equation. This difference predicts an equilibrium separation for pushing lower than the computed by modified Stokes equation when a Lifshitz-Van der Waals model for the repulsion force is used.

9:45 AM
Multiscale Approach to Modeling Particle-Solidification Front Interactions: *Justin W. Garvin*[1]; Yi Yang[1]; H. S. Udaykumar[1]; [1]University of Iowa

Predicting microstructures of metal-matrix composites (MMC) requires an understanding of how solidification fronts interact with particles. The particle-solidification front interaction is a multiscale process as the dynamics at the micro-scale hinges on the interactions between the front and the particle, which occurs across a nano-scale gap. In these systems, the solution of the Navier-Stokes equations including the nano-scale gap between their interacting surfaces would be impossible due to resolution demands placed on the mesh. Therefore an embedded model for solution in the gap is needed. This model takes the form of a lubrication equation with disjoining pressure acting as a body force and is coupled to the solution outside the gap that is solved using the Navier-Stokes equations. A sharp-interface method is used to track the interfaces. This method illustrates a new way of predicting the critical velocity and allows for an in-depth analysis of the physics that cause particle pushing/engulfment.

10:10 AM
Influence of Processing Parameters on the Structure and Mechanical Properties of Lightweight Aluminum and Magnesium-Nanoparticle Metal Matrix Nanocomposites: Benjamin F. Schultz[1]; *Pradeep Kumar Rohatgi*[1]; J. B. Ferguson[1]; Nikhil Gupta[2]; [1]University of Wisconsin-Milwaukee Center for Composites; [2]Brooklyn Polytechnic

Metal matrix nanocomposites (MMNC) reinforced with dispersed nanosized ceramics have the potential of ultra high strength and improved tribological properties. To date, most of the developments and research in the synthesis of metal matrix nanocomposites have focused on powder metallurgy techniques and deformation processing. Researchers at the University of Wisconsin have developed cast lightweight aluminum and magnesium MMNC's through solidification processing techniques. Applications and optimization of vortex mixing, ultrasonic agitation and squeeze infiltration processes to produce lightweight metal matrix nanocomposites are discussed. The synthesis parameters were varied and analyzed using statistical design of experiments. Selected mechanical and physical properties and microstructures of cast metal matrix nanocomposites will be presented.

10:35 AM Break

10:50 AM
Sharp Interface Simulation of Interactions of Dendrites with Solid Particles: *Yi Yang*[1]; J. W. Garvin[1]; H. S. Udaykumar[1]; [1]University of Iowa

The behaviour of growing dendrites when approaching particles in the melt determines the particle distribution in the finished metal-matrix composites(MMCs). A sharp interface level-set based numerical method is employed to study dendrite-particle interactions. The simulation of the interaction between particles and dendrites grown from pure material shows that for a particle to melt thermal conductivity ratio $\lambda=k_p/k_l<1$(typical for MMCs), the dendrite doesnot approach the particle close enough to activate particle pushing. Instead, the dendrite chooses to go around the particle and eventually the particle is engulfed by sidebranches. Thus the entrapment mode is the likely outcome. The simulation of interaction of particles with dendrites grown from a binary alloy is also carried out. A local mesh refinement technique is used along with the sharp-interface methodology to enable the simulation of interactions of multiple dendrites and multiple particles, so the prediction of the particle distribution in MMC microstructures is also attempted.

11:15 AM Invited
Polymeric Route for Processing NanoScale Aluminum Matrix Ceramic Composites for High Temperature Applications: Scott Patrick[1]; Atanu Saha[1]; *Rishi Raj*[1]; [1]University of Colorado

Recent research on polymer-derived ceramics whereby ultrahigh temperature silicon-carbonitride materials are made by controlled pyrolysis of highly cross-linked polysilazane polymers is extended to the insertion of the ceramic phase into an aluminum matrix by in-situ pyrolysis. Since the pyrolysis temperature lies above the melting point of aluminum, the polymer is introduced into the aluminum melt and then pyrolyzed in the liquid metal environment near 1000°C. The results show that in-situ pyrolysis is viable and that it leads to enhanced mechanical properties (the experiments are being done with pure aluminum). The mechanical properties do not degrade with elevated temperature anneals implying that the hard phase introduced by pyrolysis does not coarsen. This process raises new issues: the elimination of hydrogen from the melt, the fragmentation of the polymer particles during pyrolysis, and the constitution of the ceramic phases produced within the liquid metal and their adherence to the metal.

11:40 AM Invited
Nanostructured Ceramic Thin Films: Their Structures, Mechanical Properties, and Applications to Microfabrication: *Wen J. Meng*[1]; Dong Mei Cao[1]; Jing Jiang[1]; [1]Louisiana State University

Our recent results on two-phase nanocomposite thin films based on amorphous hydrogenated carbon (a-C:H) and amorphous silicon nitride (a-Si:N), synthesized by plasma assisted vapor phase deposition, will be summarized. Detailed nanoscale structural characterization was achieved by combining X-ray spectroscopy with high resolution electron microscopy. Mechanical properties of the films, such as modulus, hardness, and residual stress, were characterized with instrumented nanoindentation and substrate curvature measurements. Structure - mechanical property correlations will be discussed. Applications of nanostructured ceramic thin films to microscale molding replication of metal-based high-aspect-ratio microscale structures will be illustrated.

Titanium Alloys for High Temperature Applications - A Symposium Dedicated to the Memory of Dr. Martin Blackburn: Titanium Alloys for High Temperature Oxidation Resistance

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

Wednesday AM Room: 201
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Guido C. Keijzers, Del West USA; Yoji Kosaka, TIMET

8:30 AM Invited
Improvement of High Temperature Environmental Resistance of Titanium Alloys and Titanium Aluminides – Recent Trends: *Christoph Leyens*[1]; Reinhold Braun[1]; Maik Froehlich[1]; Olaf Schroeter[2]; [1]German Aerospace Center (DLR), Institute of Materials Research; [2]Technical University of Brandenburg at Cottbus

The use of high temperature titanium alloys and aluminides at moderately elevated and high service temperatures is a strong challenge to their environmental resistance. The paper will review the most recent trends in surface modification techniques for these light weight high temperature materials while focusing on the development of protective overlay coatings. Metallic coatings typically provide good oxidation resistance at high temperatures by the formation of protective oxide scales while the coatings themselves remaining "ductile". Nitride coatings can combine good oxidation resistance with high hardness, thus additionally providing protection against wear and erosion, however, typically at the expense of coatings adhesion and potential adverse effects on the fatigue behavior of the structural material. For highest operating temperatures the use of thermal barrier coatings on gamma titanium aluminides has been explored recently. Very promising results were achieved when zirconia top coatings were applied using the EB-PVD deposition technique.

9:00 AM
Recent Development of Titanium and Its Alloys in Automotive Exhaust Applications: *Yoji Kosaka*[1]; Stephen P. Fox[1]; [1]TIMET

There has been a significant growth in the application of titanium and its alloys to automotive exhaust systems over the last decade. CP Ti has been used for dual exhaust systems of Corvette Z06 in recent years. Exhaust pipes and mufflers for high performance motorcycles have been a prime target of titanium, since the visual appearance of titanium as well as performance with titanium attracts users. There has been notable progress in material side as well. Ti-1.5%Al and TIMETAL®Exhaust XT (Ti-0.45%Si-0.25%Fe) have been developed to meet more stringent requirement in terms of oxidation resistance at higher temperatures where CP Ti cannot be used. This paper will review high temperature titanium and its alloys for automotive exhaust applications. Metallurgical factors that control oxidation resistance will also be discussed primarily focusing on TIMETAL®Exhaust XT.

9:30 AM

Investigation of Ductility Loss in a Thermally Exposed Near-β Ti Alloy: *Frederic Sansoz*[1]; Hamouda Ghonem[2]; [1]University of Vermont; [2]University of Rhode Island

We studied the effects of thermal exposure in air on the plastic elongation of Ti-15Mo-2.7Nb-3Al-0.2Si (Timetal-21S) alloy. Sheet specimens (0.12-1.0 mm in thickness) were exposed in air to temperatures between 482°C and 693°C. Tensile tests conducted on these specimens at room temperature show a reduction of plastic elongation along with a change in the failure mode into a quasi-brittle fracture in the near-surface region. The kinetics of embrittlement is studied through theoretical considerations of gas diffusion into metal. This approach shows that two distinct embrittlement mechanisms operate in this alloy depending on the temperature range. Above 545°C, the embrittlement activation energy is 41.2 kcal.mol^{-1}, indicating that the embrittlement process is governed by an enhanced diffusion of oxygen into Timetal-21S. Below this transitional temperature, the activation energy approaches zero, a characteristic of slow kinetics transformation. The role of solid-solution hardening, precipitation-hardening mechanisms, and alloying-element partitioning on this effects are also discussed.

10:00 AM

Aluminum Clad Titanium for High Temperature Applications: *Lichun Leigh Chen*[1]; Yoji Kosaka[2]; Mike Hardy[1]; [1]Engineered Materials Solutions Inc; [2]TIMET Henderson Technical Laboratory

The addition of aluminum increases the oxidation resistance of titanium at elevated temperatures. There is interest in cladding aluminum to titanium or titanium alloys for elevated temperature applications. A study was conducted to roll-bond aluminum sheet over Ti substrate to form Al/Ti clad metal and then to anneal the clad metal for diffusion-alloying. TIMETAL Exhaust-XT was selected as a Ti substrate. Three different thicknesses of aluminum were used to make Al/Ti clad metals. The processes were investigated for ideal microstructure and properties. The metallurgical views behind the process design are discussed. Microstructural examination revealed the effects of the annealing temperature on microstructure evolutions. Aluminum-titanium intermetallic compounds were formed during diffusion-annealing. Ti was well diffused into the original Al surface clad layer while the diffusion of Al into the Ti substrate was insignificant. Mechanical testing showed the optimal processing for forming. Oxidation weight gain at 800°C followed the parabolic law.

10:30 AM Break

Titanium Alloys for High Temperature Applications - A Symposium Dedicated to the Memory of Dr. Martin Blackburn: Titanium Based Intermetallic Alloys for High Temperature Applications - Alpha 2 and Orthorhombic

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

Wednesday AM Room: 201
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Patrick L. Martin, U.S. Air Force; John J. Schirra, Pratt & Whitney

11:00 AM Invited

The Emergence of the Orthorhombic Aluminides: *Dipankar Banerjee*[1]; [1]Defense Research and Development Organization

The development of aluminides based on the intermetallic phases in the Ti-Al system consumed almost three decades of intensive research through the seventies and eighties, and into the nineties. Martin Blackburn played a central role in this effort. The alloying of Ti3Al was examined in the sixties, but engineering plasticity in this class of alloys appears to have emerged from programs directed by Blackburn through the late seventies and eighties at the United technologies Research Centre. The Ti-24Al-11Nb and the supera2 alloy (with about 15at% beta stabilizers) emerged from this effort. In the late eighties, Rowe at GE,CR&D observed significantly improved properties in Ti3Al alloys containing Nb upto 25at %. Almost simultaneously, we discovered that alloys with Nb contents greater than about 15at% Nb were based on a ternary intermetallic, Ti2AlNb that we christened the O phase. In the following years we defined the physical metallurgy of the higher Nb alloys covering phase equilibria and transformations, mechanical behavior and processing. In the mid nineties, working together with the School of Materiaux, Ecole des Mines and Snecma Moteurs, we significantly improved the properties of this class of alloys through extensive studies on compositional effects on processing, structure and properties. We summarize the totality of this effort in our presentation in a tribute to Martin Blackburn's pioneering work.

11:30 AM

Microstructure, Tensile, and Creep Behavior of Ti-15Al-33Nb and Ti-21Al-29Nb Orthorhombic+BCC Alloys: *Christopher J. Cowen*[1]; Carl J. Boehlert[1]; [1]Michigan State University

In this work the creep and elevated temperature tensile behavior of two orthorhombic (O)+body-centered-cubic (BCC) alloys, specifically Ti-15Al-33Nb (at%) and Ti-21Al-29Nb (at%), were evaluated in order to determine the effect of alloy composition and microstructure on mechanical properties and deformation behavior. Tensile tests, performed at RT and 650°C, indicated that the Ti-21Al-29Nb alloy exhibited significantly lower elongation-to-failure values than the Ti-15Al-33Nb alloy. Heat-treatments used to precipitate greater volume fractions of the O-phase resulted in greater strength levels, while the BCC phase provided ductility. The measured creep stress exponents ($1.1<n<6$) and apparent activation energies (99 kJ/mol<Qapp< 317 kJ/mol) suggested that different mechanisms were active dependent on temperature and applied stress level. Overall, the Ti-15Al-33Nb alloy was shown to exhibit a greater balance of RT and elevated-temperature properties than the Ti-21Al-29Nb alloy, and this presentation will discuss the implications of this result in terms of microstructure-property relationships for O+BCC alloys.

12:00 PM

Property Enhancement of Ti_2AlNb-Based Intermetallic Alloys for High Temperature Use: *Satoshi Emura*[1]; Masuo Hagiwara[1]; [1]National Institute for Materials Science

In order to improve the mechanical properties of orthorhombic Ti_2AlNb-based titanium intermetallic alloys for temperatures above 650°C, we have been conducting following researches. To improve room temperature prop-

erties without sacrificing high temperature properties, grain-size refinement was attempted using the pinning effect of second phase particles. The finer-grained materials showed a good combination of room and high temperature properties. To improve tensile and creep strength at temperatures above 650°C, substitution of transition metal elements such as W, Mo, V, Fe or Cr for a portion of Nb was conducted. Substitution of W for Nb in a Ti-22Al-27Nb alloy substantially increased the high temperature tensile and creep strength. To further improve high temperature mechanical properties, fine TiB or TiC particulate reinforced Ti_2AlNb-based composite was produced using a gas atomization P/M method. Most of the mechanical properties such as tensile strength and creep properties were superior to those of the unreinforced matrix alloy.

12:30 PM
Oxidation Studies on Ti3Al-Mo Alloys: Ramana G. Reddy[1]; *Dusti Livingston*[1]; Divakar Mantha[1]; [1]University of Alabama

The Ti3Al-Mo oxidation studies were conducted in oxygen gas using TGA. Three alloys containing 1.7 at. %Mo, 2.2 at.% Mo and 4.0 at. %Mo was studied from 800°C to 1100°C. The effects of time, temperature, and composition affect on the oxidation of the alloys were investigated. For each given alloy, weight gain per unit surface area vs. time plot was made, and reaction rate constants were used to calculate the activation energies. Reaction products were characterized using optical microscope, scanning electron microscope (SEM), X-ray diffraction, and energy dispersive spectrometer (EDS). The results showed that alloys that contain Molybdenum were higher resist to oxidation than the binary alloys. The alloy that contains 2.2 at.% Mo has lower activation energy than other alloys containing 2.6 at.%Nb. A possible oxidation mechanism of the alloys in oxygen was proposed.

Ultrafine Grained Materials - Fourth International Symposium: Mechanical 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

Wednesday AM
March 15, 2006
Room: 217D
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Michael Josef Zehetbauer, University of Vienna; Jingtao Wang, Nanjing University of Science and Technology; Irene J. Beyerlein, Los Alamos National Laboratory; Hyoung Seop Kim, Chungnam National University

8:30 AM Invited
Effect of ECAP on the Mechanical Properties of Mg Alloys: *Yuri Estrin*[1]; Ralph J. Hellmig[1]; Milos Janecek[2]; Torbjoern T. Lamark[1]; Mikhail V. Popov[1]; [1]IWW, TU Clausthal; [2]Charles University

We present results on the effect of equal channel angular pressing (ECAP) on several Mg alloys, including AZ31, AS21X, and Mg-Ni. The enhancement of the yield strength and variations in ductility, as well as the effect on the fatigue life will be discussed. In addition, the influence of ECAP on the acoustic emission will be reported. The microstructures observed in ECAP-processed Mg alloys by TEM differ significantly from those in fcc metals. In the Mg alloys studied, a pronounced small grain structure was shown to develop 'precipitously', already after a single ECAP pass. A residual strain hardening capacity (albeit relatively small) associated with the microstructure produced provides sufficient – and sometimes enhanced – ductility of the ECAP-processed Mg alloys.

8:50 AM Invited
Optimization of Strength, Ductility and Properties of Ultra-Fine-Grained Copper with Nano-Scale Twins: Lei Lu[1]; Ming Dao[2]; *Subra Suresh*[2]; [1]Institute of Metal Research, Chinese Academy of Sciences; [2]Massachusetts Institute of Technology

Tensile and nanoindentation experiments at room temperature show the pulsed-electrodeposited copper samples with high density nanoscale twins have an ultrahigh strength (1.0 GPa) and considerable ductility. Moreover, significantly enhanced rate sensitivity of plastic flow and work hardening rate are also observed in these tests. With an increase in twin density (or a decrease in twin lamellar spacing), rate sensitivity, strength and ductility increase as well. The concept of Twin Boundary Affected Zone (TBAZ) is introduced into a physically-motivated crystal plasticity model to study strength, rate sensitivity and ductility. The orientation and size dependent plastic behavior parallel (plastically softer) and perpendicular (plastically harder) to the twin boundaries is specifically modeled. Parametric studies show that the proposed TBAZ model correctly captures the experimentally observed trend. Possible deformation and failure mechanisms are discussed. Strategies for the optimization of strength and ductility as well as of electrical and mechanical responses will also be addressed.

9:10 AM Invited
Enhanced Mechanical Properties in Ultrafine Grained 7075 Al Alloy: *Yonghao Zhao*[1]; Yuntian Zhu[1]; [1]Los Alamos National Laboratory

The high strength and high ductility for 7075 Al alloy were obtained by combining the equal-channel angular (ECAP) and natural aging. The tensile yield strength and ultimate strength of the ECAP processed and naturally aged sample were 103% and 35% higher, respectively, than those of the coarse-grained 7075 Al alloy counterpart. The tensile elongation to failure is about 12%. The enhanced strength resulted from high densities of the 2nd-phase particles (G-P zones and meta-stable phase) and dislocations. The origin of the high ductility of the ECAP processed and aged 7075 Al alloy will be discussed. This study shows that severe plastic deformation has the potential to significantly enhance the mechanical properties of precipitated hardening 7000 series Al alloys.

9:30 AM Invited
On Tension/Compression Asymmetry of an Extruded Nanocrystalline Al-Fe-Cr-Ti Alloy: *Leon L. Shaw*[1]; Hong Luo[1]; [1]University of Connecticut

The tension/compression asymmetry of nanocrystalline materials has been the subject of intensive study in recent years. In this study, a multiphase nanocrystalline Al93Fe3Cr2Ti2 alloy containing 30 vol.% intermetallic particles was prepared via mechanical alloying, followed by hot extrusion. Tensile and compressive tests at ambient and elevated temperatures were performed. The alloy exhibited significant difference in deformation behavior between tension and compression at 25°C, 200°C and 300°C. However, the strengths obtained in tension and compression were similar at 400°C. Systematic microstructure examinations and deformation mechanism analyses indicate that the asymmetry of this nc Al93Fe3Cr2Ti2 alloy is related to its dislocation mediated plastic deformation mechanism, its nanoscale grain microstructure, and premature brittle failure in tension tests.

9:50 AM
Fatigue and Fracture in Bimodal Al 5083: *Peter S. Pao*[1]; Harry N. Jones[1]; C. R. Feng[1]; David B. Witkin[2]; Enrique J. Lavernia[3]; [1]Naval Research Laboratory; [2]University of California at Irvine; [3]University of California at Davis

The fatigue crack growth and fracture resistance of ultrafine-grained (UFG) Al 5083 having a bimodal grain size were investigated. The bimodal Al 5083 was prepared by mixing ball-milled particulates with various amounts of larger grained powders and then extruded into rods. The bimodal Al 5083 thus produced consists of UFG grains and coarse grain bands. With increasing larger grained material, the yield strength of the bimodal Al 5083 is lowered progressively while its tensile ductility and fracture toughness are increased significantly. Fatigue crack growth rates of bimodal Al 5083 are lower than those of all-UFG Al 5083. The higher fatigue crack growth rates in the all-UFG Al 5083 may be attributed to the much smoother fracture surface and lower crack deflection. The fracture toughness and fatigue crack growth of the bimodal and UFG Al 5083 will be discussed in terms of the differences in underlying microstructure and deformation mechanisms.

10:05 AM

Microstructure and Deformation Behavior of Nc Pd: *Julia Ivanisenko*[1]; Juergen Markmann[2]; Harald Roesner[1]; Heinz Schiels[3]; Hans J. Fecht[3]; Ruslan Z. Valiev[4]; Jorg Weissmueller[1]; [1]Forschungszentrum Karlsruhe; [2]University of Saarlands; [3]University of Ulm; [4]UFA State Aviation Technical University

A novel method for the preparation of bulk nanocrystalline materials with a grain size <30 nm using the combination of inert gas condensation and subsequent high pressure torsion (hpt) was developed. Here we present results on a comprehensive investigation of the microstructure and mechanical properties of fully dense Pd with a mean grain size of 15 nm. After hpt consolidation, texture measurements have not revealed any preferential orientations, and HRTEM investigations demonstrated the presence of stacking faults inside the nanocrystalline grains. By the contrast, a pronounced texture and developed sub-granular dislocation structures were developed in the Pd specimens with a grain size of 75 nm processed in a similar way. These observations suggest that the operating deformation mechanisms in materials with a grain size of the order of 15 nm include emission of partial dislocations along with grain rotations instead of the slip of full lattice dislocations in coarse-grained materials.

10:20 AM Cancelled

The Effect of Grain Size on the Mechanical Behavior in AA1050

10:35 AM Break

10:45 AM Invited

Intrinsic or Extrinsic Plastic Strain Gradients Enhance the Effectiveness of SPD Processes: *Javier Gil Sevillano*[1]; [1]CEIT and TECNUN, University of Navarra

The nanostructuring ability and strengthening effects of severe plastic deformation (SPD) processes significantly differ when comparison is made at equal equivalent strains and homologous temperatures. For instance, large strain wire drawing of BCC or HCP alloys, high pressure torsion (HPT) and accumulative roll bonding (ARB) systematically are quoted to be more effective than equal channel angular extrusion (ECAE), reciprocating extrusion or constrained groove pressing. HPT and ARB induce sample size-dependent extrinsic (macroscopic) plastic strain gradients; BCC or HCP wire drawing induce intense intrinsic (microscopic, associated to the grain microstructure) ones. In both cases those gradients are to be accommodated by an extra storage of geometrically necessary dislocations (GND) absent in other SPD processes. Such extra GND density can explain the superior effectiveness of HPT, ARB or BCC-WD as SPD processes. New SPD process should be designed with enhanced intrinsic or extrinsic plastic strain gradient development ability.

11:05 AM Invited

Modeling the Mechanical Response of fcc Materials Processed by ECAE: *Irene J. Beyerlein*[1]; Carlos N. Tome[1]; David J. Alexander[1]; [1]Los Alamos National Laboratory

In this work we study the asymmetry in the tension versus compression response observed in fcc materials processed by equal channel angular extrusion (ECAE). The substantial difference in the response between these two tests is attributed to a combination of the texture evolution and the grain and subgrain microstructural evolution induced by ECAE. To predict this response we incorporate a recently developed single crystal hardening model into a Visco-Plastic Self Consistent (VPSC) polycrystal model. The hardening law captures the directional anisotropy in the single crystals that is induced by the formation of planar dislocation walls. It also captures the effects of new substructure that dissolves and builds upon previously developed substructure when the strain path changes from ECAE to uniaxial deformation.

11:25 AM Invited

Softening in Constitutive Relationship of Pure Copper at High Strain Level: *Jingtao Wang*[1]; Wei Wei[1]; Yue Zhang[1]; Guang Chen[1]; [1]Nanjing University of Science and Technology

To study the constitutive relationship of pure copper at high strain levels, equal channel angular pressing (ECAP) was used to impose high levels into the pure copper samples, and tensile testing was used to estimate the yield stress after ECAP. It is found that the Stress - Strain (accumulated through ECAP) constitutive relationship shows softening as the accumulated ECAP strain level beyond an equivalent strain of ~4. This behavior could not be described by the existing constitutive relationship models of the Hollomon power-law relationship at low strains, or the Voce exponential relationship at high strains. An new constitutive equation was deduced to describe this softening behavior.And this softening behavior is dicussed considering the microstructural evolution during ECAP.

11:45 AM Invited

Strain-Rate Dependence of Tensile Ductility of Cryomilled 5083 Al Alloys: *Bing Q. Han*[1]; J. Y. Huang[2]; Y. T. Zhu[3]; E. J. Lavernia[1]; [1]University of California; [2]Boston College; [3]Los Alamos National Laboratory

The mechanical properties of nanostructured or ultrafine-grained (UFG) materials have engendered increased interest for the past two decades. Inspection of numerous published studies reveals that there is a strong effect of strain rate on the mechanical properties of nanostructured or UFG materials. In many cases, it is found that the tensile ductility increases with increasing strain rate and the strain rate sensitivity exponent is larger than that in their counterpart coarse-grained materials. In the present study, the mechanical properties and microstructural characterization of several bimodal nanostructured 5083 Al alloys processed from cryomilled nanostructured powders are investigated. It is noted that there are higher values of ultimate tensile strength and ductility as well as compression flow strength at slower strain rates in the cryomilled 5083 Al alloys. The relationships among mechanical properties at different strain rates, microstructural characteristics and deformation mechanisms of these materials are thus explored in the present study.

12:05 PM

Deformation Behavior of Cryomilled Al-Mg Alloy Consolidated via ECAP: *Jichun Ye*[1]; Bing Q. Han[1]; Dong H. Shin[2]; Enrique J. Lavernia[1]; Julie M. Schoenung[1]; [1]University of California; [2]Hanyang University

Mechanically alloying in liquid nitrogen (cryomilling) has been proven to be an effective approach to produce nanostructured aluminum powders. Recently, several nanostructured/ultrafine-grained aluminum alloys have been consolidated from cryomilled nanostructured aluminum powders and the mechanical properties have been investigated. It is generally observed that there is a stress drop after a brief work-hardening region and the necking deformation is dominant in the plastic deformation of cryomilled aluminum alloys. Inspection of previous investigation indicates that the consolidation routes have a significant influence on the mechanical performance of aluminum alloys. In the present study, the cryomilled Al-7.5%Mg alloy powders are consolidated using hot isostatic pressing followed by equal channel angular pressing. Tensile properties and microstructural characterization of the consolidated Al-Mg alloy are investigated and compared with those processed via other routes. The influence of intrinsic microstructural characterization arising from different processing routes on deformation mechanisms of cryomilled aluminum alloys is discussed.

12:20 PM

Mechanical Behavior of Nanocrystalline Ni: *Indranil Roy*[1]; Manish Chauhan[1]; Farghalli A. Mohamed[1]; [1]University of California

In the present study, the mechanical behavior of Ni with an average grain size of 100 nm has been investigated. Tensile tests have been performed on the material in the temperature range of 393 to 473 K and strain rate of 10-5 to 10-2 s-1. The experimental data reveal high strength, low-strain hardening and a defined yield point. Observations regarding ductility and fracture will be discussed.

Wechsler Symposium on Radiation Effects, Deformation and Phase Transformations in Metals and Ceramics: Irradiation Facilities and Techniques

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

Wednesday AM Room: 208
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Gary S. Was, University of Michigan; Steven J. Zinkle, Oak Ridge National Laboratory

8:30 AM Invited
Materials Issues for High Power Accelerators: *Louis K. Mansur*[1]; [1]Oak Ridge National Laboratory

High power accelerators present numerous research issues. Some questions are unique, others are similar to considerations for fission and fusion reactors. There may be conditions for which there are little or no data. Materials research may reduce risk or even establish a new concept's viability. High power accelerators are applied to neutron scattering research and radiation materials science, and proposed for waste transmutation. Others produce radioactive ion beams, or are used for particle physics. Applications are mentioned; spallation neutron sources are emphasized. The design of the Spallation Neutron Source (SNS), a new US materials research facility, is described. The target suffers the highest displacement damage; somewhat lower are beamline windows and a beam dump, where the beam is transferred between accelerators. Other components experience high ionization damage; they also must be designed for radiation resistance. Questions that require future materials research are discussed.

8:55 AM Invited
Materials Irradiation Facilities at the High-Power Swiss Proton Accelerator Complex: *Werner Wagner*[1]; Yong Dai[1]; Heike Glasbrenner[1]; [1]Paul Scherrer Institute

At the Swiss proton accelerator complex and spallation neutron source SINQ irradiation facilities are operated to investigate materials behaviour under high-dose irradiation conditions. In the frame of STIP (SINQ Target Irradiation Program) hundreds of samples, mainly austenitic and ferritic/martensitic steels like 316L, T91 or F82H, were irradiated under realistic spallation conditions, i.e. in a mixed spectrum of 570 MeV protons and spallation neutrons, to doses up to 20 dpa. As well, solid metals in contact with liquid Hg and liquid lead bismuth eutectic (LBE) were part of the program. In complement, in LiSoR (Liquid Solid Reaction) loop T91 steel was irradiated with 72 MeV protons while being in contact with flowing LBE at elevated temperatures and under tensile stress. The post-irradiation examinations yield valuable information for safety assessments and lifetime predictions of high-power accelerator driven systems using liquid lead alloys as target and coolant material.

9:20 AM Invited
A Comparison of Microstructure in Martensitic and Austenitic Steels Irradiated in SINQ Targets: *Yong Dai*[1]; Xuejun Jia[1]; [1]Paul Scherrer Institut

Martensitic steels F82H and T91 and austenitic steel SA316LN have been irradiated in SINQ targets up to 20 dpa in a temperature range of 80-400ºC. TEM observations have been performed. For F82H and T91 the results show: (1) the formation of dense defect clusters and dislocation loops at =350°C; (2) the formation of high-density visible He bubbles (> ~1 nm) at temperatures above 170°C; (3) bubbles (or voids) up to 60 nm large formed in a sample irradiated to 20 dpa / 1790 appm He at 400ºC, and a bimodal size distribution was observed; (4) in this sample, the martensite lath structure disappeared accompanied with the formation of new M23C6. For SA316LN steel, high-density small defect clusters and large frank loops were observed. High-density tiny helium bubbles were observed in samples irradiated at >350°C. The difference of microstructure in martensitic and austenitic steels will be discussed.

9:45 AM
In Situ Neutron Diffraction Studies of Artificial Aging in Uranium-Niobium Alloys at LANSCE: *Donald Brown*[1]; Mark A. Bourke[1]; Robert E. Hackenberg[1]; Larry Hults[1]; David F. Teter[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory

Niobium is soluble in uranium at high temperature, in the body-centered cubic phase, but not at room temperature. The diffusion of Nb in U is rather slow, and if the alloy is quenched at moderate rates a metastable monoclinic phase is produced at room temperature. The properties that make U-Nb attractive, such as enhanced ductility, are a strong function of the Nb content and are optimized in this metastable phase at 6 weight percent. This study was aimed at understanding the stability of the alloy through in-situ neutron diffraction measurements during accelerated aging. Samples were heated in-situ to temperatures between 100°C and 400°C and the development of the interatomic spacings monitored over roughly one-day aging times by taking diffraction patterns at 5-20 minute intervals. The observed changes in the lattice parameter are related to the decreased Nb in solution with time at temperature.

10:05 AM Break

10:20 AM
Topological Model of Martensitic Transformations in Ferrous Alloys: *Xiao Ma*[1]; Robert C. Pond[1]; [1]University of Liverpool

The Phenomenological Theory of Martensite Crystallography is based on the hypothesis that the habit plane is an invariant plane of the shape transformation. Experimental observations of the crystallography of a range of transformations are consistent with this notion, but several instances, including some ferrous alloys, are not. Recently, a topological model of martensitic transformations has been presented wherein the habit plane is a semi-coherent structure, as is observed using TEM, and transformation mechanism is shown explicitly to be diffusionless. The transformation crystallography predicted using the topological approach differs systematically from the classical approach, reflecting the differing compatibility criteria at interfaces between atomic solids, as represented in the former, and continua, as in the latter. Experimental observations of ZrO_2 and Ti alloys are in excellent agreement with the topological model; the objective of the present work is to apply the topological method to ferrous alloys and compare with the phenomenological predictions.

10:40 AM
Kinetic Pathways of Temperature- and Field-Dependent Structural Phase Transitions in Ferroelectric PMN-PT and PZN-PT near Morphotropic Phase Boundaries: *Yu U. Wang*[1]; [1]Virginia Tech

Thermodynamic analysis and kinetic modeling are developed to explain recent experimental observations of the structural phase transition sequences, phase stabilities, polarization rotations, crystallographic data, and ultrahigh electromechanical responses of PMN-PT and PZN-PT ferroelectric/ferroelastic perovskites. It is shown that the fascinating while puzzling phase behaviors in such multiferroic oxides can be understood from the perspectives of self-accommodation of spontaneous ferroelastic strain and ferroelectric polarization and self-assembling of ferroelastic and ferroelectric domains at nanometer length scale. Extensive experimental data supporting this theory are presented. Ongoing computer simulation effort to gain better quantitative insight into this problem is also discussed.

11:00 AM Invited
Deformation Dynamics Studies Using Stress Relaxation: *Placid Rodriguez*[1]; [1]Indian Institute of Technology

When a tensile or compression test (normally performed at constant extension or strain rate) is interrupted, stress-relaxation occurs since plastic deformation continues to take place as long as the applied stress is sufficiently high for the dislocations to move. Analysis of the load/stress Vs. time data leads to valuable information on the relationship between

stress and plastic strain rate and important parameters like activation area, strain rate sensitivity and the thermal component of the flow stress. Monroe Wechsler's group at Oak Ridge was one of the first to use the technique particularly for studies on irradiation hardening. This paper makes a review of the deformation dynamics results using stress relaxation in a variety of materials; a few examples are also discussed in which the technique has been used to study the kinetics of stain ageing and dynamic recovery.

11:25 AM

The NCSU Radiation Damage Database: Proton-Induced Helium Production Cross Sections: *Wei Lu*[1]; Monroe Wechsler[2]; [1]Oak Ridge National Laboratory; [2]North Carolina State University

A radiation damage database is completed at NCSU. It contains damage energy, displacement, helium, and hydrogen cross sections for 23 elemental targets by proton and neutron projectiles up to 3.2 GeV. As a part of the database, proton-induced helium cross section is described in this paper. The cross section calculation runs on CEM2k with default options and on Bertini with three level densities and multistage pre-equilibrium model (MPM) on and off. The calculation results were compared to the experimental data so far available. Such an evaluation suggests that, depending on the target mass, different intranuclear cascade (INC) models or different level densities within the INC model may be chosen for the proton-induced helium production cross section. The database indicates which cross sections give best agreement with experimental data. The criteria derived from the evaluation could be applied to neutrons as well as protons. Neutron and proton cross sections are approximately equal at high energies.

11:45 AM

Monte Carlo Simulations of Stray Neutron Radiation Exposures to Proton-Beam Radiotherapy Patients: *Yuanshui Zheng*[1]; Jonas Fonteno[1]; Nicholas Koch[1]; Wayne Newhauser[1]; [1]UT MD Anderson Cancer Center

Radiation therapy is one of the major treatment options for cancers, and proton radiation therapy is gaining increased interest around the world because it offers more normal tissue sparing when compared to conventional radiation therapy. However, second neutron production is of greater concern in proton therapy due to the presence of stray and leakage neutrons that are produced by proton interactions in both the beamline and the patient. The authors predicted the neutron doses to a typical patient receiving proton therapy at the University of Texas M. D. Anderson Cancer Center (Houston) using the Monte Carlo method simulation. The resulting risk of secondary cancer for a patient was also estimated.

2006 Nanomaterials: Materials and Processing for Functional Applications: Carbon Nanostructures

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

Wednesday PM — Room: 214C
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

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

2:00 PM Introductory Comments

2:05 PM

Microstructural Observations of Deformation Mechanisms in Nanoindented DLC Coatings on Silicon Substrates: Ayesha Jabeen Haq[1]; *Paul Richard Munroe*[1]; Mark Hoffman[1]; Phil Martin[2]; Avi Bendavid[2]; [1]University of New South Wales; [2]CSIRO Telecommunications and Industrial Physics

The surface and subsurface microstructural changes resulting from deformation induced by nanoindentation have been studied in a number of diamond-like carbon (DLC) coatings on silicon substrates. A range of amorphous hydrogenated carbon films (a-C:H) were deposited onto silicon substrates using a plasma assisted chemical vapour deposition technique. The compositions of the as-deposited coatings were characterised by Raman spectroscopy and transmission electron microscopy (TEM). The coatings were indented to loads up to 500 mN using a 5 μm spherical indenter. Detailed microstructural studies were performed on the indented regions using atomic force microscopy, focused ion beam milling and TEM. The planned presentation will correlate features observed in the load-displacement curves, such as pop-ins and pop-outs, to observed microstructural events, such as the onset of slip on {111}, cracking, delamination and localised phase transformations in the silicon substrate.

2:25 PM

Conductivity Predictions in Carbon-MMC's Based on Contact Resistance Considerations: *Ivica Smid*[1]; Erich Neubauer[2]; [1]Pennsylvania State University; [2]Austrian Research Centers

Metal Matrix Composites (MMCs) based on carbon based reinforcements exhibit a high potential for applications as heat sink materials. From theoretical considerations the thermal properties can be tailored by a simple variation of the volume fraction of the reinforcement. An additional parameter of importance in the copper-carbon system is the thermal contact resistance (TCR). By a variation of the TCR the whole range between an insulating interface and a perfect heat transfer can be simulated in theoretical calculations. The realization of the desired experimental values for the TCR, however, is not an easy task. The experimental values of the TCR in the carbon-copper system were determined by the help of photothermal methods. The resulting bulk thermal properties have been modeled using finite element methods, allowing a prediction of the maximum achievable conductivity as a function of processing, and filler particle size and shape.

2:45 PM

Grow and Control the Length of Carbon Nanotubes on Silicon Substrates by Chemical Vapor Deposition: *Zhengjun Zhang*[1]; Ya Zhou[1]; Yang Yue[1]; [1]Tsinghua University

Carbon nanotubes were believed not able to grow directly on silicon substrates by chemical vapor deposition from a mixture of ferrocene and xylene, due to the reaction of Fe with Si. Via controlling the growth kinetics to suppress the reaction of Fe with Si, we have successfully deposited carbon nanotubes on silicon substrates. Using the fact that nanosized materials exhibit mostly a lower melting point that the bulk form, we thus used the vaporization of nanosized Au films as a block to suppress nanotubes growth, and established a technique to fabricate arrays of aligned nanotubes on silicon substrates, and to control the length of nanotubes from site to site. This controllable growth of carbon nanotubes on silicon substrates might be further utilized to build large-scale electronic devices, which brings no further contamination to the substrates.

3:05 PM Break

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Carbon Nanotube Deformation - In-Situ SEM Observation and Force Measurement: *Christian P. Deck*[1]; Kenneth Scott Vecchio[1]; Chi-Nung Ni[1]; Prabakar R. Bandaru[1]; [1]University of California

Carbon nanotubes (CNTs) have been the subject of great interest in many fields, due to their unique material properties and geometry. In particular, they possess exceptional mechanical strength, which is desirable in many diverse applications, such as field emission, sensing devices, and composite reinforcement. A stage was designed and built to allow in-situ scanning electron microscopy observation of compression, tension, and shear testing of densely-packed mats of well-aligned multi-walled CNTs. These tubes were grown using both thermal and vapor phase chemical vapor deposition (CVD) methods, and different tube lengths and morphologies were investigated. Force measurements were taken during testing, and mechanical properties of the CNT mats were obtained, as well as other properties such as tube-substrate bond strength. The relevance of these results to nanotube-based tactile and flow sensing devices is also discussed.

3:40 PM
Evaluation of the Field Emission Properties of Carbon Nanotubes: *Victor Kumsomboone*[1]; Stephan Turano[1]; Brent Wagner[1]; Jud Ready[1]; [1]Georgia Tech Research Institute

Carbon nanotubes (CNTs) are investigated for field emission (FE) properties for applications in ion electric propulsion and field emission display by altering synthesis parameters. CNTs are grown on quartz and silicon substrates utilizing thermally evaporated Iron (Fe) as a catalyst layer on top of electron-beam evaporated Titanium (Ti) as a conductive layer. CNTs are grown via chemical vapor deposition pyrolysis of hydrocarbon gases at various flow rates. CNTs are observed via scanning electron microscope (SEM). FE properties are evaluated in a diode configuration at a pressure of 10^-5 Torr, applied voltage ~500V, and cathode/anode separation (CAS) ~500µm. Electrons flow from the voltage source through the CNTs cathode, across the CAS, and illuminate a layer of phosphor at the anode. Qualitative results include SEM images and macroscopic images of illuminated phosphor. Quantitative results include percentage illumination of the anode, current across the anode, and graphs modeling each vs. various growth controls.

4:00 PM
Growth of Self-Organized Carbon Nanotubes Using Anodic Aluminum Oxide Template on a Si Substrate: *Ching-Jung Yang*[1]; Jia-Min Shieh[2]; Chang-Hsuan Lee[1]; Chih Chen[1]; Fu-Ming Pan[1]; Bau-Tong Dai[2]; [1]National Chiao Tung University; [2]National Nano Device Laboratories

In this study, we report the development of a carbon nanotube using a thin film of anodic aluminum oxide template on a Si wafer. We sputter 20 nm TiN followed by 10 nm Ni. The TiN layer acts as a diffusion barrier to prevent silicidation of the Ni, which is needed as the catalyst for CNT growth. For the preparation of the AAO template, 1.5 µm Al film was deposited on the Ni layer by thermal evaporation. The nanopores feature a uniform size with a hexagonal pattern. By using the AAO template, the CNTs of a very high density of 1.7×1010 tubes/cm^2 can be grown. The turn-on electric field was 2.8 V/µm and emission current density was 80 mA/cm^2 at 8 V/µm. Our fabrication technique enables us to control the tube diameter, length and density easily. This approach offers a potentially elegant technique for fabricating cold-cathode flat panel displays.

4:20 PM Break

4:35 PM
Thermo-Gravimetric Analysis of Synthesis Variation Effects on CVD Generated Multi-Walled Carbon Nanotubes: *Gregg S. B. McKee*[1]; Kenneth S. Vecchio[1]; [1]University of California

The unique properties of carbon nanotubes have suggested a myriad of applications in a variety of fields. However, consistent and optimal growth of high purity, high quality nanotubes has proven to be a challenge. Small variations in synthesis conditions have significant effects upon the final nanotube product. We examine changes in the thermo-gravimetrically determined oxidation behaviors of CVD-grown multi-walled carbon nanotubes with varying synthesis conditions. Catalyst type and synthesis temperature are found to have a measurable impact upon nanotube stability, suggesting differing levels of crystalline perfection in the resulting nanotubes. The results provide evidence showing the catalytic effects of nanotube catalyst particles and their oxides upon the oxidation of nanotube carbon and graphite. The significance of thermo-gravimetric analysis as a characterization tool for carbon nanotubes is discussed.

4:55 PM
Oxidation Activation Energy Determination and Comparison for CVD Grown Multi-Walled Carbon Nanotubes: *Gregg S. B. McKee*[1]; Kenneth S. Vecchio[1]; [1]University of California

Oxidation rates are measured and an activation energy determined for several length ranges of as-grown and treated chemical vapor deposition grown nanotubes within a range of 725K to 900K. The activation energy barrier is found to be within the range calculated in the literature and may not change significantly with synthesis method or with changing nanotube length. The results show that the oxidation of carbon nanotubes need not originate in the nanotube caps alone, but may originate in other areas of increased strain energy.

5:15 PM
Energetic Comparison of Single-Walled Carbon Nanotube Computer Simulations: *Shalayna L. Lair*[1]; Lawrence E. Murr[1]; William Herndon[1]; Stella Quinones[1]; [1]University of Texas

An easily applied graphical approach for facilitating precise tailoring during computational construction of modeled uncapped or capped carbon nanotubes or fullerenes is delineated and utilized in this paper. The main enabling concept is nucleation of single and multi-walled carbon nanotubes from end cap structures. A novel construction protocol is used to rapidly create any type of armchair, zigzag or chiral defect-free nanotube. Any feasible combination of length and diameter, along with specific placement of hexagonal and pentagonal rings in end caps, can be controlled. The suggested methodology is used to systematically model heats of formation of a variety of carbon nanotubes and related fullerenes using AM1 semiempirical calculations. The main factors affecting the calculated physical properties, other than size, are the structures of the various base and terminating end caps. The possible relationship of the construction methodology to mechanisms for carbon nanotube nucleation will also be commented on.

3-Dimensional Materials Science: Serial Sectioning

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

Wednesday PM — Room: 205
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: George Spanos, Naval Research Laboratory; Michael D. Uchic, Air Force Research Laboratory

2:00 PM Invited
Automated Serial Sectioning: An Enabling Technology for 3D Microstructural Analysis: *Jonathan Edward Spowart*[1]; Herbert M. Mullens[2]; [1]U.S. Air Force; [2]UES, Incorporated

The aim of this presentation is to introduce some of the tools and techniques that have been developed at AFRL for performing automated serial sectioning of advanced materials. These techniques are seen as enabling for many 3D microstructural analysis problems. The presentation will draw from selected examples, spanning a broad range of materials science areas and illustrating the current state of the art in automated serial sectioning. In addition, projections for both the near-term and long-term development of the technology will be provided, highlighting the limitations of current practices and exploring the potential for future enhancements in terms of higher throughput, greater fidelity and increased access to the 3D data.

2:25 PM
Three-Dimensional Characterization of Damage in Shocked Tantalum: *Benjamin L. Henrie*[1]; John F. Bingert[1]; [1]Los Alamos National Laboratory

Tantalum plate subjected to shock loading was serially sectioned to interrogate the effect of shock parameters on damage accumulation. A primary goal was to determine correlations between microstructural details and damage in the form of voids and strain localization. Serial sectioning was performed at 5 µm increments using optical microscopy and electron backscatter diffraction (EBSD) as characterization tools. Image segmentation resulted in the reconstruction of void networks that allowed for insight into their nucleation and growth characteristics, and spatial relationships. In addition, the interaction of voids was investigated with regards to the role of strain localization and their connectivity with the void network. Crystallographic orientation data was also applied to enhance the reconstructed data set with anisotropic information. Relevant

microstructural statistics regarding feature distributions were also calculated.

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Visualization of Three-Dimensional Microstructures Reconstructed from Serial Sections in Modified Ti-6Al-4V Alloys with TiB Whiskers: *Scott I. Lieberman*[1]; Arun M. Gokhale[1]; Sesh Tamirisa[2]; [1]Georgia Institute of Technology; [2]Ohio University

In the development of modified titanium alloys containing in-situ formed titanium boride whiskers (TiB_w), 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 variations of Ti-6Al-4V-TiB materials.

3:05 PM

Recovery of the Grain Boundary Character Distribution through Oblique Double-Sectioning: *Eric R. Homer*[1]; Brent L. Adams[1]; [1]Brigham Young University

A new method for the retrieval of the complete grain boundary character distribution (GBCD) by a new oblique double-sectioning (ODS) method is presented. Section cuts taken from the sample at oblique angles are prepared by performing a pair of parallel material removals and their corresponding OIM scans. In this manner an incomplete GBCD, including grain boundary inclination, is measured for each double section. The overall GBCD of the material is then obtained from the set of oblique double-sections in a manner similar to that applied for recovery of orientation distributions from incomplete pole figures. Comparison of ODS with calibrated serial sectioning and the L_A/S_V stereology will be presented along with the solution to the fundamental equations of ODS in the Fourier space. Implementation of the method for rolled and annealed alloy 304 stainless steel will be presented.

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Three-Dimensional Reconstruction of Alpha Laths in Alpha/Beta Ti Alloys: *Robert E. A. Williams*[1]; Michael Uchic[2]; Dennis Dimiduk[2]; Hamish L. Fraser[1]; [1]Ohio State University; [2]Air Force Research Laboratory/MLLMD

Alpha/beta titanium alloys have a complex microstructure involving features spanning a wide range of size scales that can vary from sub micron to millimeters depending on thermo-mechanical history. Recent advances in stereology and microscopy have made quantification of titanium microstructures possible, but in order for stereology to be validated a physical three-dimensional understanding of the features is necessary. 2-D images provide limited information regarding the 3-D nature of the microstructure and can often be misleading. In order to gain a better understanding and reduce error due to stereological measurements, a FEI NOVA 600 microscope was used to section serially through alpha/beta titanium microstructures for digital reconstruction of morphological features. EBSD patterns were also collected in order to visualize and validate the values measured by stereology. The 3-D reconstruction also provides a physical representation of interactions between microstructural features.

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Statistical Microstructural and Crystallographic Analysis in 3D: *Alexis C. Lewis*[1]; Andrew B. Geltmacher[1]; David J. Rowenhorst[1]; George Spanos[1]; [1]Naval Research Laboratory

Three-dimensional microstructural measurement, modeling, and analysis techniques are being applied to large datasets which contain the spatial and crystallographic data for statistically representative volumes comprising hundreds of grains. These 3D volumes contain experimental data derived from serial sectioning, Electron Backscatter Diffraction (EBSD), Focused Ion Beam (FIB) characterization, and X-ray tomography. Properties which have been quantified in three dimensions include grain volume, true 3D grain shape (aspect ratio, and number of faces, edges and corners for each grain), crystallographic orientation, 3D grain boundary networks and populations, and distributions of grain boundary crystallographic normals. This data is input into 3D Image-Based Finite Element Models for analysis of stress and strain evolution in the microstructure. In addition to analytical results, 3D measurement and visualization techniques will be discussed.

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3D Analysis of Early Stages of Creep Void Development: Azmi Abdul Wahab[1]; *Milo V. Kral*[1]; [1]University of Canterbury

Hydrogen reformer tubes fail by a sequence of creep void nucleation, growth and coalescence. Previous 3D analysis by serial sectioning and computer reconstruction on a severely crept reformer tube indicated that creep voids occurred at grain edges and corners and voids were always found adjacent to chromium-rich $M_{23}C_6$ precipitates. However, due to the extensive nature of the damage, it was not possible to determine the precise nucleation site. In the present work, samples have been taken at various positions along the length of an ex-service tube (operating different service temperatures) in order to analyze the progress of creep damage. By examining a single reformer tube in various stages of creep, early stages of creep void development can be better understood. Comparisons to interrupted creep test samples and as-cast material will also be presented.

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Three Dimensional Visualisation of Splat-Substrate Interactions in Plasma Spray Coated Systems: Damien McGrouther[1]; *Paul Munroe*[1]; William Trompetter[2]; Margaret Hyland[3]; [1]University of New South Wales; [2]Institute of Geological and Nuclear Sciences; [3]University of Auckland

A series of coated substrates were prepared by high velocity oxy-fuel (HVOF) spray processing. In this case, nickel powders were sprayed over an aluminium substrate. Spray parameters were controlled such that the powders were sprayed under conditions where their velocity is high, but temperature is low. The particles remain solid, or semi-solid, in flight and their high momentum means that they become embedded in the substrate. Of particular interest is the nature of the interaction between the particle (or splat) and substrate and whether their interaction is solely mechanical or results in partial melting. In this study dual-beam focused ion beam instrumentation has been used to acquire and generate three-dimensional sections through these splats. It is shown that these visualisations allow the degree of melting of the splat to be readily discernable, in addition to the extent of the plastic deformation of the unmelted portions of the splat.

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The Influence of Material Type and Milling Parameters on the Generation of High Quality EBSD Patterns for 3-D Orientation Mapping by FIB Tomography: *Michael Ferry*[1]; Nora Mateescu[1]; Robin Ma[1]; [1]University of New South Wales

The present paper describes the influence of both material type and milling parameters on the quality of electron backscatter diffraction (EBSD) patterns produced by focussed ion beam (FIB) milling. A sound understanding of the influence of these parameters is necessary for accurate three dimensional reconstruction of EBSD orientation maps for generating crystallographic information in small volumes of material. Samples studied in this study include single crystals of a range of metals and intermetallic compounds with FIB milling parameters and EBSD conditions varied to optimise EBSD pattern recognition. It was found that a reasonable correlation exists between EBSD pattern quality and atomic number of the material with the FIB milling parameters needed to be adjusted accordingly. The work indicates that rapid generation of EBSD orientation maps by FIB milling is most difficult in low atomic number materials.

Advanced Materials for Energy Conversion III: A Symposium in Honor of Drs. Gary Sandrock, Louis Schlapbach, and Seijirau Suda: Magnets, Superconductors, Thermoelectrics and Energy Materials 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

Wednesday PM Room: 214B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Donald Anton, United Technologies Research Center; Dag Noreus, Stockholm University; Renato G. Bautista, University of Nevada

2:00 PM Invited
Very Fine Controlled Nd-Fe-B Microstructure for High Performances Magnets: *Daniel Fruchart*[1]; P. de Rango[1]; J. Luo[2]; S. Miraglia[1]; I. Popa[1]; S. Rivoirard[1]; [1]Centre National de la Recherche Scientifique; [2]North Western Institute for Non-Ferrous Metals

Hydrogen Decrepitation (HD) and Hydrogenation Disproportionation Dehydrogenation Recombination (HDDR) are recognized techniques allowing both to deliver very fine microstructures for the design of high performances Nd-Fe-B magnets. HD permits to refine the size of powder particles thus exhibiting the same level of coercivity as the precursor alloy, but interestingly large remanence level were achieved provide a texture was first installed, e.g. via high temperature fast forging. HDDR leads to very high level coercivity but the remanence remains generally low. Introduction of very small amounts of Zr, Nb, Ga etc (e.g. less than 1 at.%) in the precursor alloys allows to optimize well all intrinsic properties. EXAFS analyses have permitted to localize unambiguously the extra precipitates. Kinetic analyses of hydrogenation/dehydrogenation processes were performed and then fairly fitted via Jander and Avrami's type laws for the best understanding of hydrogen interaction and time diffusion mechanisms via extra precipitates.

2:25 PM
Processing and Study of Magnetic and Magnetostrictive Properties of Fe-Zn and FeGaZn Alloys: *Swieng Thuanboon*[1]; Robert P. Corson[1]; Sivaraman Guruswamy[1]; [1]University of Utah

Zinc has a large solubility in Fe and a completely filled "d" shell. With the discovery of dramatic increase of the magnetostriction in Fe by Ga addition, an examination of element Zinc that is adjacent to Ga in the periodic table is of significant interest. In this work, the effect of binary alloying of Zn with Fe and ternary alloying with FeGa alloy on the magnetic and magnetostrictive properties is examined. The alloys were processed using powder metallurgy and vapor phase synthesis, and Zn contents were varied from 2.5% to 20%. An examination of the influence of different processing parameters and the Zn content on the resulting alloy is properties are presented. The alloys were characterized using X-ray diffraction and SEM. Magnetostriction measurements were carried out at different pre-stress levels. Magnetic properties were measured using a vibrating sample magnetometer.

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Effect of Ordering on the Elastic and Magnetostrictive Properties of Fe-27.5 at% Ga Alloy Single Crystals: *Tanjore V. Jayaraman*[1]; Swieng Thuanboon[1]; Sivaraman Guruswamy[1]; [1]University of Utah

FeGa alloys show large magnetostriction that is attractive in sensor and actuator applications. Fe-Ga alloys with composition around 27.5 at.% Ga can be heat treated to obtain ordered phases based on alpha" (ordered bcc), DO19 (ordered hexagonal) and Ll2 (ordered fcc) structures. Our earlier work using polycrystalline samples had shown that magnetostriction is strongly influenced by ordering in this alloy. A more detailed study of the correlation between ordering and magnetostriction using single crystals of Fe 27.5 at% Ga alloys is reported in this paper. X-ray and electron diffraction were used to study the ordering in this alloy. Magnetostriction measurements were carried out at different pre-stress levels. Magnetic properties were measured using a vibrating sample magnetometer. Elastic constants were measured using Resonant Ultrasound Spectroscopy.

3:05 PM
Thermoelectric Properties of High Temperature Boron Cluster Materials: *Takao Mori*[1]; [1]National Institute for Materials Science

The search for thermoelectric materials is being carried out with great intensity because of the huge possibilities for useful energy conversion of waste heat. There is obviously a particular need to develop materials which can function at high temperatures. Boron-rich cluster compounds are attractive materials for their stability under high temperature typically exhibiting melting points above 2200 K. The framework of new rare earth boron cluster compounds that we have discovered is basically composed of boron clusters while heavy rare earth atoms reside in spaces among the clusters, and we note that they have substantially lower thermal conductivities (favorable for thermoelectric applications) compared to β-boron systems which have been previously investigated. REB_{50}-type compounds exhibit Seebeck coefficients greater than 200 μV/K at high temperatures and unlike most compounds, the figure of merit shows a steep increase at T>1000 K. Properties of other novel B_{12} icosahedral cluster-containing compounds will also be presented.

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High Pressure Raman Spectroscopy Studies on Organic "Plastic Crystal" Thermal Energy Storage Materials: *Raja S. Chellappa*[1]; Dhanesh Chandra[1]; [1]University of Nevada

Alcohol derivatives of neopentane are potential candidates for thermal energy storage applications. The phase transformation temperatures at one atmosphere of pure compounds are well established and these materials typically undergo a solid-solid transition from a low temperature ordered structure to a high temperature orientationally disordered "plastic crystal" cubic phase. Examples of these "plastic crystals" include Pentaerythritol [(PE):C(CH2OH)4], and Neopentylglycol [NPG:(CH3)2C(CH2OH)2]. We are conducting a systematic study to characterize the pressure induced phase transformations in these compounds. We will present results from our high pressure Raman spectroscopy studies conducted at room temperature for PE, and NPG. Based on our analysis of the various Raman modes and pressure induced wavenumber shifts, PE exhibits phase transformations at ~4.8 GPa, ~6.9 GPa, and ~9.4 GPa before amorphization at ~15 GPa.

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Peculiarity of Ternary Stannides and Antimonides Exhibiting Potentially Thermo-Electric Properties: Crystal Structures, Physical Properties, Electronic Structures: L. P. Romaka[1]; O. Bodak[1]; Yu. Y. Stadnyk[1]; M. G. Shelyapina[2]; E. K. Hlil[3]; *Daniel Fruchart*[3]; P. Wolfers[3]; [1]Ivan Franko Lviv National University; [2]St. Petersburg State University; [3]Centre National de la Recherche Scientifique

Three different types of new ternary stannides and antimonides have been recently synthesized and investigated for their structural and physical properties, and systematically analyzed in terms of band structure determination. These materials are formed with an early d-metal such as Ti, Zr, Hf (as well as Sc for a part), a late d-metal such as Co, Ni, Cu, and X being Sn or Sb. The crystal structure analyses show that they crystallize either with a derivative Cu2Sb, or a TiNiSi or mostly with the MgAgAs (semi-Heusler) type structure. Strong covalent bonding are evidenced from this analyses. The band structure calculations reveal that the late d-metal bands do not contribute significantly to the conduction, but the conductivity (if any) issues from the d- or p- type electrons of the early d-metals. This allows to control fairly the opening a gap (or semi-gap) at EF via the VEC, and to initiate interesting thermo-electric properties.

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Polymer Derived Carbon Electrode Materials for Electrochemical Capacitors: Ravinder Reddy Nagireddy[1]; *Ramana G. Reddy*[1]; [1]University of Alabama

Electrochemical capacitors are charge storage devices. Electrochemical capacitors can be classified into two types, electrochemical double

layer capacitors (EDLC's) and capacitors based on psuedocapacitance. In EDLC, when an high surface area electronic conductor material like carbon is brought in contact with an ionic conducting electrolyte, a charge accumulation is achieved electrostatically on either side of interface, leading to the development of an electrochemical double layer. In this study carbon materials are derived from a polymer sources and are studied as electrode material for electrochemical double layer capacitors. The polymer source is dried at high temperatures to yield carbon. Carbon electrode surface is characterized using NOVA 1200 gas sorption analyzer. It is also characterized using XRD and SEM. Carbon electrode was electrochemically characterized using cyclic voltammetry (CV). Modeling results of potential distribution in porous carbon electrode under cyclic voltammetric conditions will be presented.

4:40 PM

Phase Transition and Thermal Studies of Polyalcohols Thermal Energy Storage Solid Solutions: *Wen-Ming Chien*[1]; Dhanesh Chandra[1]; [1]University of Nevada-Reno

Low temperature X-ray diffraction and thermal property studies of the solid-solid state phase transition for the polyalcohols (pentaerythritol (PE), pentaglycerine (PG) and neopentylglycol (NPG)) thermal energy storage solid solutions have been investigated by using X-ray diffractometry and differential scanning calorimetry (DSC) methods. X-ray diffraction studies on polyalcohols solid solutions, PE-NPG and PG-NPG, are from –10°C to 50°C. XRD results shows the + mixed phase at the related low temperature (–10°C) for both PE-NPG and PG-NPG solid solutions. At high temperature range, XRD results show phase for PG-NPG solid solution and + mixed phase for PE-NPG solid solution. There are only two Bragg peaks, (111) and (200), shown in phase XRD patterns. The polyalcohols solid solutions have been cycled 5 times between -20°C to 100°C at different DSC scan rates to study the phase transition properties. DSC results of both PE-NPG and PG-NPG solid solutions show that the solid-solid state phase transition is near room temperature. Details of low temperature phase transition XRD and DSC results of PE-NPG and PG-NPG solid solutions will be presented.

5:00 PM

Microstructure and Texture of Cast BixSb1-x Alloy Processed by Angular Reduction Extrusion: *Jae-Taek Im*[1]; K. Ted Hartwig[1]; Jeff Sharp[2]; [1]Texas A&M University; [2]Marlow Industries, Inc.

Multipass Equal Channel Angular Extrusion (ECAE) with an area reduction in the exit channel enables one to produce heavily deformed and textured materials. Such an approach is effective for microstructure breakdown and for developing texture to bulk materials. Cast BixSb1-x alloy was extruded above the recrystallization temperature through 90° die for up to four extrusion passes and with area reduction to 75%. The microstructure is characterized by polarized optical microscopy and SEM, and the texture by X-ray diffraction. The results are reported.

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The Development of Metal Oxide Based Supercapacitors: Beth McNally[1]; Nandakumar Nagarajan[1]; *David S. Wilkinson*[1]; Igor Zhitomirsky[1]; [1]McMaster University

The development of a viable power storage system for electric vehicles is gaining importance. The cyclic behaviour caused by repeated acceleration and braking requires a robust system for both short and long term energy storage. A system combining a battery and an electrochemical supercapacitor (ES) has been proposed. The battery provides long-term storage and a constant energy output, with the ES assisting during peak load situations. The discovery of electrochemical capacitance in nickel and cobalt oxides has lead to investigation of these materials as alternatives to ruthenium and iridium oxides. Our work with ESs focuses on the recent developments in the cathodic electrolytic deposition of oxide films. Using polyethylenimine we can produce thick, crack-free oxide films after heat treatment. The presence of the polymer also reduces the particle size within the film both as deposited and after heat treatment. Capacitance values of over 100 F/g have been achieved using cyclic voltammetry for both nickel oxide and cobalt oxide.

Alumina and Bauxite: Plant Design, Operation and Maintenance

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

Wednesday PM — Room: 7B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Jean-Pierre Riffaud, Alumina Partners of Jamaica

2:00 PM Introductory Comments

2:10 PM

Operating Cost - Issues and Opportunities: *Peter-Hans Ter Weer*[1]; [1]TWS Services and Advice BV

The operating cost of a bauxite and alumina project is often regarded as a burden to have to deal with or at best as an unavoidable, sometimes even bureaucratic, management requirement. However in a sense the operating cost and its underlying rationale provide tools to assess the "health" of a project and they may offer improvement opportunities. This paper provides an insight in facets and issues related to the operating cost of a bauxite and alumina project and it explores ways for improvement.

2:35 PM

"Alumina Refineries 21st Century" Computerized Non-Polluting and Green Fields: *Milenko Drakic*[1]; [1]Technip

This article is based on practical experience on gibbsite (hydrargillite) and boehmite bauxite processing with worldwide technology. The author gained industrial experience involving feasibility studies, conceptual design, construction, operation and maintenance of alumina refineries worldwide. This paper addresses the best new technical solutions regarding low operating costs and highest quality product, including higher environmental protection. Greenfield equipment used in these technologies is manufactured in various developed countries. The author has tested different technology and equipment successfully in Kombinate Aluminiuma Podgorica - Montenegro and particularly in Brazilian Company of Aluminium – Brazil while working in bauxite processing, power plant, and environmental engineering. This new technology is different regarding all the actual Bayer process technology.

3:00 PM

New Chemistry for Alumina Recovery from Lateritic Ores: *William F. Drinkard*[1]; [1]Drinkard Research and Development Corporation

We have been developing new chemistry for the recovery of alumina from lateritic ores for the last seventeen years. This new process chemistry promises the elimination of red mud wastes, the ability to use a wider range of alumina containing ores because of superior silica and organic rejection, reduction of operating costs by not requiring settling, and possible elimination of the costliest and most energy-intensive operations in alumina refining, "calcination".

3:25 PM

Digital Fieldbus Implementation for Mineral Processing: *Manoj Pandya*[1]; [1]Alcan Engineering

This paper talks about the implementation of digital communication for process control equipment from concept through to commissioning. The topics include the technology migration plan, associated risk management, engineering design process, and commissioning including benefits gained and the tips and traps for a successful project implementation. A technological change is emerging in the process control and monitoring environment. The enormous economic benefits of the application of digital communication for instrumentation equipment are influencing the decision to abandon the conventional point-to-point technology making the shift to digital technology imminent. To date the large-scale use of this technology has been limited to the petro-chemical sector; however, Alcan has taken the step to keep abreast of this technological change with

the introduction of the Foundation Fieldbus and Profibus as part of the A$2.2bn expansion of Alcan Gove's alumina refinery.

3:50 PM Break

4:10 PM
Maximizing Bauxite Grinding Mill Capacity to Sustain Plant Production: *Pierre G. Cousineau*[1]; Jean Larocque[1]; Colin Thorpe[2]; [1]Alcan Bauxite & Alumina; [2]Alcan Engineering Pty, Ltd

In 2002, a new bauxite wet grinding circuit was commissioned in Alcan's Vaudreuil alumina plant. It replaced the original 60-year-old dry grinding circuit. The former circuit restricted the type of bauxite that could be processed. The new installation, consisting of two rod and ball mills, enabled the plant to improve alumina recovery, process ores with higher moistures and reduce bauxite supply costs. In March 2004, important premature equipment damage was noticed on both mills. In order to avoid significant production losses, it was necessary to upgrade each mill to handle full plant needs, as the repairs on each mill were estimated to last about 6 weeks. A task force was formed with the aim of identifying quick and efficient solutions. Alcan Engineering's milling model was used to evaluate various options. Conclusions and recommendations coming out of the study and plant results are presented.

4:35 PM
Upgrade of Existing Circulating Fluidized Bed Calciners at CVG Bauxilum without Compromizing Product Quality: *Hans W. Schmidt*[1]; Michael Missalla[1]; Vladimir Hartmann[2]; Guzman Lugo[2]; Olivier Hennequin[3]; Andrew Carruthers[4]; [1]Outokumpu Technology GmbH; [2]CVG Bauxilum; [3]Alcan Bauxite & Alumina/Alcan Engineering Gardanne; [4]Alcan Bauxite & Alumina Montreal

To date 50 Circulating-Fluidized-Bed-Calciners have been installed in the alumina industry worldwide or are under construction. They represent a production capacity of 28 million tpa. Over the years, 17 existing calciners were upgraded. Recently, the capacities of the 3 initial CVG-Bauxilum calciners in Puerto-Ordaz, Venezuela were increased to over 2,000 tpd from 1500 tpd, using new concepts. The paper presents performance figures from the existing calciners before and after the upgrade as well as relevant product data. Particle breakage achieved with the upgraded calciners was reduced, while the capacity was increased by approximately 30%. All other product quality data, e.g. LOI, BET, remained unchanged. The specific energy consumption was reduced. Particle emissions of the calciners were reduced by adding a third ESP-field. The close co-operation between Owner, Contractor, and Designer, during the project resulted in a major success: Project schedule was met without compromising high safety standards. Guaranteed performance characteristics were fulfilled.

5:00 PM
Influence of Variables of Process in the Agglomeration Phase on the Attrition of the Hydrate and Its Relationship with Its Morphology: *Erik Farias*[1]; [1]CVG Bauxilum

Considering attrition and granulometric distribution an important part in the quality of alumina and the small amount of morphologic information on this produced in CVG Bauxilum, the following work has shown the effects of the independent variables on dependent variables (productivity, agglomeration, attrition) of the process in the agglomeration phase in relation to its morphology. The contribution of this research will help to initiate the appropriate changes in the process to obtain a product of high quality.

5:25 PM
Crystal Growth Modifying Reagents; Nucleation Control Additives or Agglomeration Aids?: *James A. Counter*[1]; [1]Nalco Australia

Crystal growth modifiers are commonly used in the Bayer process to increase the average gibbsite particle size and improve the particle size distribution during crystallisation from supersaturated sodium aluminate solutions. With respect to the mechanism of action there has been much debated as to whether this result could be due to the additives aiding agglomeration or reducing the number of secondary nuclei. Presented here is work completed on both synthetic and Bayer plant liquors, over various experimental conditions, in order to study the mechanism of action of CGM additives. Crystallisation kinetics, secondary electron microscopy (SEM), molecular modeling and atomic force microscopy (AFM) data are collated to examine the impact that these surface active additives have on the precipitation process.

5:50 PM
Plantwide Replacement of the Existing Control Equipment by a New DCS at AOS: Joerg Ruester[1]; Holger Grotheer[1]; *Rolf Arpe*[1]; [1]Aluminium Oxide Stade GmbH

Since the start-up of the plant in 1973 AOS has been running with single loop process control equipment. The majority of the controllers need to be replaced, since there is no more support available. Therefore, AOS decided to install a modern DCS. Along with the implementation of the system a reduction of staff is supposed to be achieved by means of centralization of the current eleven control rooms and by a higher level of automization. This paper describes the experience with the hot cut-over of the DCS in a running system, the opportunities of a DCS at AOS and the design of a new central control room.

Aluminum Reduction Technology: Cell Development Part III and Emerging Technologies

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

Wednesday PM — Room: 7A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Joaquín J. Fernández Fernández, Alcoa

2:00 PM
Experiences with Long Power Interruption Periods and Lower Amperage Operation in a VS Soderberg Potline: *Jean Yamamoto*[1]; Leonardo Paulino[1]; Carlos Eduardo Zangiacomi[1]; [1]Alcoa Aluminum Inc, Brazil

On December 27th, fuses failed in three rectifier units of Potline #1 in an Alcoa Smelter causing a long and dangerous power interruption. This paper presents the efforts made to reestablish full control of pots with imminent bath freezing process after spending a long period without current and operating with only 70% of nominal amperage. The presentation will focus on two main issues: 1) Actions to reestablish the full pot control after power interruption; 2) Procedures implemented to operate pots at lower amperage level during scheduled switchyard rectifier/transformer maintenance. Actions taken to reestablish pot control during longer power-off period will be presented to operate pots with a risk of freezing electrolyte. Additionally, some strategies will be presented to minimize the risks involved during the successive amperage reduction adopted for switchyard maintenance. Finally, some recommendations will be given to foresee and prevent transformers and rectifiers from unexpected failure mode.

2:25 PM
Influence of Thermo-Hydraulic Fields on Structural Mechanics of Aluminum Reduction Cells: *Yasser Safa*[1]; Michel Flueck[1]; Michel Rappaz[1]; [1]Swiss Federal Institute of Technology EPFL

The elasto-thermal deformation in pot shell of aluminum reduction cell is obtained by solving a stationary elasticity problem with thermal expansion effect as an applied force. The temperature distribution in the whole smelting cell is obtained by two approaches, the first one is based on electro-thermal model without velocity fields, an artificial thermal conductivity is thus used in the heat equation. The second approach is based on a coupled model involving thermal, electromagnetic and hydrodynamic fields. An elasto-thermal calculations corresponding to these two approaches is carried out. The results show the influence of thermal convection on the heating of side walls of the cell. They also exhibit a correlation between velocity fields and mechanical deformation. Strain and stress fields are also presented.

2:50 PM
Towards a Proper Understanding of Sideledge Facing the Metal in Aluminium Cells: *Asbjørn Solheim*[1]; [1]SINTEF

Existing calculation models and hypotheses concerning the sideledge facing the metal in aluminium electrolysis cells are critically examined. It can be concluded that the commonly used heat transmittance theory for heat transfer and growth of ledge is not correct, although the existence of the sideledge can probably only be explained by a bath film between metal and sideledge. Most likely, the bath film is maintained by a source of liquid bath at the bottom of the cell, and the film is alumina-saturated at a temperature close to the metal temperature. An alternative conceptual model for the formation of sideledge facing the metal is briefly outlined. According to the model, formation and removal of sideledge at the metal level will be slow, and the dynamic behaviour can not be directly related to the superheat or to the heat transfer coefficient.

3:15 PM
Technoeconomic Assessment of the Carbothermic Reduction Process for Aluminum Production: Established versus Future Technologies: *Bill Choate*[1]; John A. S. Green[2]; [1]BCS, Inc.; [2]JASG Consulting

In pursuit of the aluminum industry Vision and Roadmap goals, the Department of Energy has partially supported a consortium of Alcoa and Elkem in the development of the Aluminum Carbothermic Technology – Advanced Reduction Process (ACT-ARP), which promises significant energy and emission reductions. This report explores the progress of the ACT-ARP as a potential replacement for the Hall-Hèroult process in the context of several evolving Hall-Hèroult development scenarios. Considerable progress has been made and demonstrated, including new furnace wall designs integral to successful operation of Stage 1 reactor, operational characteristics of vapor recovery reactor and aluminum de-carbonization reactor, as well as significant modeling and simulation. Despite these considerable accomplishments, there are still formidable technical and economic challenges to overcome before the ACT-ARP can replace the conventional Hall-Hèroult process, such as slag and scale formation, metal and carbon quality issues, mini-mill operation, etc. All these and other issues will be discussed.

3:40 PM Break

4:00 PM
Ionic Liquids Electrowinning of Aluminum in Batch Mode Cells: *Mingming Zhang*[1]; Ramana G. Reddy[1]; [1]University of Alabama

A batch-recycle electrowinning cell system was developed to deposit aluminum from 1-hexyl-3-methylimidazolium chloride and aluminum chloride mixture (ionic liquids) at low temperatures. In this system, two types of laboratory cells were used to recycle electrolyte in batch mode: one was cylindrical while the other was in rectangular geometry. During the course of batch experiments, the behavior of current density, current efficiency, cell voltage, aluminum ion concentration, specific energy consumption and process time on both types of cells were analyzed. The results showed the rectangular cell was superior to the cylindrical cell in terms of electrolyte recycling efficiency and cell current efficiency. Energy consumption for rectangular cell system was less than 3 kWh/kg aluminum at current densities up to 350 A/m^2 and cell current efficiencies ranged from 80-90%. Based on these results, the development of large scale Electrowinning Cells for the production of aluminum at low temperatures was also discussed.

4:25 PM
Size Distribution of the Bubbles in the Hall-Héroult Cells: *Sandor Poncsak*[1]; Laszlo Istvan Kiss[1]; Dominic Toulouse[1]; Alexandre Perron[1]; Sebastien Perron[2]; [1]Universite du Quebec a Chicoutimi; [2]Alcan International Ltd.

Bubbles in the Hall-Héroult cells act as a momentum source, but they also represent an additional electrical resistance. In order to understand how bubbles play their role, the evolution of the bubble layer structure – namely the number, size and spatial distribution of the bubbles – need to be known. As direct observation is not possible, we have little information about bubble sizes. Neither small scale electrolysis cells nor real scale water-air models can reproduce correctly the morphology of the bubble layer in the electrolysis cell. A mathematical model using the Lagrangian description of the bubble layer has been built and presented in earlier papers. The simulator was first validated with industrial scale air-water systems then the bubble size distribution was calculated using physical properties of a real cell. This paper presents the impact of cell design on the structure of the bubble layer as computed with our simulator.

4:50 PM
Study on Bubble Behavior on Anode in Aluminum Electrolysis-Part I: *Zhaowen Wang*[1]; Bingliang Gao[1]; Haitao Li[1]; Zhongning Shi[1]; Xiaodong Lu[1]; Zhuxian Qiu[1]; [1]Northeastern University

A tailor made data recorder was used to continuously measure cell voltage fluctuation status during electrolysis in lab scale. Anode gas bubbles generation and departure process that causes cell voltage fluctuation was analyzed. Average values of the voltage peaks were calculated. The effects of anode current density and anode size on bubble size and bubble departure rate were discussed. It was found that bubble size changed largely at different anode current density and also affected by anode sizes. The bubble departure rate was also affected by anode current density and anode size. The bubble-releasing rate became stable at a special current density for certain size of anode.

5:15 PM
Study on Bubble Behavior on Anode in Aluminum Electrolysis-Part II: *Bingliang Gao*[1]; Xianwei Hu[1]; Junli Xu[1]; Zhongning Shi[1]; Zhaowen Wang[1]; Zhuxian Qiu[1]; [1]Northeastern University

The see-through cell was used to investigate the bubble behavior on carbon anode and inert anodes. The observation confirmed that the bubble generation process on metal anode could be divided into three steps: anode oxidation and oxygen generation and bubble departure. The bubbles generated on the carbon anode coalesced into a big bubble before suddenly releasing from the anode. According to the time-cell voltage trace during electrolysis and see-through cell tests, the bubble behavior under the anode showed many differences among three types of anodes, and probably linked to the wettability of the materials by the electrolyte. The see-through cell is a very effective method in studying bubble behavior even at high anode current density.

5:40 PM End

Amiya Mukherjee Symposium on Processing and Mechanical Response of Engineering Materials: Steady State Deformation of Materials - Part 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: 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

Wednesday PM
March 15, 2006
Room: 217C
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Norman Ridley, University of Manchester; K. Ted Hartwig, Texas A&M University

2:00 PM Invited
Creep Mechanism for Grain Growth: *James C. M. Li*[1]; Bhakta B. Rath[2]; [1]University of Rochester; Naval Research Laboratory

Grain growth is usually a diffusion process across grain boundaries. It can be accompanied by subgrain rotation (Li, J. Appl. Phys. 33, 2958 (1962)) or nanograin rotation (Haslam, et al. Mat. Sci. Eng. A318, 293 (2001). However, grain boundary migrati on can be achieved also by dislocation motion or creep. (Li, Trans. TMS-AIME 245, 1591 (1969)) The evidence is the power law relationship between driving force and boundary velocity and an activation energy which approaches that of self diffusion at low driving forces and decreases with increasing driving force. (Rath and Hu, Trans. TMS-AIME, 245, 1577 (1969)). The creep mechanism may involve grain rotation (Li, et al. Acta Metall. 1, 223 (1953)) or may not. (Rath and Hu, cited above). When grain rotation is involved, it

provides a mechanism for the coupling between grain boundary migration and grain rotation. (Cahn and Taylor, Acta Mat. 52, 4887 (2004)).

2:20 PM

Assessment of the Creep Behavior of Molybdenum Silicide Alloys for Ultra-High Temperature Applications: Pascal Jehanno[1]; *Martin C. Heilmaier*[2]; Holger Saage[2]; Mike Boening[1]; Heinrich Kestler[1]; [1]Plansee AG; [2]Otto Von Guericke University

Due to their outstanding mechanical and creep properties refractory metal silicide alloys may be first choice candidates for ultra-high temperature applications in oxidizing environment beyond Ni-base superalloys. The creep behavior of a 3-phase Mo-9Si-8B (at.%) alloy was characterized by tensile and compressive tests with volume fractions of about 15% of Mo_3Si and 30% T2 phase and the remainder being a $Mo_{(ss)}$ solid solution. Two processing variants possessing either an intermetallic compound or a $Mo_{(ss)}$ matrix were comparatively assessed. Values for the stress exponent n and for the activation energy of creep *Q* were determined showing that material with the continuous and ultra-fine Mo matrix (grain size around 1 μm) exhibits superplastic behavior with a (record-breaking) strain to failure of more than 400%. Annealed material with a coarsened microstructure, however, yields promise for satisfactory high temperature creep resistance at temperatures up to 1300°C.

2:40 PM

Microstructure and Damage Tolerance of Al-Cu-Mg-Li Alloys for Age Forming: Marco J. Starink[1]; *Nong Gao*[1]; Nicolas Kamp[2]; Shun Cai Wang[1]; Ian Sinclair[1]; [1]University of Southampton; [2]University of Manchester

Age forming of Al based alloys for damage tolerant applications requires an alloy with good age formability and good post-forming mechanical properties. To investigate optimisation of this balance, several newly designed Al-Cu-Mg-Li (Mn,Zr,Sc) alloys were subjected to artificial ageing representative of age-forming. It was seen that combinations of yield strength and fatigue crack growth resistance could be achieved that are at least comparable to the incumbent damage tolerant material for such applications, whilst creep rates at the ageing temperatures applied were better than those achieved in commercially applied age forming processes of heat treatable Al based alloys. Coarse grain structure and high Li content are associated with good fatigue crack growth resistance but reduce age formability. The underlying physical aspects responsible for the balance between creep rates and resulting properties are discussed.

3:00 PM Invited

A New Approach to Grain Boundary Engineering for Ceramics: *Tadao Watanabe*[1]; Sadahiro Tsurekawa[1]; Varanasi Sri Rama Chandra Murthy[1]; [1]Tohoku University

In recent years, the potential and the usefulness of the grain boundary engineering for polycrystalline and nanocrystalline materials have been well demonstrated for metallic materials by several groups including the authors'. This paper will introduce a new approach and challenge of the grain boundary engineering for ceramics which the authors have recently performed to develop high performance structural ceramics. It was found that the control of oxidation brittleness and improvement in wear resistance of SiC were achieved by controlling the grain boundary microstructure, defined by the grain boundary character distribution (GBCD),the grain boundary connectivity, and the grain boundary density (grain size), at least. A new processing of the grain boundary microstructure based on Reactive Metal Penetration (RMP) was found to produce an extremely high fraction of low-energy fracture resistant grain boundaries, which may result in an improvement of mechanical properties of Al/Al2O3 composites.

3:20 PM Invited

Superplasticity as Scientific and Practical Challenges: *Oskar A. Kaybyshev*[1]; [1]Institute for Metals Superplasticity

The review on the nature of superplastic deformation is presented. The possibilities of low temperature high rate superpasticity occurrence and microstructure refinement as means for converting commercial alloys into superplastic state are analyzed. Data on manufacturing large size billets with nano, submicro and microcrystalline structures are presented. Examples of practical use of superplasticity are provided.

3:40 PM Invited

Diffusion-Controlled Processes on the Grain Boundaries and Plasticity/Superplasticity of Polycrystalline and Nanostructured Metals and Alloys: *Yury R. Kolobov*[1]; Ilya V. Ratochka[2]; [1]Belgorod State University; [2]Institute of Strength Physics and Materials Science Sb RAS

The main both original and literature research results of the peculiarities of the effect of grain boundary diffusion atom flow on structure evolution and mechanical properties of metals and alloys as well as during superplastic deformation have been reviewed. The interconnection and intereffect of diffusion processes, sliding and grain boundary migration as factors, determining the development of plastic deformation at examined conditions are analyzed. The peculiarities of grain boundary diffusion of substitution from impurity environment (coating) in nanostructured metals and alloys relative to the respective ones in coarse grained metals and alloys have been investigated. The physical reasons for considerable (by some orders of magnitude) increase of diffusion penetration of grain boundaries in nanostructured state are discussed. The features of manifestation of low temperature and/or high-strain rata superplasticity in nanostructured metals and alloys have been considered.

4:00 PM Break

4:10 PM Invited

Effect of High Temperature Pre-Straining on Superplastic Properties: *Bhagwati Prasad Kashyap*[1]; [1]Indian Institute of Technology, Bombay

Typical quasi-single phase and two-phase superplastic grade materials were subjected to pre-straining over wide ranges of temperatures and strain rates, including that which belongs to superplastic region. There occurred, texture evolution, grain growth, grain-morphological changes, cavitation etc., some of which were subsequently found to have deleterious effects while others were found to improve superplastic properties. The nature of stress-strain and log(stress)-log(strain rate) curves were analyzed towards quantifying and generalizing the correlations between flow behaviour and microstructural evolution during pre-straining. Superplastic deformation of the pre-strained tensile specimens was found to ascertain steady state flow behaviour to limited extent but, deformation to large strains, once again resulted in strain sensitive flow stress or pseudo-steady state. The effects of pre-straining and non-steady nature at large strains were examined vis-à-vis steady state flow property in order to evolve an indirect measure of microstructural parameters.

4:30 PM Invited

Effect of Strain Rate Path on Cavitation in Superplastic AA5083: *Norman Ridley*[1]; P. S. Bate[1]; B. Zhang[1]; [1]University of Manchester

Previous work on aluminium alloys has demonstrated that rapid pre-straining can lead to enhancement of superplastic(SP) behaviour. Both experimental and modelling studies have shown that the rapid pre-strain is often associated with a reduction in flow stress and an increase in strain rate sensitivity, m, in the later stages of deformation, relative to constant strain rate deformation. The higher value of m leads to a greater uniformity of thickness in a formed part. However, aluminium alloys are prone to cavitation during superplastic flow, and metallographic observations suggest that cavities are most likely to develop at grain boundary particles. The present work investigates the effect of rapid pre-strain on cavitation behaviour in SP AA5083 deformed under both uni-axial and bi-axial conditions, and compares cavity density, size distribution and cavity shape, with that for material deformed under constant strain rate conditions. The mechanism(s) of cavity growth are also examined.

4:50 PM

Grain Boundary Processes in High Temperature Deformation: *Atul H. Chokshi*[1]; [1]Indian Institute of Science

High temperature plastic deformation may occur by intragranular dislocation processes or intergranular processes that depend on grain sizes. Grain boundary sliding is an important mode of deformation in fine grained materials, and it is requirement for superplastic deformation. The occurrence of grain boundary sliding can also lead to premature failure by the nucleation, growth and interlinkage of cavities. Grain boundary processes are also of current interest in examining plastic deformation in nanocrystalline materials. Professor Amiya Mukherjee has made several important contributions to evolution and scientific examanition of high

temperature plastic flow and fracture, and this report will highlight current understanding of such phenomena.

5:10 PM Invited

Transformation Superplasticity of Cast Ti and Ti-6Al-4V with Coarse Grain Size: Qizhen Li[1]; Edward Y. Chen[2]; Doug R. Bice[2]; *David C. Dunand*[1]; [1]Northwestern University; [2]TiTech, International, Inc.

Unlike fine-structure superplasticity which relies on grain-boundary sliding and necessitates fine, stable grains below about 10 micrometers, transformation superplasticy relies on internal stresses produced during thermal cycling around an allotropic transformation temperature, and is thus expected to be active even for very large grain sizes. We test this prediction by subjecting as-cast, coarse-grained CP-Ti and Ti-6Al-4V to thermal cycling under stress, and demonstrate superplastic deformation under both uniaxial deformation and multiaxial dome forming. We compare superplastic properties for the present cast, coarse-grain CP-Ti and Ti-6Al-4V to previous results on powder-metallurgy CP-Ti and Ti-6Al-4V with intermediate grain size.

5:30 PM

Superplasticity and Cooperative Grain Boundary Sliding in Nanocrystalline Ni_3Al: *Nathan A. Mara*[1]; Alla V. Sergueeva[1]; [1]University of California

Cooperative grain boundary sliding (CGBS) has shown to account for the majority of macroscopic strain seen in microcrystalline metallic systems undergoing superplastic deformation. While CGBS has been observed on the surface of microcrystalline samples deforming superplastically through the shifting of diamond scribe lines, there have been no TEM results showing occurrance in the bulk of the material, and the details behind the micromechanism of CGBS. In this work, nanocrystalline Ni_3Al produced via High Pressure Torsion is deformed superplastically in the TEM. High-temperature (~700°C) in-situ tensile testing shows the nature of CGBS at the nanoscale through direct observation of this phenomenon. This investigation is funded by National Science Foundation grant: (NSF-DMR-0240144).

5:50 PM

Constitutive Modeling of Superplastic Deformation of AZ31 Mg Alloy: *Marwan K. Khraisheh*[1]; Fadi Abu-Farha[1]; [1]University of Kentucky

As the lightest constructional metal on earth, magnesium (and its alloys) offers a great potential for weight reduction in the transportation industry. Many automotive components have been already produced from different magnesium alloys, but they are mainly cast components. Production of magnesium outer body components is still hindered by the material's inferior ductility at room temperature. Magnesium alloys are usually warm-formed to overcome this problem; however, it was found that magnesium exhibits superior ductility and superplastic-like behaviour at higher temperatures. In this work, the deformation behaviour of Magnesium Alloy AZ31 is investigated under a wide range of forming temperatures and true strain rates. The results of the mechanical and microstructural tests are used to develop a microstructure-based constitutive model that can capture the behaviour of the material under the various forming conditions. The model is based on the viscoplasticity theory and includes a microstructure-based overstress function.

Biological Materials Science: Functional Biomaterials and Devices

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

Wednesday PM
March 15, 2006
Room: 212A
Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Roger Jagdish Narayan, University of North Carolina

2:00 PM Invited

Nanoporous Materials for Biosensors: *Alex V. Hamza*[1]; Juergen Biener[1]; Andrea M. Hodge[1]; Joel R. Hayes[1]; Sergei O. Kucheyev[1]; Thomas Huser[1]; Chad E. Talley[1]; [1]Lawrence Livermore National Laboratory

The optical properties and chemical stability of noble metal nanoporous materials make them ideal for studying biological systems. The morphology of nanoporous metals allows for an efficient excitation of surface plasmons by photons in the visible spectral range. These surface plasmons are also responsible for the increase in the Raman scattering observed in surface-enhanced Raman scattering (SERS). SERS makes it possible to exploit the chemical specificity inherent in Raman spectroscopy for chemical sensing with low detection limits. Examples of the control over the nanoporous materials that can be achieved and the relationship between the nanoporous structure and SERS signal will be presented. This work was performed under the auspices of U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

2:30 PM

Towards Protein-Based Bio-Electronic Circuit Components: *Jack Adam Tuszynski*[1]; John M. Dixon[1]; [1]University of Alberta

The surface of tubulin molecules exposes a pattern of amino acid residues that provides active sites for nucleation, organization, and binding of metal particles. Under appropriate conditions, every tubulin molecule is able to nucleate silver, gold, platinum and palladium nanoparticles thus forming regular arrays reflecting the tubulin array patterns. This provides a potential approach to develop electronic devices with novel I-V characteristics and attractive physical and biochemical properties such as self-organization and ease of manipulation. Electronic conductivity calculations for the bare MT structure have been based on the Hubbard model in which the dynamic conductivity matrix has been determined using the Kubo formalism in conjunction with the periodic square well approximation. We have investigated MTs decorated with metallic nanostructures using a percolating resistor network approach where metallic wires are interconnected with protein segments represented as insulators with resistance values we have calculated earlier.

2:50 PM

Laser Processing of Advanced Bioceramics: *Roger Jagdish Narayan*[1]; [1]Georgia Institute of Technology

Lasers are also finding greater use in processing biomaterials processing. A relatively new concept in biomaterials is the use of advanced biomaterials, which contain "value-added" features or components. Novel laser processes for creating advanced bioceramics, including diamondlike carbon-metal nanocomposites, hydroxyapatite-osteoblast composites, tissue engineering scaffolds, and microneedles will be discussed. Pulsed laser deposition, matrix assisted pulsed laser deposition-direct write, and two photon induced polymerization processes provide these advanced bioceramics with unique structures and added functionalities for next generation medical and dental applications.

3:10 PM
Nanoindentation of Biomaterials – Novel Applications and Challenges in the Biological Field: *Michelle E. Dickinson*[1]; [1]Hysitron Inc.

Nanoindentation is an accurate and established method for obtaining the nanomechanical properties of traditional homogenous materials. As this technique moves into the biological world, new challenges such as viscoelasticity, heterogeneity and hydration become important considerations. Results from nanoindentation of highly diverse biomaterials ranging from red blood cells and contact lenses to teeth and bones will be discussed. Focus will be given to the specific environmental conditions used to create testing conditions realistic of those *in vivo*. Fixation techniques such as functionalized substrates and hydrated chambers will be described as will different fluid solutions for specialized applications. By discussing the challenges that biomaterials bring to nanomechanical testing and presenting some of the newest solutions to these issues, accurate testing conditions can be developed for future experimental research.

3:30 PM
Nanotechnology-Based Water-Filtering and Purification Solutions for Emerging Biomedical Applications and Advanced Health Benefits: *Ion Nemerenco*[1]; [1]Nanowater

The idea presented in this paper is a novel concept of kremen-activated water-filtering and purification technology shows that a molecule-level mechanism can considerably enhance purity and quality of drinking water via biocatalyzed nanostructuring, positive energizing, and purification of processed water ("nanowater") from any microorganisms or contaminations. It provides normal water with emerging biomedical applications and unique health benefits. The water cleaning technology is based on the bio-catalyst properties of black flint mineral stone ("kremen") found in certain parts of the world. The observed positive effects of nanowater result from its unique capabilities of saturating the human body with oxygen (O2), distributing water molecules evenly across the human body, and increasing the organism's antioxidant complex through better assimilation of regulated nanowater particles which contain health-boosting elements. The health benefits acquired by nanowater create an easy to apply the benefits in areas of human health, agriculture and medicine.

3:50 PM Invited
Effects of Loading Rate on the Mechanical Behavior of a Natural Rigid Composite: S. G. Walter[1]; B. D. Flinn[1]; *George Mayer*[1]; [1]University of Washington

The effects of loading rate variations on the stress-strain behavior, failure mechanisms, and fracture modes of the spicules of the sponge *Euplectella aspergillum* have been investigated. Comparisons were made with similar measurements on a silicate glass. The influence of very thin (5-10 nm layers) of protein that are interspersed with thicker layers of hydrated glass in the concentric ring structure of the spicules provide strong influence on the mechanical behavior. Ramifications for building synthetic composites with similar architecture are discussed.

WEDNESDAY PM

Bulk Metallic Glasses: Processing and Mechanical Behaviors

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

Wednesday PM — Room: 217B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Hahn Choo, University of Tennessee; Yanfei Gao, University of Tennessee

2:00 PM Invited
Ductile Ti-Based Metallic Glass and Composite Alloys: *Faqiang Guo*[1]; Joe Poon[1]; Gary Shiflet[1]; [1]University of Virginia

In this talk we present two kinds (in terms of microstructure) of Ti-based alloys, one is monolithic amorphous alloy and the other one is a composite containing amorphous phase and a/b Ti(Zr) solid solution. These two kinds of alloys share the similar components but differ significantly in individual constituent concentrations. The mechanisms responsible for their respective formation are explored. Mechanical properties, including compressive and tensile test as well as fracture toughness, are evaluated and compared between these two kinds of alloys. Both alloys show impressive plastic deformation under compressive test (up to 10%), especially for the composite alloys, a plastic elongation of around 4% can be readily achieved. These results indicate that both alloys are almost ready-to-be-used as structural materials.

2:25 PM Invited
Two-Glass Phase Formation by Phase Separation and Crystallization Behavior in Ti-Y-Al-Co Alloys: *Do Hyang Kim*[1]; Byung Joo Park[1]; [1]Yonsei University

Recently, it has been shown that phase separation into two-glass phases occurs in several metallic glass forming systems. Since different types of phase separated microstructures have been reported in the previous studies, the research on the effect of alloy compositions on the formation of phase separated microstructures is required. Moreover, crystallization behavior of the phase separated alloys has not been investigated, yet. In the present study, it will be shown that the resulting microstructure after phase separation in (Ti,Y)-Al-Co alloy will appear differently, depending on the alloy composition, i.e. relative amount of two glass phases. If the volume fraction of one glass phase is much higher than the other glass phase, droplet type phase separated microstructure is observed, while if the volume fraction of two glass phases is similar, interconnected type phase separated microstructure is observed. In addition, the crystallization behavior of the phase separated (Ti,Y)-Al-Co alloys will be discussed.

2:50 PM
Development of Low Cost Amorphous Steels: *Justin Lee Cheney*[1]; Kenneth S. Vecchio[1]; Hesham Khalifa[1]; [1]University of California, San Diego

The role of alloying additions on the formation of iron-based metallic glasses was studied by computational analysis of their influence on the alloy's liquidus temperature and ideal solution melting temperature. It was found that alloys having a reduced liquidus temperature relative to the ideal solution liquidus temperature, in combination with sufficient atomic size mismatches, produced bulk metallic glasses most readily. Experimentally, solute elements consisting of Cr, Mo, W, C, and B were varied systematically; the resulting glass forming ability of the alloy produced was measured via differential scanning calorimetry. The alloys produced contained above 60 atomic percent iron, and can be made largely from Fe-based scrap materials, such as cast iron, HSLA steels and structural steel scrap. This approach leads to very low cost Fe-based BMGs. The specific contributions of each alloying element were justified in terms of various modeling techniques, which have been created to theoretically describe glass formation.

3:10 PM
Approaching the Universal Yield Point of Bulk Metallic Glasses from Molecular Dynamics Simulations: *Ju Li*[1]; Futoshi Shimizu[1]; Shigenobu Ogata[2]; Hideo Kaburaki[3]; [1]Ohio State University; [2]Osaka University; [3]Japan Atomic Energy Research Institute

Most bulk metallic glasses yield at about 2% strain in uniaxial tension/compression tests. A careful analysis of the elementary shear behavior in contrast to crystalline concepts such as the generalized stacking fault energy reveals a simple explanation. We perform molecular dynamics simulations on 2-component model systems and a 5-component BMG system, observing and characterizing the nucleation and evolution of shear bands. Despite gross uncertainties in the interatomic interactions and the pre-deformation glass structure, our MD results give a reasonable account of the 2% universal yield point. The general concepts of glass rejuvenation and aging, which we call alienation and recovery processes in the context of intense localized shear, and occurring mainly within a timescale of 1-100 atomic vibration periods, is postulated to play a critical role. This mechanistic model can explain why the yield point is relatively insensitive to the interatomic potential and the structure of thepre-deformed glass.

3:30 PM
Multi-Scale Instrumented Indentation Studies of Deformation in a Zr-Based Bulk Metallic Glass: *Subhaashree Sridharan*[1]; C.

Suryanarayana[1]; Rajan Vaidyanathan[1]; [1]University of Central Florida

We report on the use of spherical diamond indenters of different diameters to probe both elastic and inelastic deformation in a fully amorphous Zr-based alloy. The spherical geometry results in a simpler stress distribution under the indenter (when compared to a sharp geometry) and the choice of diameter controlled the location of the maximum stress below the indenter. While the elastic response did not depend on the diameter of the indenter used, the inelastic response was influenced by a geometrical length scale associated with the evolution and interaction of shear bands and their subsequent propagation to the surface of the specimen. The influence of pre-existing shear bands on the indentation response was also investigated. The observations were substantiated with microscopy and finite element modeling. This work was supported by a grant from NSF (DMR 0314212).

3:50 PM Break

4:00 PM

Mechanical Behaviors of Zr-Based Bulk Metallic Glasses at Cryogenic Temperatures: *Hongqi Li*[1]; Cang Fan[1]; Hahn Choo[1]; Peter K. Liaw[1]; [1]University of Tennessee

In this study, the effect of low temperature on mechanical properties, such as strength, ductility, deformation, and fracture behaviors will be investigated in Zr-based bulk metallic glasses (BMGs) using uniaxial compression testing. Also, the influence of strain-rate on mechanical response at room and cryogenic temperatures is investigated. The Zr-based BMGs were fabricated via an arc-melting system, and X-ray diffraction analysis showed that their microstructures are fully amorphous. The mechanical behavior, especially ductility of BMGs, is related to the local development of shear bands, whose evolution is believed to be associated with the free volume in materials. That is, the diffusion process could be involved. Therefore, it is anticipated that both temperature and strain-rate will play an important role in governing BMGs' mechanical response under loading. This work was supported by the National Science Foundation (NSF) under the grant No. DMR-0231320, with Dr. Carmen Huber as the Program Director.

4:20 PM

Corrosion Resistance of Cu-Zr-Al-Y Bulk Metallic Glasses: *Uwe Koster*[1]; Daniela Zander[1]; [1]University of Dortmund

Bulk metallic glasses based on late transition metals, e.g. Ni or Cu have many potential advantages, such as higher elastic limit and strength, in comparison to those based on early transition metals: $Cu_{46}Zr_{42}Al_7Y_5$ exhibits a tensile fracture strength in excess of 2000 MPa and a Young's modulus of about 120 GPa. Electrochemical tests of amorphous $Cu_{46}Zr_{42}Al_7Y_5$ were conducted by potentiodynamic polarization at room temperature in $NaCl_{aq}$. The influence of corrosion on the surface topography was studied by X-ray diffraction, SEM and TEM. Electrochemical measurements indicate a good corrosion resistance in solutions with low molarity (pH 8) due to the formation of protective oxide films, but no passive layer is formed at high molarity thus resulting in a high susceptibility to pitting. The corrosion mechanisms of formation of oxide films as well as nucleation and growth of pitting were clarified by associating microstructural investigations with the results of the electrochemical measurements.

4:40 PM

Oxidation of Cu-Based Bulk Metallic Glasses: *Uwe Koster*[1]; Monika Meuris[1]; Daniela Zander[1]; [1]University of Dortmund

Excellent mechanical properties combined with good glass forming ability and low cost materials make Cu-based bulk metallic glasses the material of choice for a variety of applications, appropriate oxidation and corrosion behaviour provided. Oxidation reactions were studied in detail in $Cu_{60}Zr_{40}$, $Cu_{50}Zr_{50}$, $Cu_{60}Zr_{30}Ti_{10}$ and $Cu_{46Zr42}Al_7Y_5$ metallic glasses by thermogravimetry and cross sectional scanning and transmission electron microscopy. Since crystallization often interferes with the oxidation, oxidation of already crystallized glasses exhibiting a nanocrystalline structure will be investigated. Whereas the formation of homogeneous oxide scales was observed in many Zr-rich glasses a layered structure was found to develop in Cu-rich glasses with an assembly of Cu-oxide needles at the outer surface and Cu depletion of the inner layer. During ongoing oxidation parts of the scale lose contact due to developing stresses and formation of voids and start to peel off. This behavior will be compared with that of crystalline Cu.

5:00 PM

Cu-Based Bulk Metallic Glass Composites Containing In-Situ TiC and TiB Particles: *Hao Wang*[1]; Huameng Fu[2]; Haifeng Zhang[2]; Zhuangqi Hu[2]; [1]University of Queensland; [2]Chinese Academy of Sciences

Cu-based bulk metallic glasses are of relative low cost and high strength, which have been considered to have more potential for engineering applications. Two Cu-based BMGs containing TiC and TiB particles respectively have been successfully developed using copper mould injection casting. TiC and TiB particles with a size of about 4 mm were in-situ formed and uniformly distributed in the BMG matrix. The volume fraction of the reinforcement particles was up to 30%. The two-phase BMG composites exhibited 2% plastic strain after yielding and the hardness of the materials increased by 25%. On the fracture surface, besides of vein patterns, honeycomb-like characteristics were observed, which might be responsible for the plasticity enhancement. The in-situ particle also restricted shear band propagation and changed its direction, which resulted in an increase of compressive fracture strength. However, a high particle level caused stress concentration on the particle agglomeration and liquid droplets were observed.

5:20 PM

A Finite Element Method for Simulating Inhomogeneous Deformation of Amorphous Alloys: *Yanfei Gao*[1]; [1]University of Tennessee

Inhomogeneous deformation of amorphous alloys is related to the initiation and propagation of shear bands. Based on Spaepen's free volume model and its generalization to multi-axial stress states, this work develops a finite element scheme to model the evolution of free volume and shear bands. The method follows that of a small-strain rate-dependent plasticity theory. The incremental nonlinear equations are solved by the Newton-Raphson method, while the corresponding Jacobian matrix is obtained from the evolution of internal state variables. This micromechanical model allows us to study the interaction between individual shear bands and background stress fields. Numerical examples will be presented.

Carbon Technology: Cathode Preheating/ Wettable Cathodes

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

Wednesday PM Room: 8A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Ketil Rye, Elkem Aluminium ANS

2:00 PM

Thermo-Chemo-Mechanical Modeling of a Hall-Héroult Cell Thermal Bake-Out: *Daniel Richard*[1]; Patrice Goulet[2]; Marc Dupuis[3]; Mario Fafard[2]; [1]Hatch Associates Ltd.; [2]Laval University; [3]GéniSIM Inc.

Start-up of a Hall-Héroult cell is a delicate task. Modern practices for high amperage cells involve preheating the lining before the molten electrolyte is poured in. The optimum preheating method for a rapid production of metal and a long pot life is elusive. Numerical modeling is an invaluable tool to gain insights into the complex phenomena taking place during start-up. The adequate modeling of the mechanical response of the lining is critical to detect risks of cathode block cracking or the development of gaps where liquids could leak. Taking into account the ramming paste baking, the quasi-brittle nature of carbon and the contact interfaces are examples of key elements to consider. A finite element demonstration cell slice model was built and simulations of different thermal bake-out scenarios were performed using the in-house code FESh++. Potential industrial application of the model is discussed.

2:25 PM

Development of Coke-Bed Preheating Method for 200kA Cells: *Shaher Mohamed*[1]; M. M. Megahed[2]; H. Hashem[1]; M. El-Ghonimy[1]; [1]Aluminum Company of Egypt; [2]Cairo University

Many factors affecting pot life of aluminium reduction cells. Preheating and start-up had been evaluated to be of the most effecting parameters on cathode life. Different methods are used to preheat the cells. Coke bed preheating with shunt rheostat is one of the most popular methods. The Aluminium Company of Egypt (Egyptalum) utilizes coke bed for preheating of aluminium reduction cells since 1991. Several modifications on the preheating process had been done. Coke bed height modified from 50 to 30 to less than 20mm now. To reduce the preheating current different designs of shunting units have been used. This paper describes the different preheating conditions, as well as analysis and discussions of preheating measurements. A finite element model is used to evaluate the different preheating schemes. The results indicate that the best scheme is the one who has less coke bed and smooth current increase.

2:50 PM

Study on the Heating-Up Rate for Coke Bed Preheating of Aluminum Reduction Cell: Jie Li[1]; *Qinsong Zhang*[1]; Yanqing Lai[1]; Yexiang Liu[1]; [1]Central South University

Impacts of shunting schedules on the heating-up rate during coke preheating and heating-up rate variation of temperature are of great importance to get the proper heating-up rate and prolong the cell life during and after the preheating process. A nonlinear transient heat transfer model of a 160kA prebaked cell was developed by ANSYS codes. Results show that longer interval and more shunt groups resulted in slower average heating-up rate. The average heating-up rate from 200 to 600°C had the slowest value at some starting current percentage, which was approximate to 50% of the whole current. Concerned with the same cell structure and coke-bed thickness, the heating-up rate of certain period of time was dependent on time, temperature and current but not on the shunting schedules after the current loading moment. A shunting schedules design method was proposed to meet the given requirement of heating-up rate during any temperature intervals.

3:15 PM

Titanium Diboride and Wolfram Silicide Composite Used as Aluminum Electrolysis Inert Cathode Materials: *Huimin Lu*[1]; Huanqing Han[1]; Ruixin Ma[1]; Dingfan Qiu[2]; [1]University of Science and Technology Beijing; [2]Beijing General Research Institute of Mining and Metallurgy

In this paper, various performances of TiB2-WSi2 composite with WSi2 content 10mass%, 30mass%, 50mass% and 70mass% are studied. The results show that WSi2 can improve TiB2 sintering performance apparently, when WSi2 content of TiB2- WSi2 composite excesses 30mass% the compactivity of the composite sintered 1hour at 1900ºC is higher than 98%; its conductibility is prior to TiB2; WSi2 exhibits significantly better resistance aluminum corrosion but weaker resistance cryolite corrosion than TiB2. Aluminum corrosion to TiB2- WSi2 composite is mainly that Al and TiB2 reaction becomes TiAl and so on intermetallics, but cryolite corrosion to composite is mainly that WSi2 dissolves and fractures in the cyolite solution. Because in aluminum electrolysis process, there is a thin aluminum liquid layer on the inert cathode surface, the inert cathode failure is mainly aluminum liquid corrosion, so TiB2- WSi2 composite is a better inert cathode material for aluminum electrolysis.

3:40 PM Break

3:55 PM

Stability of TiB2-C Composite Coatings: *Mohamed Ibrahiem*[1]; Trygve Foosnaes[1]; Harald A. Oye[1]; [1]Norwegian University of Science and Technology

Several recipes of pitch-bonded TiB_2 were studied with respect to adherence, cracking and stability during electrolysis. A successful recipe for a crack-free coating was obtained. The coating showed good adherence and stability after 34 hours of electrolysis. The wetting of the coating by aluminium in the presence of cryolite melt was time dependent and almost complete wetting was observed after 120 minutes. Furan resin-based TiB_2 coatings were not wetted by aluminium during electrolysis due to the presence of a carbon layer at the coating surface. Coating samples were polished by SiC paper after curing. The polished samples showed good stability and aluminium wetting.

Cast Shop Technology: Cast Processes and Chain Analysis

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

Wednesday PM — Room: 7C
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Bjorn Rune Henriksen, Elkem ASA

2:00 PM

Copper Shells for Twin Roll Casting: *Hans-Guenter Wobker*[1]; Gerhard Hugenschuett[1]; Dietmar Kolbeck[1]; [1]KM Europa Metal AG

The drawback of the twin-roll method of aluminium strip casting as compared with other casting processes has been low productivity. The cause of these productivity limits has been mainly in the difficulty of removing the process heat from the zone of melt/strip – roll contact. Substitution of the steel alloys used today by high-conductivity engineering materials opens up very interesting possibilities of improving the productive capacity of such casting systems. The present paper gives an account of the development, properties and application of Cu based materials for casting roll sleeves. The first part portrays the Cu materials that have been developed and their particular material properties. The second part highlights performance results achieved with Cu alloy casting sleeves when used in actual practice. It describes the casting speeds/productivity levels which have been reached, the wear characteristics of such sleeves, and the properties of the produced cast Al-strip.

2:25 PM

A New Continuous Casting Process: *Hubert Sommerhofer*[1]; Peter Sommerhofer[1]; [1]Sommerhofer Technologies GmbH

Continuous casting using water as coolant has some important disadvantages. Water is evaporating at low temperatures and causes difficulties in process control and product quality. Furthermore the use of water as cooling material causes the danger of explosions. To prevent these disadvantages, we use liquid metal to cool the strand. Laboratory scale experiments have been done to investigate the possibility to cast an aluminiumalloy, a magnesiumalloy and copper using a low melting liquid metal as cooling material. Now a pilot scale plant has been constructed by Sommerhofer Technologies. After several tests with the pilot scale plant, results on the castability of Aluminium- and Magnesiumalloys are existing now.Advantages of the new process: Much lower crack risk; constant high heat transfer coefficients; higher casting rates; larger process window for the coolant temperatures; nearly no subsurface layer; no explosions; no contaminated cooling water; exchanged heat at usable temperature level.

2:50 PM

Maximizing the Quality of DC Cast Aluminum Billet: *Roger A. P. Fielding*[1]; [1]Benchmarks

The quality of DC cast aluminum billet is affected by the control of the incoming materials: smelter ingot, scrap aluminum, and of the alloying and grain refining additions to the melt. The quality of the billet is affected by the preparation and treatment of the incoming materials; by the design and operation of the melting and holding furnaces, the metal treatment systems, the casting and homogenizing equipment, and the training and skills of the operators. Failure to understand the impact of the various factors affecting the quality of the billet results in improperly engineered facilities whose features adversely affect the quality of the billet, increase the cost of converting the incoming materials to billet, and compromise the profitability of the facility.

3:15 PM Break

3:30 PM

Turnkey Solutions for the Modern Day Primary Aluminium Casthouse: The Alba Line 5 Experience: *Andrew Haberl*[1]; Barry J. Houghton[1]; Hussain Hussan Al-Ali[2]; [1]Solios Thermal; [2]Aluminium Bahrain B.S.C.(c)

Placing a turnkey order with one supplier for all the equipment that interfaces together, as on a casting line, is a very cost effective means of providing an efficient and flexible Casthouse Solution. Decisions can be taken more quickly, interface issues can be resolved with minimal recourse to the customer and delays and cost over runs can be minimised. In 2003 ALBA one of the world's leading aluminium producers, gave Solios Thermal the opportunity to demonstrate their ability to deliver when they awarded them the contract for turnkey supply of the casting lines for their Line 5 expansion. The scope of supply included 11 – furnaces, 3 – VDC casters, 1 – Continuous ingot caster, 2 – electromagnetic stirrers and all interconnecting launders, associated degassing and filtration units. Various key stage decisions, layout considerations, and start up together with the benefits to the end user are discussed in this paper.

3:55 PM

Industrial Application of DOE Energy Savings Technologies to Aluminum Melting: *Cynthia K. Belt*[1]; Brian M. Golchert[2]; Paul E. King[3]; Ray D. Peterson[1]; Joseph L. Tessandori[1]; [1]Aleris International; [2]Fluent Inc; [3]U.S. Department of Energy

Aleris International was part of a four-year DOE program on "Improving Energy Efficiency in Aluminum Melting". Key findings from this work wereinterpreted and implemented on a corporate-wide basis. 1) Natural gas savings were realized by adjusting burner input based on the bath area for melting and holding furnaces. 2) Modifying burner loading for an asymmetrical and symmetrical reverbatory furnace was investigated. 3) The PHAST program was utilized to understand and quantify the potential of preheated combustion air. Additional modeling was done for this paper to analyze response from a different furnace design. Methods and results will be discussed in this paper.

4:20 PM Break

4:35 PM

A Quick-Change Ceramic Filter Assembly for Filtering Molten Aluminum and Other Metals: *David A. Larsen*[1]; Dean Vander Jagt[2]; [1]Blasch Precision Ceramics; [2]New Century Heaters Inc.

A new concept of a quick-change interchangeable ceramic filter assembly for filtration of molten aluminum and other metals is described. The ceramic filter cartridge assembly may be replaced without draining the molten metal filter box, and without permitting unfiltered metal to pass downstream. Oxygen/air is purged from this ceramic filter, and it is preheated before immersion into the molten metal. The purging prevents oxides from forming during priming of the filter, which would plug filter pores, and the preheating prevents thermal shocking. The filter cartridge thus can be easily interchanged without manual removal of surface oxides or corundum, and without contamination of the melt.

5:00 PM

Modeling Methods for Managing Raw Material Compositional Uncertainty in Alloy Production: *Gabrielle Gaustad*[1]; Preston Li[1]; Randolph Kirchain[1]; [1]Massachusetts Institute of Technology

Operational uncertainties create inefficiencies in metal alloy production. One that greatly influences remelter batch optimization is variation in raw material composition, particularly for secondary materials. Currently, to accommodate compositional variation, firms commonly set production targets well inside of the window of compositional specification required for performance reasons. Window narrowing, while effective, does not make use of statistical sampling data, leading to sub-optimal usage of secondary materials. This paper explores the use of a chance constrained optimization method, which allows explicit consideration of statistical information on composition. The framework and a case study of cast and wrought production with available scrap materials are presented. Results show that it is possible to increase scrap consumption without compromising the likelihood of batch errors, when using this method compared to conventional window narrowing.

Characterization of Minerals, Metals and Materials: Composites and Other Materials

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

Wednesday PM — Room: 206A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Tzong T. Chen, Natural Resources Canada; Jiann-Yang James Hwang, Michigan Technological University

2:00 PM

Pullout Test of Coir Fiber to Evaluate the Interface Strength in Polyester Composites: *Sergio Neves Monteiro*[1]; Janine Feitosa de Deus[1]; Regina Coeli Paes Aquino[2]; Jose Roberto Moraes d'Almeida[3]; [1]State University of the Northern Rio de Janeiro; [2]Federal Center for Technological Education; [3]Pontificia Universidade Catolica do Rio de Janeiro

Polymeric composites reinforced with natural fibers are increasingly used in several engineering applications, from automotive parts to building construction elements. In addition to the relatively low cost, the natural fibers are also appraised by their renewable characteristics and biodegradability. Fibers extracted from the crust of coconut fruits, also known as coir fibers, have been investigated for a possible use in composite materials. The present work evaluated the coir fiber/polyester matrix interface shear strength by means of pullout tests. The variation of the pullout tensile stress vs. the embedded coir fiber length permitted to determine the critical fiber length and thus to calculate the interface strength. Observations by SEM complemented the results from pullout tests, indicating that coir fibers have a potential for application in polymeric composites that could replace conventional materials such as wooden and gypsum panels.

2:25 PM

Structural and Morphologic Characterization of the Natural Graphite after Application of High Pressure and High Temperature Treatment: *Ana Lucia Diegues Skury*[1]; Sergio Neves Monteiro[1]; [1]UENF

In the synthesis of diamond, the precursor graphite must present a degree of crystalinity to asure optimum yield and quality for the transformed crystals. X-ray diffraction, SEM and TEM analyses were carried out to investigate the structural an morphologic transformations produced in natural graphite by high pressure-high temperature (HPHT) treatment. The present study had as its objective to characterize the morphological and structural changes suferred by a natural graphite that was subject to HPHT in the region of diamond thermodinamic stability. The results indicate the material that constitutes the gasket for the reaction capsule, could act as catalyst in the graphite recrystallization observed after the treatments.

2:50 PM

Environmentally Correct Jute Reinforced Polyethylene Composites: *Sergio Neves Monteiro*[1]; Amanda Camerini Lima[1]; Lucio Jose Terra Petrucci[1]; Luiz Augusto Hernandez Terrones[1]; Jose Roberto Moraes d'Almeida[2]; [1]State University of the Northern Rio de Janeiro; [2]Pontificia Universidade Catolica do Rio de Janeiro

Wastes generated by our society and the question on how to dispose them is, nowadays, a relevant matter of concern. The objective of the present work was to characterize the technical properties of composites made of discarded jute bags, with their plain weave fabric, serving as reinforcement phase, and polyethylene wastes, collected from city dumps, as continuous matrix. Different amounts, up to 30 wt%, of jute plain weave fabric were press molded at 160°C together with sieved polyethylene particles. The resulting composites showed mechanical properties that could compete with those of low density wooden boards. Both by presenting a solution to, otherwise, disposed wastes and by helping to prevent deforestation, the investigated composite may be considered as an environmentally correct material.

3:15 PM
Microstructural Characterization of Ice: *Ian Baker*[1]; R. Obbard[1]; K. Seig[1]; D. Iliescu[1]; C. P. Dahglian[1]; [1]Dartmouth College

In this presentation we describe techniques that have been developed to characterize the microstructures of ice and firn from polar ice sheets and seasonal ice from temperate lakes and rivers. Characterization includes controlled sublimation of uncoated ice in an environmental cold-stage-equipped SEM equipped with an energy dispersive x-ray spectrometer to examine impurity locations in ice and an electron backscatter pattern system to map orientations. Confocal scanning optical microscopy coupled with Raman spectroscopy has also been used to determine the compounds present in grain boundaries and triple junctions. The limitations of the techniques are discussed. Supported by ARO contract DAAD 19-03-1-0110 and NSF grant OPP-0440523.

3:40 PM Break

3:50 PM
Mechanical and Structural Characterization of Curaua Fibers: *Sergio Neves Monteiro*[1]; Janine Feitosa de Deus[1]; Jose Roberto Moraes d'Almeida[2]; [1]State University of the Northern Rio de Janeiro; [2]Pontificia Universidade Catolica do Rio de Janeiro

Natural lignocellulosic fibers are successfully replacing synthetic fibers in many engineering applications. In addition to well known conventional fibers such as cotton, sisal and lax, others with promising properties are now being considered. The present work investigated the mechanical and structural characteristics of a relatively high strength fiber, curaua (Ananas erectifolius), from the Amazon region in Brazil. The results obtained from tensile tests confirmed the superior strength while scanning electron microscopy (SEM) analysis displayed features that justify the possibility of using the curaua fibers as an effective reinforcement for polymeric composites.

4:15 PM
Microstructural Characterization of Epoxy Matrix Composites with Dispersed Diamond Particles: *Sergio Neves Monteiro*[1]; Ana Lucia Diegues Skury[1]; Gustavo Wagner Menezes[1]; Ruben Sanchez Rodrigues[1]; [1]UENF

Polymeric matrix composites with dispersed hard particles are being used as wear resistant tool in industrial polishing operations. In the present work the microstructure of epoxy composites with different amounts of embeded diamond particles, up to 30%, was investigated for the effect on the fracture characteristics. It was found that introduction of diamond particles promoted an increase in the porosity of the epoxy matrix. Moreover, the particle morphology associate with a non-uniform distribution within the matrix, were responsible for nucleation of cracks, that caused the rupture of the composite.

4:40 PM
Morphological, Mechanical and Optical Characterization of Flyash Filled Nylon-6: *P. A. Mahanwar*[1]; Suryasarathi Bose[2]; H. Raghu[1]; [1]Mumbai University; [2]Indian Institute of Technology, Bombay

Flyash were melt mixed with nylon-6 at a 30wt% concentration. TYZOR® TPT, a coupling agent, was used to facilitate the link between filler and matrix. The morphological characterization, using SEM, revealed sufficient matrix residue on the filler surfaces thus confirming the strong interfacial bonding between the filler and the matrix on treating. Tensile, impact and the flexural properties of the composites were also improved with the incorporation of coupling agent. As flyash particles tend to impart a grayish color to the plastic, TiO2 pigment was employed to make the formulations aesthetic. The pigment was added to the filled system in 0.1, 0.5, 1.0, 1.5, 2.0 wt/wt ratios. Scattering of incident light and spectral reflectance was higher for smaller particle size flyash. Moreover an increase in the scattering with increase in pigment concentration indicates better dispersion of the pigment, which again is due to the presence of coupling agent.

5:05 PM
Texture Gradient in OFHC Copper Processed by Equal Channel Angular Extrusion: *Daudi R. Waryoba*[1]; P. N. Kalu[1]; [1]Florida State University

Most deformation processes such as rolling, forging and extrusion, produce bulk through-thickness microstructure and texture gradient, in addition to the local inhomogeneities. Microstructural and textural gradient that has been observed in wire drawing is attributable to the inhomogeneous deformation along the radial direction. This inhomogeneity in the microstructure is visible as three distinct concentric regions: the inner core, the mid section, and the outer surface. Such microstructural inhomogeneities have been observed to adversely contribute to recrystallization heterogeneity. In the last three decades, equal channel angular extrusion (ECAE) has received a considerable attention as one of the methods for producing ultrafine grain structures. Apart from its effectiveness of producing bulk grain refinement, ECAE results in a homogeneous microstructure and nearly random texture. The goal of this investigation was to study texture gradient in oxygen free high conductivity (OFHC) copper after being deformed by ECAE. Texture gradient is discussed based on electron backscattered diffraction (EBSD) measurements, and results are compared with gradient observed in wire drawn materials.

Computational Thermodynamics and Phase Transformations: Atomic Kinetics Processes - Joint Session with Point Defects in Materials

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

Wednesday PM — Room: 210B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Adri C. van Duin, California Institute of Technology; Mark D. Asta, University of California

2:00 PM Invited
Materials Analysis at the Atomic Scale: The Role of Atom Probe Tomography in Materials Modeling: *Thomas F. Kelly*[1]; David J. Larson[1]; [1]Imago Scientific Instruments

Atom probe tomography (APT) provides 3-D atomic-scale structural and compositional analysis of materials. The Local Electrode Atom Probe or LEAP® made by Imago Scientific Instruments, greatly increases the capacity of APT for materials analyses on a wide spectrum of materials types. Because of the close match between the size of APT data sets and the capabilities of materials modeling approaches, they are a natural complement to each other. Materials modeling is of great interest for tracking of structural evolution in time or properties estimation based on atomic-scale information. A selection of materials analyses will be shown and a few examples of this complementarity will be highlighted.

2:30 PM Invited
Impact of Vacancy Diffusion on the Early Decomposition Stages of Alloys and the Role of Heterophase Interfaces on Coarsening: Zugang Mao[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; *David N. Seidman*[1]; Georges P. Martin[1]; [1]Northwestern University

The kinetic pathway for nucleation and growth is commonly thought to be dictated by the initial supersaturation of solutes in the solid solution undergoing phase separation. We demonstrate that the details of the diffusion mechanism, in the solid solution affect deeply the early stage morphologies of precipitates. Our argument is based on a combined use of atomic-scale observations, with atom-probe tomography (APT) and lattice kinetic Monte Carlo simulation of a Ni(Al,Cr) alloy. By an optimized choice of thermodynamic and kinetic parameters we first reproduce the experimental APT observations. We then modify only the long-range vacancy-solute binding energy, without altering the thermodynamic driving force for phase separation, thereby demonstrating that the microstructural evolution changes from a coagulation to an evaporation-condensation coalescence mechanism. The changes can only be accounted for with non-

zero values for the vacancy chemical potential and off-diagonal terms of the Onsager matrix, at variance with classical models.

3:00 PM Invited
Diffusion in Multicomponent Solids from First-Principles: *Anton Van der Ven*[1]; [1]University of Michigan

Atomic diffusion plays an important role in determining both the mechanisms as well as the rates of many phase transformations in multi-component solids. In this talk, I will describe how diffusion coefficients can be calculated from first-principles in multi-component solids. Kubo-Green relations from statistical mechanics provide the link between macroscopic kinetic quantities such as diffusion coefficients and properties of the solid at the atomic scale. Multi-component solids are characterized by differing degrees of short and long-range order and diffusing atoms sample many different local environments along their trajectories. The cluster expansion from alloy theory is ideally suited to extrapolate first-principles energies and activation barriers to describe the activation barriers for any state of configurational disorder. Combined with kinetic Monte Carlo simulations, this approach enables the first-principles calculation of diffusion coefficients as a function of concentration and temperature in multi-component solids.

3:30 PM Invited
Kinetic of Fe Alloys under Aging and Irradiation Based on Ab Initio and Kinetic Monte Carlo Simulations: *Christophe Domain*[1]; [1]Electricite De France

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 and of the distribution of the alloying elements. In order to model the evolution of pressure vessel steels under ageing or under irradiation, kinetic Monte Carlo simulations have been performed. The energetic of systems containing Cu, Ni, Mn, Si, P as well as C and the activation barriers for migration (which depend on the local environment) have been derived from ab initio calculations and compared to thermodynamical and as well as diffusion based experimental data. As the link between the ab initio results and the kinetic Monte Carlo parameterisation is not straightforward several strategies have been adopted. The effect of these models on the kinetic pathways is discussed. The simulation results are compared to model experiments on simple model alloys.

4:00 PM Break

4:10 PM Invited
Application of ReaxFF Reactive Force Fields to Chemical Diffusion of Hydrogen and Oxygen through Fuel Cell Membranes: *Adri C. van Duin*[1]; Valeria Molinero[1]; Xin Zhang[1]; Boris Merinov[1]; William A. Goddard[1]; [1]California Institute of Technology

ReaxFF is a bond-order dependent force field that can perform reactive simulations on systems too large to be amenable to quantumchemical (QM) simulations. By fitting ReaxFF parameters to extensive databases, derived from QM simulations on small molecules and condensed phase systems and covering both ground state molecules and full reaction pathways, we have developed reactive potentials for a wide range of materials, including organic compounds, metals/metal oxides and semiconductors and interactions between these material classes. Here we present applications of several recently developed ReaxFF potentials to hydrogen and oxygen diffusion through fuel cell membranes. For the membrane material a wide range of compositions are considered, including imidazoles, water-filled zeolites, yttrium stabilized zirconia and yttrium-doped barium zirconates.

4:40 PM Invited
Abnormal Grain Growth Due to Boundary Mobility Variations: *Elizabeth A. Holm*[1]; Mark A. Miodownik[2]; Kristopher Healey[1]; [1]Sandia National Laboratories; [2]King's College London

Abnormal grain growth occurs in many engineered materials and can be desirable or detrimental. Many theories attempt to explain this phenomenon, and it is likely that there are several causes. In pure polycrystals, one mechanism for abnormal growth is the existence of special, high mobility grain boundaries. If such boundaries can persist during evolution, they impart a significant growth advantage to certain grains. We apply highly realistic, microstructural-scale computer simulations to examine abnormal growth in several systems, including textured polycrystals, deformed polycrystals with spatial orientation gradients, and thermally inhomogeneous polycrystals. We find abnormal events similar in morphology, frequency, and crystallography to those observed in experiments. The key requirement for abnormal growth is the persistence of high mobility boundaries due to crystallographic, spatial, or thermal considerations. In contrast, systems with random high mobility boundaries show no abnormal growth. These simulations provide new insight into one mechanism for abnormal grain growth.

5:10 PM
Computing the Mobility of Grain Boundaries: Koenraad G. F. Janssens[1]; *Elizabeth A. Holm*[1]; Steven J. Plimpton[1]; Stephen M. Foiles[1]; David Olmsted[1]; [1]Sandia National Laboratories

Many modern technical materials are designed and manufactured by controlling the evolution of the microstructure. Most of the solid-state microstructure transformations, like recrystallization, grain growth, phase transformation or precipitation, involve the motion of grain boundaries. The mobility of a solid-state grain or phase boundary is determined by the atomistic mechanisms by which the boundary moves. While uncertainty remains about the exact nature of these mechanisms, it is generally accepted that mobility strongly depends on the crystallographic misorientation between neighboring grains. Current experimental and simulation methods cannot determine the mobility of flat boundaries across the large misorientation phase space. We have developed a new, molecular dynamics method for imposing an artificial driving force on boundaries. This allows us to induce motion in flat boundaries of arbitrary misorientation using considerably smaller systems and shorter times than previously attainable. For different series of boundaries, we find both expected results and unexpected results.

5:30 PM
Spatial and Temporal Analysis of Nonequilibrium Order Fluctuations in Alloys Driven by Energetic Ion Irradiation: *Pascal M. Bellon*[1]; Jia Ye[1]; Yaofeng Chen[1]; [1]University of Illinois

External driving forces often leads to the self-organization of alloys microstructures. Using analytical models and atomistic simulations, we recently predicted that ion beam processing can lead to the spontaneous formation of nanoscale patterns of the degree of chemical order. One fundamental question relates to the dynamics of order fluctuations in these nonequilibrium states. This would provide new insight on the origin of the stabilization of these patterns, on the selection of length scales, and, more practically, on the stability of these nanostructures when used in engineering materials. Using atomistic kinetic Monte Carlo simulations, we performed a spatial and temporal analysis of the nonequilibrium fluctuations of order in a binary alloy that would form an L12 ordered structure at equilibrium. This analysis provides new and unambiguous criteria for identifying states of pattern of order, both in simulations and experimentally, and it sheds new light on the origin of their formation.

5:50 PM
Precipitation Kinetics in a Cu-4 wt.% Ti Alloy: *Victor Manuel Lopez-Hirata*[1]; Felipe Hernandez-Santiago[1]; Maribel Leticia Saucedo-Muñoz[1]; [1]Instituto Politecnico Nacional

The precipitation kinetics was studied using SEM, TEM, XRD and Vickers hardness in an isothermally aged Cu-4 wt. % Ti alloy. The XRD and TEM results indicated that the phase decomposition occurred by spinodal decomposition during the early stages of aging. The growth kinetics of composition modulation wavelength is very slow at the early stages of aging. The precipitation of metaestable Cu4Ti phase preceded to that of the equilibrium Cu3Ti phase, which formed through cellular precipitation. The coarsening process of Cu4Ti phase followed the LSW theory for diffusion-controlled growth. The activation energy for this coarsening process was determined to be about 190 kJ/mol. The discontinuous precipitation of Cu3Ti phase has an activation energy of about 207 kJ/mol and an exponent time of about one. The highest hardness and fastest transformation kinetics occurred at aging temperatures of 673 and 873 K, respectively.

Computational Thermodynamics and Phase Transformations: Phase Field Models II

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

Wednesday PM — Room: 210A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Ken R. Elder, Oakland University; Adam C. Powell, Massachusetts Institute of Technology

2:00 PM Invited
Multiscale Modeling of Solid-Solid Phase Transformations: *Valery I. Levitas*[1]; [1]Texas Tech University

Several approaches to continuum modeling of phase transformations are analyzed. To model martensitic microstructure formation, nanoscale and microscale phase field theories are developed. They are based on the combination of a continuum thermodynamic approach, diffuse interface and a material instability concept. These approaches are applied to the martensite nucleation at various dislocation configurations and formation of multivariant stress-induced martensite in single and polycrystals. An alternative sharp interface approach is developed which takes into account large strains and interaction between phase transformation and plastic straining. A general theory of phase transformations in plastic materials is developed which includes phase transformation criterion and extremum principle for determination of all unknown parameters. Nucleation and growth of martensitic plate and strain-induced nucleation at shear-band intersection is studied.

2:30 PM Invited
Modeling Elastic and Plastic Deformation of Nanostructured Materials: Peter Stefanovic[1]; Mikko Haataja[2]; *Nikolas Provatas*[1]; [1]McMaster University; [2]Princeton University

A continuum field theory approach is presented for modeling elastic and plastic deformation, free surfaces, and multiple crystal orientations in nano-crystalline systems with both hexagonal and cubic symmetry in two and three spatial dimensions. The model is based on a free energy for the local atomic density, which is minimized by spatially periodic structures. It incorporates both elastic phenomena (deviations from the preferred spatial period cost energy) as well as defects in the form of, e.g., vacancies, dislocations, and grain boundaries. Furthermore, its dynamics is constructed such that it incorporates both diffusive and elastic relaxation phenomena. By introducing a variable elastic time scale, we are able to maintain mechanical equilibrium while simulating microstructural evolution on time scales well beyond those accessible by conventional atomistic MD simulation methods. We apply this model to elucidate the role of dislocations during deformation of nanocrystalline materials.

3:00 PM
Applications of Moving Mesh Method to Phase Field Simulations: *Weiming Feng*[1]; Peng Yu[1]; Shenyang Hu[2]; Zikui Liu[1]; Qiang Du[1]; Longqing Chen[1]; [1]Pennsylvania State University; [2]Los Alamos National Laboratory

Phase-field simulations have increasingly been used in modeling microstructure evolution during phase transformations and structural coarsening. However, large-scale simulations are still computationally expensive. The semi-implicit Fourier spectral method (FS_implicit) is found to be particularly efficient for this problem. One of the main features for the spectral method is the use of uniform grids that utilizes existing Fast Fourier Transform (FFT) packages. In this presentation, we report our recent progress in making grid points spatially adaptive in the physical domain while maintaining a uniform grid in the computational domain and semi-implicit in time. The new scheme not only provides a more accurate resolution around the interfaces but also retains the numerical efficiency of the FS_implicit method. Numerical examples using the new moving mesh method will be presented for both two and three space dimensions. We will compare the accuracy and efficiency of our results with those obtained by other methods.

3:20 PM
Solving Phase Transformation Problems with FiPy: *Jonathan E. Guyer*[1]; Daniel Wheeler[1]; James A. Warren[1]; [1]National Institute of Standards and Technology

The solution of coupled sets of partial differential equations (PDEs) is ubiquitous in continuum models of phase transformations, such as in phase field or level set simulations. We have developed a phase transformation modeling framework called FiPy that combines the finite volume method, widely used in fluid dynamics, and the Python scripting language, which has enjoyed considerable attention from the scientific community. The result is a tool that is extensible, powerful, freely available and, most importantly, tailored to the needs of materials scientists. The framework includes terms for transient diffusion, convection, and standard sources, enabling the solution of arbitrary combinations of coupled elliptic, hyperbolic and parabolic PDEs, including higher-order expressions such as Cahn-Hilliard. We will present the representation and results of current models, including phase field and level set treatments of electrochemical, polycrystalline, and multi-component dendritic phase transformations.

3:40 PM Break

4:00 PM
Phase Field Modeling of γ' Rafting in Single Crystal Ni-Base Superalloys: *Ning Zhou*[1]; Chen Shen[1]; Mike Mills[1]; Yunzhi Wang[1]; [1]Ohio State University

Rafting of γ' precipitates in single crystal Ni-base superalloys under external load is investigated by computer simulations. The simulation technique is based on an integrated phase field model that characterizes simultaneously spatio-temperal evolution of both precipitate morphology and dislocation structures. The initial γ/γ' microstructure consisting of cuboidal particles and dislocation configuration in the γ-channels are constructed according to experimental observations and phase field simulation of dislocation filling process in the γ-channels. The spatial variation of chemical potential of solute atoms is evaluated based on local concentration and stress, and diffusion fluxes in different γ-channels are analyzed. For a given state (sign and magnitude) of lattice misfit and external load, the predicted morphologies of the rafted γ' precipitates agree well with experiment observations. The kinetics of rafting is also characterized as a function of lattice misfit and external load for a binary Ni-Al alloy with an effective diffusivity calibrated against experiment.

4:20 PM
Phase Field Modeling of High-Temperature Electrochemistry: Application to Subhalide Reduction of Titanium: *Wanida Pongsaksawad*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology

Phase field model of transport-limited with rapid-charge redistribution electrochemistry is used to simulate magnesiothermic reduction of TiCl2. This model derived from the Cahn-Hilliard and Poisson equations can capture both electrolysis and electronically-mediated reactions (EMR) which play a major role in this process. For a solid-solid system, the Peclet number, dimensionless electrode separation and metal/electrolyte conductivity ratio control the cathode interface stability; in a liquid-liquid system, modeled by coupling with the Navier-Stokes equations, the Schmidt number and a modified Weber number also play a role as flow provides an additional stabilizing mechanism. Preliminary 2D simulation results of a solid-liquid system with fluid flow are presented and its stability criteria are discussed. Three-dimensional results without flow illustrate formation of the titanium structure. This methodology can be used to model shape and topology changes in any electrochemical system satisfying the above assumptions.

4:40 PM
Phase Field Model of Solid-State Sintering: *Yu U. Wang*[1]; [1]Virginia Tech

Sintering is a well-known complicated material process involving multiple diffusion mechanisms (along surface and grain boundary, through lattice and vapor), grain boundary migration, particle rigid-body translation and rotation. The high-diffusivity paths along the solid-solid inter-

faces, i.e. grain boundaries, play a key role in the densification of sintered powder compact. The diffusion of atoms from grain boundaries to nearby high-curvature growing neck surfaces leads to approaching of centers of particles through rigid-body motions. This work develops a phase field model of solid-state sintering, which treats the rigid-body motions of particles and surface diffusion, grain boundary diffusion, volume diffusion, vapor transport, as well as grain boundary migration. Consideration of rigid-body motions results in modifications to both Cahn-Hilliard nonlinear diffusion equation and Ginzburg-Landau (Allen-Cahn) structural relaxation equation. Computer simulations are presented.

5:00 PM

Phase-Field Modeling of Homogenization Process of Binary Aluminum Alloys: *Igor Kovacevic*[1]; Bozidar Sarler[1]; [1]Nova Gorica Polytechnic

The simulation of homogenization process of aluminum alloys is made by the phase-field model. The model is focused on the dissolution kinetics of interdendritic eutectic phase in industrial conditions of homogenization. The simulation is performed in two places of the billet, the center and the surface. The realistic industrial direct-chill as-cast microstructure is used as the initial condition for simulation of homogenization. The initial concentration profile of alloying element is obtained by the Scheil-Gulliver solidification model. The thermo physical properties of material are obtained by the thermodynamic database JMatPro for aluminum alloys. The microstructure evolution of Al-5% wt Cu binary alloys during homogenization process in both simulated places is modeled.

5:20 PM

Phase Field Simulations for Grain Growth in Polycrystalline Films: *Nele Moelans*[1]; Bart Blanpain[1]; Patrick Wollants[1]; [1]K. U. Leuven

Grain size, grain size distribution and grain orientation in polycrystalline films strongly influence their strength, electronic properties and durability. Once the grain size is larger than the thickness of the film, grains become columnar with their grain boundaries perpendicular to the plane of the film. As a result, grain growth shows many 2-dimensional features, although is not really 2-dimensional because of the importance of surface energy. Grooves at the triple lines where grain boundaries meet the surface, formed to balance surface energies and grain boundary energy, hinder grain boundary movement. Furthermore, surface energy of the grains may depend on orientation. As favourably oriented grains have a high driving force for growth, the anisotropy in surface energy may provide the necessary additional driving force for abnormal grain growth (= secondary recrystallization). A phase field model that takes into account surface energy is presented and simulation results are discussed.

Deformation and Fracture from Nano to Macro: A Symposium Honoring W. W. Gerberich's 70th Birthday: Simulations of Mechanical Behavior

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

Wednesday PM — Room: 214D
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Erica T. Lilleodden, Forschungszentrum Karlsruhe

2:00 PM Invited

Molecular Dynamics Simulations of Deformation and Fracture Behavior in Nanocrystalline Ni: *Diana Farkas*[1]; [1]Virginia Tech

This talk will discuss large scale atomistic studies of the plastic deformation, fracture and fatigue mechanisms occurring in nanocrystalline metals of various grain sizes. We have studied Ni digital samples with grain sizes from 5 to 12 nm in diameter and randomly generated fully three dimensional microstructures. Using molecular statics and dynamics techniques, we observe various mechanisms of plasticity in these digital samples. The mechanisms observed include dislocation based mechanisms, grain boundary accommodation of slip and twinning. We observe grain boundary mechanisms of plasticity at the smallest grain sizes. At room temperature these include grain boundary motion as a stress accommodation mechanism. Our results show a distribution of grain boundary mobilities present in the samples, and the role of grain boundary motion in the deformation process at the nano-scale is discussed in detail.

2:20 PM

Dislocation Dynamics Simulations of Rough Surface Contact: *Lucia Nicola*[1]; Alan Needleman[1]; Allan Bower[1]; Erik Van der Giessen[2]; [1]Brown University; [2]University of Groningen

Due to unavoidable roughness, contact between surfaces is characterized by localized plastic deformation. When the loading is removed this leads to the development of a residual stress state that can promote crack nucleation. Therefore the study of plasticity in rough surface contact is of major importance for understanding friction and wear, as well as contact and fretting fatigue. We carry out simulations of indentation of an infinitely long two dimensional deformable single crystal by a rigid flat indenter. The profile of the crystal surface is taken to be a sinusoidal wave. Plasticity in the crystal occurs by the collective motion of discrete dislocations, modeled as line singularities in an otherwise isotropic linear elastic medium. A set of constitutive rules is supplied for the glide of dislocations as well as their generation, annihilation and pinning at point obstacles. The simulations track the evolution of the dislocation structure during loading and unloading.

2:35 PM

Atomistic Simulation of Metal Surface Indentation Including Interface Friction and Adhesion: *Virginie Dupont*[1]; Frederic Sansoz[1]; [1]University of Vermont

At the nanoscale, the plasticity of films and surfaces is strongly influenced by friction and adhesion effects. The present paper investigates the role of these effects on the elastic deformation and defect nucleation of FCC crystals via simulation of mechanical contacts. We simulated the indentation of surfaces with a single crystal cylinder using molecular dynamics and statics. The results are compared to the continuum elastic theories of M'Ewen and Johnson, Kendall and Roberts including friction and adhesion, respectively. Single crystal simulations were performed with different crystal orientations and atomic potentials in order to change the friction and adhesion properties of the contact interface. These simulations agree well with the continuum theory at early deformation stage, but deviate from this theory near the elastic limit. Polycrystal simulations are also addressed and show that friction and adhesion have strong impact on the grain boundary evolution, associated with GB movement and deformation twins.

2:50 PM

Combined MD and Continuum Approaches towards Modeling Inter-Granular Failure Using Cohesive Zone Models: *Veera Sundararaghavan*[1]; Nicholas Zabaras[1]; [1]Cornell University

Molecular dynamics (MD) is a valuable tool to understand GB response to loading conditions and hence, derive accurate cohesive laws for finite element simulations. In a MD study, we carried out a series of simulations on an arrangement of constrained atoms under loading in order to identify traction-separation laws in nanocrystalline GBs. Several dependencies were parametrically studied; these include dependence of grain boundary property on strain rate, mis-orientation, deformation modes (tension or shear), temperature etc. Modified cohesive laws which include thermal, rate dependence and deformation mode effects as provided by MD are presented and bicrystal simulations are initially carried out. These simulations employ a continuum slip theory based model for the interior of the grain. Extension from bicrystals to polycrystals with additional MD studies of cohesive response of tri- and quad- junctions to loading and subsequent incorporation of these effects in cohesive laws will be shown.

3:05 PM

A Molecular Dynamics Study of the Ductile to Brittle Transition in Dilute Iron Alloys: *Neeraj S. Thirumalai*[1]; Peter A. Gordon[1]; Michael J. Luton[1]; Diana Farkas[2]; [1]ExxonMobil Research and Engineering Company; [2]Virginia Tech

Model empirical interatomic potentials were used to study crack tip processes in single crystals of iron containing substitutional nickel and chromium additions. Molecular Dynamics techniques were employed to simulate the atomic level configuration of the crack tip region at temperatures up to 300 K. Various orientations were selected for study, with Ni and Cr additions up to 10 atomic %, in a random solid solution. The configuration of the crack-tip region was found to be dependent on the crystal orientation and the associated activity of ½<111> Burgers vector dislocations on the {110}, {112} and {123} slip planes. The emission of such dislocations at the crack-tip causes crack blunting and suppression of brittle crack extension. These results are discussed in terms of the effect of solute on the unstable stacking energy and the surface energy. The influence of the use of alternative empirical interatomic potentials on the observations is also discussed.

3:20 PM

Atomistic Simulation and Continuum Mechanics Analysis of the Formation of a Crack in a Disclinated Bicrystalline Nickel Nanowire: *Airat A. Nazarov*[1]; Kun Zhou[2]; Mao See Wu[2]; [1]Ufa State Aviation Technical University; [2]Nanyang Technological University

The stability of a cylindrical bicrystalline nickel nanowire containing a negative wedge disclination is studied via continuum mechanics calculations and atomistic simulations. The continuum theory considers stable and unstable double-ended equilibrium cracks initiating from the disclination in an isotropic cylinder, and takes into account the redistribution of stress due to crack disturbance. It predicts a critical disclination strength above which the disclination is unstable and an equilibrium crack can grow from it. For the atomistic simulations, a disclination is inserted into a cylinder of radius up to containing a special (310) [001] tilt grain boundary. Molecular statics relaxations are then performed starting from structures both without and with an initial interfacial crack. The continuum and atomistic calculations show very close agreement in the critical disclination strength, and general agreement in the stable crack length, the crack opening profile, and the stress field of the disclinated crack in the nanowire.

3:35 PM Break

3:55 PM Invited

Modeling Instrumented Indentation in Linear Viscoelastic Solids: *Yang-Tse Cheng*[1]; Che-Min Cheng[2]; Wangyang Ni[1]; [1]General Motors Corporation; [2]Institute of Mechanics

Inspired by Professor Gerberich's work on indentation in viscoelastic solids and motivated by the needs for probing small-scale mechanical behavior of "soft" matters, we have recently studied indentation in linear viscoelastic solids using analytical and numerical modeling. In this presentation, we derive the initial unloading slope equation for indentation in linear viscoelastic solids using rigid indenters with arbitrary axisymmetric smooth profiles, including conical and spherical indenters. While the same expression is known for indentation in elastic and elastic-plastic solids, we show that it is also valid for indentation in linear viscoelastic solids, provided that the unloading rate is sufficiently fast. When the unloading rate is slow, a "hold" period between loading and unloading can be used to provide a correction term for the initial unloading slope equation. Finite element calculations are used to illustrate the methods of fast unloading, "hold-at-the-peak-load," and "hold-at-the-maximum-indenter-displacement" for determining instantaneous modulus by instrumented indentation.

4:15 PM

A Numerical Study on the Effect of Grain Morphology on Anelasticity of Polycrystals: *Angelo Simone*[1]; Erik van der Giessen[1]; C. Armando Duarte[2]; [1]University of Groningen; [2]University of Illinois at Urbana-Champaign

Although the distribution of grain sizes and shapes in real materials is usually far from homogeneous, the importance of grain morphology in polycrystalline materials has been often neglected in theoretical and numerical studies. We present a study on the effect of grain morphology (size, size distribution and shape) on anelasticity of polycrystals caused by free grain boundary sliding. Free sliding is accounted for by means of the 'Generalized Finite Element Method for polycrystals', a novel computational technique developed by the authors. In the GFEM for polycrystals the finite-element mesh does not need to mimic the grain morphology since grain boundaries and junctions are described by means of elements with embedded discontinuities. With this computational tool, we carried out a series of simulations on a wide range of grain morphologies in several arrangements of grains. Realistic polycrystalline aggregates were generated with a vertex dynamics simulation.

4:30 PM

3-D Atomistic Pathways of Slip Transmission across Twin Boundaries: *Ting Zhu*[1]; Hyoung Gyu Kim[2]; Amit Samanta[2]; Ju Li[2]; [1]Georgia Institute of Technology; [2]Ohio State University

Recent experiments show that the introduction of nano-sized twins within ultrafine crystalline metals leads to significant increases in the strain-rate sensitivity. To understand the role of the nano-scale twins in affecting the rate dependence of plastic flow, we study in this work the 3-D thermally activated dislocation motions near the coherent twin boundaries. Using the reaction pathway sampling scheme of nudged elastic band and dimer methods, we identify the atomistic pathways of slip transmissions of both perfect and partial dislocations across the twin boundaries. The activation energies and activation volumes are quantified, thus making contact with previous continuum analyses and experimental measurements.

4:45 PM

Short Range Ordering and Room Temperature Creep in Metallic Titanium-Alumium Alloys: *M. Brandes*[1]; Peter Anderson[1]; Michael J. Mills[1]; [1]Ohio State University

Titanium-aluminum alloys are widely known to undergo pronounced creep deformation at low stresses and temperatures. Recently, short range ordering (SRO) of aluminum atoms in the HCP titanium matrix of these materials was found to provide significant creep strengthening. The presence of SRO leads to planar slip and the pairing of b = <1120> dislocations. In this work, the pairing of these dislocations is observed and measured via transmission electron microscopy in creep deformed Ti-6 wt% Al of several SRO states. Utilizing a combination direct measurement and modeling, the diffuse anti-phase boundary energies, the Peierls stresses, and solid solution strengthening parameters for these alloys are determined.

5:00 PM

A Multi-Scale, Dislocation Mechanics Based Model to Characterize the Transition Region Fracture Toughness Behavior of Ferritic Steels: *Matthew Wagenhofer*[1]; Marjorie EricksonKirk[1]; Mark EricksonKirk[1]; [1]Phoenix Engineering Associates, Inc.

Efforts to characterize the transition region fracture toughness behavior of ferritic steels have primarily been focused on the development of statistical models derived from empirical data sets. Recent work produced a quantitative model of the plastic work local to second phase particles that is required to initiate unstable cleavage fracture. This model captures the temperature dependence of the macroscopic toughness distribution and shows that it is a function of the strength of long range obstacles to dislocation motion. Further development has led to the refinement of a multi-scale, dislocation mechanics-based model to bridge the gap between the local nature of the plastic fracture work and the global nature of the macroscopic toughness values. Results presented here for A533B plate steel show that the multi-scale model is capable of capturing both the transition region temperature dependence as well as the distribution of toughness values across each temperature.

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

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

Wednesday PM Room: 216
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: R. Sunder, BiSS Research; Detlef Lohe, Universität Karlsruhe

2:00 PM Invited
Influence of Residual Stresses and Mean Load on the Fatigue Strength of Case-Hardened Notched Specimens: *Karl-Heinz Lang*[1]; Thomas Krug[2]; Detlef Löhe[1]; [1]Universitaet Karlsruhe; [2]Robert Bosch GmbH

Case-hardened gear wheels in are cyclically most stressed components. To evaluate the tooth-foot load-capacity, the knowledge of the mean stress and residual stress sensitivity of the fatigue strength of the material conditions at the failure critical sites is necessary. The examinations on hand usually concern homogeneous materials. Systematic analyses for case-hardened conditions are missing. In this work case-hardened notched specimens with different residual stress states are investigated under cyclic bending. The results are analyzed using a local concept and an additional fracture mechanical approach. With the local concept it was possible to determine the crack initiation site which was partly at the surface and partly below the surface. The fracture mechanical approach was necessary to understand the crack stop behavior. A uniform description of the lifetime behavior could be achieved using a modified Haigh diagram which takes into account the local stress state at the failure critical site.

2:25 PM
Effect of Residual Stresses and Relaxation on Fatigue Crack Propagation at Notches: *Dennis Buchanan*[1]; Reji John[2]; Alisha Hutson[1]; [1]University of Dayton Research Institute; [2]U.S. Air Force Research Laboratory

Fatigue crack propagation at critical locations such as holes and notches in turbine engine components pose many challenges. The presence of residual stresses and residual stress relaxation add considerable complication to these analyses. Baseline (as-machined) and shot peened double edge notch nickel-base superalloy specimens were tested at elevated temperature conditions. Corner crack propagation was monitored using electric potential, optical inspection and acetate replication during testing. FE analysis of corner crack propagation was accomplished using the 3D crack growth code ZENCRACK™ and FE code ABAQUS™. Residual stress depth profiles, measured via XRD on as-machined and shot peened surface finishes, were incorporated into the analyses. Crack propagation predictions with and without residual stresses were performed and compared with experimental measurements. The predicted crack propagation life correlated well with experimental results. A significant increase in crack growth life was observed for shot peened specimens under the imposed test conditions.

2:50 PM
Effects of Drawing Strain on the Bending Fatigue Properties of Hyper-Eutectoid (1.02%) Steel Wires: *Yang Yo Sep*[1]; Park Seong Yong[1]; Park Chan Gyung[1]; Bae Jong Gu[2]; Ban Deok Young[2]; [1]POSTECH; [2]TrefilARBED Korea

In this study, influences of drawing strain on bending fatigue properties of hyper-eutectoid (1.02%) steel wire with high strength were investigated. A series of the fatigue tests was carried out depending on drawing strain by using Hunter-type tester at a frequency of 60 Hz under the bending stress level of 900 to 1500 MPa. Microstructural changes of the wires were identified in the lateral direction by using transmission electron microscopy (TEM). Increasing drawing strain reduced lamellar spacing and cementite thickness, which effectively increased tensile strength of steel wires from 4400 to 4600 MPa. However, the fatigue limit gradually decreased from 1450 to 1300 MPa. Overall mechanical properties of the filaments, depending on drawing strain, have been discussed in terms of the microstructural parameter change of lamellar spacing and cementite thickness. In addition, the changes of cementite morphology on the fatigue crack propagation of hyper-eutectoid steel wires will be discussed.

3:15 PM Invited
Fatigue Strength Improvement of Notched Structural Steels: *Shin-Ichi Nishida*[1]; Nobusuke Hattori[1]; Congling Zhou[1]; Shengwu Wang[2]; [1]Saga University; [2]Dalian Jiaotong University

Fatigue tests have been performed using two kinds of specimens made of a conventional structural steel; that is, one is statically pre-strained specimen with changing from 2% until 8% by tension and the other is roller worked one with changing roller working ratio from 0.25mm until 1.50mm in depth. In the case of pre-strained specimen, the fatigue limit decreases under the small pre-strain and gradually increases with the pre-strain. In addition, the increase of fatigue limit is only 5% under the 8% pre-strain in tension. On the other hand, in the case of roller worked specimen, the fatigue limit increases extraordinarily by 120% with increase in the roller working ratio and is gradually saturated and then deteriorates a little. This remarkable increase of fatigue limit would be caused by the existence of large compressive residual stress, work-hardening, elongated micro-scopic structures, etc. in comparison with the case of monotonic tension.

3:40 PM Break

3:55 PM Invited
Isolation of Crack Closure from Residual Stress Effect in Variable Amplitude Fatigue: *R. Sunder*[1]; [1]BiSS Research

Residual stress variation is modeled in notch fatigue analysis as the primary variable affecting load sequence sensitivity of metal fatigue. In crack growth modeling, however, closure has emerged as the predominant variable to model sequence effects. This paper reviews recent research that appears to suggest that residual stress and crack closure may in fact qualify as independent variables that operate in synergy. Fractographic evidence from specially designed experiments appears to underscore the different nature of the two mechanisms. Crack closure truncates fatigue driving force, while residual stress moderates material resistance to fatigue driving force. It would follow that the residual stress effect should diminish or disappear altogether in vacuum, a possibility that appears to be indeed supported by available data from different materials.

4:20 PM
Effects of Laser Peening Treatment on High Cycle Fatigue Property of Degassing Processed Cast Aluminum Alloy: *Kiyotaka Masaki*[1]; Yasuo Ochi[1]; Takashi Matsumura[1]; Yuji Sano[2]; [1]University of Electro-Communications; [2]Toshiba Corporation

It is well known that the degassing (DG) process is very useful for decreasing cast defects such as porosity, micro-shrinkage, and inclusions in casting process of metallic alloys. The DG process is also the effective method for improvement of fatigue property of cast aluminum alloy. In this study, a laser peening (LP) treatment is applied to the DG processed cast aluminum alloy to improve the fatigue property. Fatigue tests of rotating bending loading are carried out on the LP treated DG processed cast aluminum alloy. It is found that the fatigue life in the whole range and the fatigue strength at 10^7 cycles of the DG cast aluminum alloy are improved by the LP treatment. The reasons of the fatigue property improvement by the treatment are decelerated effects of the surface crack growth rate by the compressive residual stress of the surface layer induced by the LP treatment.

4:45 PM
Effects of Residual Stresses on Probabilistic Lifing of Engine Disk Materials: *Harry R. Millwater*[1]; James Larsen[2]; Reji John[2]; [1]University of Texas at San Antonio; [2]Air Force Research Laboratory

Residual stresses are known to be beneficial with respect to fatigue life of metal components. Shot peening or other techniques are routinely used to improve the fatigue performance. This study examines the importance of the residual stress on the fatigue life using a probabilistic methodology. Specifically, the residual stress at a bolt hole in a compressor disk com-

posed of a superalloy is modeled as a random variable along with the initial crack size, life scatter and stress scatter. The sensitivities of the predicted probability-of-fracture with respect to the parameters of the random variables are computed and comprise a metric for comparison determining the significance of the residual stress on reducing the probability-of-fracture.

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

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

Wednesday PM Room: 215
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Peter K. Liaw, University of Tennessee; Stan Lynch, DSTO

2:00 PM Invited
The Influence of Soft, Precipitate Free Zones at Grain Boundaries of Ti- and Al-Alloys on Their Fatigue and Fracture Behavior: *Gert Lütjering*[1]; J. Albrecht[1]; C. Sauer[2]; T. Krull[1]; [1]Technical University Hamburg-Harburg; [2]Lufthansa Maintenance

High strength Al-alloys and beta Ti-alloys containing weak precipitate free zones (PFZ) along grain boundaries usually fracture within these soft zones upon monotonic or cyclic loading. In these cases, the fracture properties are mainly influenced by the strength difference between the age-hardened matrix and the soft PFZ. In the case of pancake shaped grains, the mechanical properties are anisotropic. This paper presents results obtained from peak-aged 7475/7075 Al-alloys and from the beta Ti-alloys Ti-6246 and Beta-CEZ for different grain sizes and grain shapes. In case of Ti-6246, the yield stress was varied between 1000 MPa and 1500 MPa to demonstrate the influence of the strength difference between matrix and PFZ on fracture toughness and HCF strength. This influence of the yield stress was studied for small and large equiaxed beta grains (bi-modal and beta-annealed microstructures) as well as for pancake shaped beta grains (beta-processed microstructure).

2:25 PM
Intrinsic Fatigue Crack Propagation Kinetics for Al-Cu-Mg/Li: *Yun Jo Ro*[1]; Sean R. Agnew[1]; Richard P. Gangloff[1]; Gary H. Bray[2]; [1]University of Virginia; [2]Alcoa Technical Center

The composition and aging condition of Al-Cu alloys govern intrinsic fatigue crack propagation for vacuum, but have less effect in the saturation regime of water vapor pressure/frequency, *p/f*. For Al-Cu-Mg, vacuum growth rate at high K_{MAX} increases dramatically with the transition from clusters to S' precipitation. This increase is large near-threshold and correlates with a slip band to nondescript transgranular crack path transition. Conversely, Al-Cu-Li exhibits outstanding vacuum growth resistance for all aging and ΔK conditions, correlating with slip band cracking in all cases. Water vapor increases growth rates and promotes a brittle mode of transgranular cracking for each alloy. Considering natural aging, Li has no measurable effect on da/dN in vacuum, compared to Mg, but is beneficial (1.5-3 times slower da/dN) for water vapor saturated N_2. Fatigue experiments with varying *p/f* test the hypothesized difference in Li vs Mg reaction with water to produce hydrogen at the crack tip.

2:50 PM
Identifying Sources of Fatigue Variability in a Powder Processed Nickel-Base Superalloy for Improved Life Prediction: *Michael J. Caton*[1]; Sushant K. Jha[2]; James M. Larsen[1]; K. Li[3]; William John Porter[3]; Andrew Henry Rosenberger[1]; [1]U.S. Air Force Research Laboratory; [2]Universal Technology Corporation; [3]University of Dayton Research Institute

In improving life management methods for turbine aircraft engines, a thorough understanding of the sources and scale of fatigue variability in relevant alloys is required. The fatigue behavior of a powder processed IN-100 nickel-base superalloy was examined at 650 C and at a frequency of 0.33 Hz. The influence of a 6 second hold time at peak load was also investigated. Total fatigue life, crack initiation, and long and small fatigue crack growth rates were examined. The maximum observed scatter in fatigue life is over an order of magnitude, and the vast majority of this variability is introduced during initiation and very early crack growth. A critical crack size is identified, above which the relative variability in crack growth behavior from specimen to specimen is almost negligible. A method for predicting worst-case failures based upon a life dominated by crack growth is presented.

3:15 PM
Fatigue Crack Growth at 223k in High Strength Aluminum Alloys: *Cedric Gasqueres*[1]; Christine Sarrazin-Baudoux[1]; Jean Petit[1]; David Dumont[2]; [1]ENSMA/Centre National de la Recherche Scientifique; [2]Alcan CRV

Aluminum alloys aircraft structures are confronted with temperatures ranging from 300K on the ground down to 223K during a fly. This paper deals with a study of the fatigue crack propagation in two high strength 2xxx alloys at 223K. Tests were performed in an environmental chamber with a controlled atmosphere (dew point of 223K). Cooling of the specimen was performed by mean of two blocks fixed on both sides of the C.T. specimens. Crack closure was detected using the compliance method. The crack propagation mechanism in the naturally-aged alloys was shown strongly modified at 223K with an abrupt transition from a stage II propagation at 300K to a retarded crystallographic stage-I like propagation at 223K. But no change was observed on the peak-aged alloys. The data are analyzed in terms of a previous modeling and influence of microstructure, temperature and air dryness is discussed on the basis of scan observations.

3:40 PM Break

3:55 PM
Mixed-Mode I/II Fracture of Polytetrafluoroethylene: *Eric N. Brown*[1]; Cheng Liu[1]; George T. Gray[1]; Dana M. Dattelbaum[1]; [1]Los Alamos National Laboratory

In the current work we present on mixed mode I/II fracture in polytetrafluoroethylene (PTFE). Four complex crystalline phases have been reported in PTFE with three crystalline structures (phases II, IV, and I) are observed at atmospheric pressure with transitions at 19 and 30°C. Mode I fracture in PTFE is strongly phase dependence with a brittle–to–ductile transition associated with the room temperature phase transitions. Increases in J integral fracture toughness around room temperature and above result from the onset of stable fibril formation bridging the crack plane and increased plastic deformation. The stability of drawing fibrils is primarily determined by temperature and crystalline phase with additional dependence on loading rate and microstructure anisotropy. Mixed mode I/II loading conditions are achieved using a modified compact tension specimen with Arcan type fixtures and digital image correlation. The bulk failure properties are correlated to failure mechanisms through fractography of the fracture surfaces.

4:20 PM
An Empirical Relationship between Crack Advance and Strains during Stage II Fatigue Crack Growth in a FCC Metal: *Seon-Ho Choi*[1]; Pedro Peralta[1]; James Gee[2]; Zhiyong Xie[2]; [1]Arizona State University; [2]University of Pennsylvania Medical Center

An empirical relationship between crack growth via plastic blunting and strain ahead of the crack tip is proposed. Cracks were grown under quasi-constant ΔK in the stage II region using pure polycrystalline nickel CT specimens. Two different sizes of CT specimens were prepared to study geometrical effects. A half cycle of the fatigue load that corresponds to the maximum K was applied during an in-situ loading experiment. The in-plane displacement fields were measured using digital image correlation and the corresponding strains were quantified using large deformation theory. It was found that there is a power law relationship between the maximum opening strain ahead of a crack tip and the applied load, ΔK, which indicates a proportionality between crack advance and plastic strain

at the crack tip. Finite element analysis using hybrid material properties based on hardness change in specimens also confirmed the power law relationship.

4:45 PM
Relationships between Tensile and Fracture Mechanics Properties and Fatigue Properties of Large Plastic Mold Steel Blocks: *Donato Firrao*[1]; Paolo Matteis[1]; Giorgio Scavino[1]; Graziano Ubertalli[1]; Maria Giuseppina Ienco[2]; Maria Rosa Pinasco[2]; Enrica Stagno[2]; Riccardo Gerosa[3]; Barbara Rivolta[3]; Antonio Silvestri[3]; Giuseppe Silva[3]; Andrea Ghidini[4]; [1]Politecnico Di Torino; [2]Università di Genova; [3]Politecnico di Milano; [4]LucchiniSidermeccanica

Molds for plastic automotive components such as bumpers and dashboards are usually machined from large pre-hardened steel blocks. Due to their dimensions, the heat treatment produces mixed microstructures, continuously varying with the distance from the quenched surface, at which fracture toughness and fatigue properties are not well known and generally lower than those corresponding to a fully quenched and tempered condition. The response of the mold to defects (for example, microcracks due to improper weld bed deposition) and strengths during service depends on steel properties, that in turn depend upon the heat treatment and the microstructure. A pointwise determination of the tensile, Charpy V-notched, fracture toughness and rotating bending fatigue properties was carried out in a large bloc. Infinite fatigue life was investigated by the stair-case method. The samples were obtained from different depths of the blooms. The relationship between mechanical properties, fracture surfaces morphology and microstructure was also investigated.

Furnace Systems Technology Workshop: Emerging Technologies and Energy Efficiency: Energy Efficiency and Emerging Technologies in Secondary Aluminum Melting

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division
Program Organizers: Paul E. King, U.S. Department of Energy; Subodh K. Das, Secat Inc

Wednesday PM
March 15, 2006
Room: Exhibit Floor
Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Paul King, U.S. Department of Energy

2:00 PM Introductory Comments

2:05 PM Invited
Gas-Fired Immersion Melting of Aluminum: Technical Challenges: Shridas T. Ningileri[1]; Qingyou Han[2]; *John A. Clark*[3]; Arvind Thekdi[4]; [1]Secat Inc; [2]Oak Ridge National Laboratory; [3]U.S. Department of Energy; [4]E3M, Inc

Energy efficiency ranging from 55% to 75% is possible if natural gas-fired, immersed tube burners were used to melt aluminum from secondary sources instead of open flames. Currently, recuperated tube burners with capacities to remelt aluminum are available. Some manufacturer claim thermal efficiencies above 80%. Previous efforts by Babcock and Wilcox, under GRI contract in the late 1980's, demonstrated the feasibility of immersion melting of aluminum. However, technological gaps have stalled further full-scale development and application of this melting method. Present furnace geometries hurt efficient transfer of energy from tubes into the aluminum. Traditional refractory tubes are quite porous and promote metal penetration and dross build-up resulting in poor performance. Secat, Inc. is leading an effort on a U.S. Dept. of Energy - Industrial Technologies Program sponsored project to develop a scaleable, natural gas-fired, immersion melter for the secondary aluminum industry with potential to replace traditional open-flame reverberatory furnaces.

2:30 PM Question and Answer Period

2:35 PM
Improved Aluminum Melting Using Pumping: *Brian Golchert*[1]; Hossam Metwally[1]; Paul King[2]; Chris Vild[3]; [1]Fluent, Inc.; [2]Albany Research Center; [3]Metaullics Systems

It has been suggested that stirring of molten aluminum during the melting process would increase the melting efficiency since it would allow the hotter aluminum at the top of the melt to mix with the colder aluminum at the bottom of the melt. Instead of using mechanical stirrers, one could use pumping, either mechanical or electromagnetic. This paper will present the results of a computational fluid dynamics study of the effect of pumping on the energy efficiency of an aluminum melter. How the pumps affect the temperature distribution and heat requirements needed to melt the aluminum will be analyzed. The effect of pump location and pumping power on the furnace efficiency will also be analyzed to determine if there exists an optimal location and size of the pump.

3:00 PM Question and Answer Period

3:05 PM Invited
High Temperature Industrial Furnace Based on Radiative Homogeneous Combustion for Improved Efficiency and Reduced Emissions: *Arvind Atreya*[1]; [1]University of Michigan

A solution to the problem of "configuring combustion in industrial furnaces to increase efficiency and reduce emissions" is examined in a laboratory-scale furnace. The incoming oxygen-enriched air and fuel are highly preheated with wasted flue gas enthalpy. While simple, this method increases the NOx production and contributes to heat flux non-uniformities within the furnace. A solution to this problem is proposed where EGR, flame radiation and increased residence time are employed to reduce the flame temperature and thus NO. Nearly homogeneous burning occurs in distributed reaction zones under slightly rich conditions that enable increasing the flame radiation and also promote NO reburn reactions. The aim is to increase flame emissivities at temperatures not exceeding 1900K and provide uniform radiation heat flux at a magnitude exceeding 400kW/m^2, while maintaining strict constraints on NOx, CO, unburned hydrocarbons and particulate emissions. This enables increasing furnace productivity or decreasing its size and cost.

3:30 PM Question and Answer Period

3:35 PM Break

3:50 PM Invited
Energy Efficient Operation of Secondary Aluminum Melting Furnaces: *Paul E. King*[1]; Jarrod J. Hatem[1]; Brian M. Golchert[2]; [1]U.S. Department of Energy; [2]Fluent Inc.

It has been shown that re-melted aluminum can be produced at approximately 5% of the energy required of primary production. However, industry wide studies suggest an average melt efficiency of 25% in secondary furnaces. Efficiency improvement of the re-melt process is a challenge that currently faces the industry. Refinement of this process requires analysis of the intricate combustion and heat transfer processes within the furnace. The Department of Energy, Albany Research Center (ARC) has had tremendous success at predicting the performance of industrial secondary furnaces. Through scale modeling and verifiable computational results, the ARC has been able to simulate the processes of industrial furnaces with a high degree of confidence. These methods have suggested small design modifications that improve overall furnace performance. Using the results from previous computational models, the ARC aims to define the design parameters that will result in peak efficiency considering both power input and emission composition.

4:15 PM Question and Answer Period

4:20 PM Concluding Comments

General Abstracts: Light Metals Division: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Magnesium Committee, TMS: Reactive Metals Committee, TMS: Recycling Committee
Program Organizers: Jim McNeil, Novelis Inc; Neale R. Neelameggham, US Magnesium LLC

Wednesday PM Room: 7D
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Michael J. Kaufman, University of North Texas

2:00 PM
Refinement of As-Cast Microstructure of Aluminum 319 Alloy Using High Intensity Ultrasonic Vibration: *Xiaogang Jian*[1]; Thomas Tom Meek[1]; Qigui Wang[2]; Qingyou Han[3]; [1]University of Tennessee; [2]General Motors Corporation; [3]Oak Ridge National Laboratory

High intensity ultrasonic vibrations were utilized in an attempt to achieve grain refinement for aluminum-silicon-copper based 319 alloy. Ultrasonic energy up to 1500 W was injected into the alloy during its solidification. Casting temperature was varied in order to obtain optimal grain refinement effect. The experimental results indicated that ultrasonic vibration has a significant effect on the as-cast microstructure of 319 alloy. Small globular grains were obtained instead of large dendritic grains. The morphology of the eutectic phase was also altered.

2:25 PM
Pullout Strengths of Keenserts in High Performance Aluminum Alloys: Alexander Ordonez-Chu[1]; Marcus Prietto[1]; Magdolna Hugi Haberl[2]; *John Harold Kabisch*[3]; Omar S. Es-Said[1]; Richard Clark[4]; John Ogren[1]; [1]Loyola Marymount University; [2]Los Angeles Valley College; [3]Long Beach City College; [4]College of the Canyons

Numerous structural components on expendable launch vehicles require the use of blind installations for attachment purposes. For this study, a type of insert known as a Keensert was used. Keensерts are permanently installed into one of the structural members and a bolt is used to attach such structure to mating structure. The Keenserts in this study were installed into specimens made from 2219 aluminum alloy and 2099 aluminum-lithium alloy. Both T6 and T8 tempers were tested with Keenserts of three different lubricants and three different diameters. Maximum load was determined by pulling the inserts from their installations.

2:50 PM
Role of Scandium on Mechanical Properties of Al-Zn-Mg Alloys: *Pathickal K. Poulose*[1]; M. Ashraf Imam[2]; Jerry Feng[2]; Krishnan K. Sankaran[3]; [1]University of the District of Columbia; [2]Naval Research Laboratory; [3]Boeing Company

The effect of small amounts of scandium on strengthening characteristics of an Al-Zn-Mg alloy was studied. A composition close to aluminum alloy 7055 having 0.1wt% Sc was used (8 wt% Zn-2 wt% Mg-2wt% Cu-0.1wt% Zr-0.1wt% Sc-balance Al). The solution treated and quenched alloy was aged at 120°C in air. Progress of hardening as a function of time was monitored using hardness and impression tests until past peak hardness. Structure, density and homogeneity of distribution of intermetallic phases including Al3(Sc1-xZrx) were studied using transmission electron microscopy and diffraction techniques. Enhancement of stability of the grain structure due to the presence of Al3(Sc1-xZrx) was analyzed. The results are compared with aluminum alloy 7055. Changes in the homogeneous and heterogeneous precipitation characteristics due to scandium, including effect on precipitate-free zones and role of Al3(Sc1-xZrx) in providing nucleation sites for other precipitates, were examined. The relationship between microstructure and mechanical properties are discussed.

3:15 PM
Effect of Microstructural Features on the Mechanical Behavior of A356 Alloy: *Yong Nam Kwon*[1]; Y. S. Lee[1]; J. H. Lee[1]; [1]Korea Institute of Machinery and Materials

A356 alloy is one of most widely used cast Al alloys for automotive components. Mechanical properties of A356 alloy largely depend on the casting defects, such as gas porosity, oxide film and so on. Recently, there has been an interesting report on the inverse Hall-Petch type relation on A356 alloy, which means the larger primary alpha gives rise to the higher strength. In the present study, the effect of the basic microstructural features of A356 alloy on the mechanical behavior of was studied. Various samples of A356 were prepared with different casting routes. In order to get rid of porosity effect, compressive deformation was applied. The results tell that the overall mechanical behavior is closely related with the porosity level, eutectic Si distribution as well as volume fraction of primary alpha. Discussion of the microstructural effect on deformation mechanism will be given in detail.

3:40 PM
Age Hardening Behavior of Modified Al-Si-Cu Cast Alloys: *Junyeon Hwang*[1]; Herbert Doty[2]; Michael Kaufman[1]; [1]University of North Texas; [2]GM Powertrain

Two common cast aluminum alloys used in the automotive industry are alloys 319 (Al-Si-Cu) and 356 (Al-Si-Mg). These alloys have good castability, low density, acceptable mechanical properties and low cost. Slight property improvements are sometimes achieved by (1) alloying additions (modifiers, solid solution strengtheners, etc.), (2) modified heat treatments and (3) improved degassing procedures. In this study, 319 Al modified with Mg, Mn and Sr was examined after peak aging, over aging, step aging, etc. using a variety of advanced analytical characterization methods (e.g., transmission electron microscopy). The results indicate that small additions of Mn have a slightly beneficial effect on the baseline properties whereas larger additions decrease both strength and ductility. These and other effects are correlated with the observed variations in strength in order to clarify their role on structure-property relations in these alloys. [Supported by General Motors through the Powertrain Advanced Materials Engineering Group].

Hume Rothery Symposium: Multi-Component Alloy Thermodynamics: Kinetics and Microstructural 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

Wednesday PM Room: 202A
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Shuanglin Chen, CompuTherm, LLC

2:00 PM Invited
CalPhad and Phase Field Modelling: A Successful Liaison: *Ingo Steinbach*[1]; Bernd Boettger[1]; Nils Warnken[1]; [1]ACCESS

The connection between CalPhad models and Phase Field models is discussed against the background of minimization of the total Gibbs energy of a system. Both methods are based on separation of a multiphase system into individual contributions of the bulk phases, which are described by appropriate models. While the CalPhad method uses a global minimization of the total Gibbs energy, the Phase Field method introduces local interactions, interfaces and diffusion and allows for nonequilibrium situations. As such the Phase Field method is much more general by its concept, however it can profit a lot if realistic thermodynamic descriptions, as provided by the CalPhad method, are incorporated. The present paper discusses details of a direct coupling between the Multi Phase Field method and the CalPhad method. Examples are presented from solidification of technical Mg and Ni base alloys and some problems arising from common practice of thermodynamic descriptions in order-disorder systems.

2:30 PM Invited

Application of Thermodynamic Database to Phase Field Modeling of Microstructural Evolution in Complex Multi-Component and Multi-Phase Alloys: *Yunzhi Wang*[1]; Ning Ma[1]; [1]Ohio State University

Most engineering alloys are multi-component and multi-phase with complex microstructures. The traditional constitutive models representing microstructural features by their average values without capturing the spatially varying aspects may not be sufficient to quantitatively define the microstructure and hence allow for establishing a robust microstructure-property relationship. The phase field approach has proven to be able to handle complex microstructural patterns. However, quantitative modeling at real length and time scales requires model inputs be linked to thermodynamic and mobility databases. In this presentation we review our recent efforts in developing quantitative phase field models by linking model inputs to multi-component and multi-phase thermodynamic databases. Critical issues related to the formulation of a non-equilibrium free energy as a function of both concentration and long-range order parameters based on the equilibrium free energies of individual phases available from the database will be addressed. Examples of applications will be given for advanced Ti alloys.

3:00 PM Invited

How Far Can We Rely on Multi-Component Equilibrium Thermodynamic Data in Non-Equilibrium Kinetic Simulations?: *Ernst Kozeschnik*[1]; Ivan Holzer[1]; Bernhard Sonderegger[1]; [1]Graz University of Technology

When performing precipitation kinetics simulations in multi-component materials, input data such as chemical potentials or driving forces can be evaluated from equilibrium thermodynamic databases. Even interfacial energies can be estimated from these databases with surprising success, thus reducing the number of critical fit parameters to zero. However, solid-state precipitation is a non-equilibrium process by nature and the path along which the system evolves toward final equilibrium can significantly deviate from any kind of equilibrium state. With a novel model, the authors investigate the predictive potential of precipitation kinetics simulations based on thermodynamic equilibrium databases in a number of alloy systems and for various different materials. It is shown that in systems where the equilibrium thermodynamics is well known, the predictions are reasonably close to experiment.

3:30 PM Break

3:50 PM Invited

Modeling of Microstructure Evolution during Solidification of Multi-Component Alloys: *M. F. Zhu*[1]; Weisheng Cao[2]; Shuanglin Chen[3]; Chunpyo Hong[4]; Y. Austin Chang[2]; [1]Southeast University; [2]University of Wisconsin-Madison; [3]CompuTherm LLC; [4]Yonsei University

Driven by industrial demand, extensive efforts have been made to investigate microstructure evolution during solidification of multicomponent alloys. This paper briefly reviews the recent progress in modeling of microstructures in multicomponent alloy solidification using various models including phase field, front tracking and cellular automaton approaches. Then a two-dimensional modified cellular automaton (MCA) model coupled with phase diagram software PANENGINE is presented for the prediction of microstructures and microsegregation in solidification of ternary alloys. The model adopts MCA technique to simulate the nucleation and growth of dendrites. The thermodynamic data needed for determining the dynamics of dendritic growth are calculated with PANENGINE. After validating the model by comparing the simulated values with the prediction of the Scheil model for solute concentration profile in the primary dendrites as a function of solid fraction, the model was applied to predict the evolution of microstructure and microsegregation in the directionally solidified Al-rich ternary alloys.

4:20 PM

Thermodynamic and Microstructural Modeling of Nb-Si Based Alloys: *Sundar Amancherla*[1]; Bernard Bewlay[2]; Ying Yang[3]; Y. Austin Chang[3]; [1]GE India Technology Center; [2]GE Global Research; [3]University of Wisconsin-Madison

Nb-Si alloys have gained much attention over the last decade as the next generation alloys for high-temperature aero-engine applications due to their low density and better properties. However the microstructures of these alloys are quite complex and vary significantly with the addition of elements such as Ti and Hf. Hence an improved understanding of the phase stability and the microstructural evolution of these alloys is essential for rapid alloy design for advanced high-temperature applications. In the present paper we will describe the thermodynamic construction of the Nb-Si-Ti-Hf system and the microstructural evolution modeling results, obtained using a phasefield model. The results of the calculations for these Nb-Si based alloys will be compared with a range of experimental observations.

4:45 PM

Evaluation of Microstructure Evolution in Multiphase TRIP Steels by Thermodynamic and Diffusion Calculations Combined with a Dilatometry Analysis: *Chang-Seok Oh*[1]; Dong-Woo Suh[1]; Sung-Joon Kim[1]; [1]Korea Institute of Machinery and Materials

During the past decades thermodynamic calculations with reliable databases for multicomponent alloy systems have been successfully applied to the development of new materials and understanding processing route thereof. In case of steels, use of thermodynamic calculation in delineation of microstructure evolution during heat treatment can't be fully validated due to the fact that interstitial element usually governs overall kinetics of phase transformation. Recently, methodology based on restricted equilibrium concept, such as para-equilibrium, was widely used in order to put more real factors to the thermodynamic calculation of steels. Meanwhile, in selected cases, numerical simulation of diffusion-controlled phase transformation has shown a sufficient level of accuracy and agreement with experimental observations. In this presentation, few examples are shown how thermodynamic and diffusion calculations combined with a dilatometric analysis can be used to predict microstructure evolution of the multiphase TRIP-aided steels, and limitation and usefulness of the multicomponent thermodynamic calculation are also discussed.

Lead Free Solder Implementation: Reliability, Alloy Development, and New Technology: Electromigration and Reliability

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

Wednesday PM
March 15, 2006

Room: 214A
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: K. N. Subramanian, Michigan State University; Laura J. Turbini, University of Toronto

2:00 PM Invited

Electromigration in Pb-Free Flip Chip Solder Joints on Flexible Substrates: *Jae-Woong Nah*[1]; Fei Ren[1]; King-Ning Tu[1]; Sridharan Venk[2]; Gabe Camara[3]; [1]University of California, Los Angeles; [2]Belton-Group; [3]Western Digital

Flexible circuit technology enables smaller, lighter, faster products, and lower cost because flexible circuit can be rolled, bent, and folded to fit a limited space where required. Therefore, flip chip on flex may have a wide application for liquid crystal display (LCD), hard disk drive (HDD), automotive, and medical electronics applications, etc. As the electronics industry continues to push for high performance and miniaturization, electromigration is one of serious reliability issues in flip chip solder joints on flexible substrates. In this study, we report electromigration behavior of flip chip Sn96Ag3.5Cu0.5 solder joints on flexible substrates. The in-situ resistance change of the flex circuit during the electromigration test was measured. And the voids movement inside the solder joints due to electromigration was investigated. This voids movements was matched with the resistance change during the electromigration test.

2:25 PM

Threshold Current Density of Electromigration in Eutectic SnPb and SnAgCu Solder Lines: *Min-Seung Yoon*[1]; Min-Ku Ko[1]; Young-Chang Joo[1]; Oh-Han Kim[2]; Young-Bae Park[2]; [1]Seoul National University; [2]Andong National University

The flip chip packages with the higher current densities have given rise to the electromigration-induced failure. Threshold current density is defined by the current density in which the electromigration flux is zero, i.e. atomic migration did not occur any longer. Drift velocities of eutectic SnPb and SnAgCu lines with the length of 1000 μm at the various current densities were measured through the interruptive test. From the drift velocities, threshold current density was calculated. The threshold current densities of the eutectic SnPb and SnAgCu lines are $7.4X10^3$ A/cm^2 at 100°C and $1.8X10^4$ A/cm^2 at 140°C, respectively. The threshold current density was measured in the solder lines with the various line lengths. Threshold current density is determined by the balance of the forward flux (electric-wind-force) and the backward flux (the stress-gradient or the atomic-concentration-gradient). Details on the immortal condition of the eutectic SnPb and SnAgCu will be discussed.

2:45 PM

Effects of Solder Volume on the Cross-Interaction between Cu and Ni in Cu/Sn3.5Ag/Ni Solder Joints: *C. E. Ho*[1]; S. C. Yang[1]; Chien Wei Chang[1]; C. Robert Kao[1]; D. S. Jiang[1]; [1]National Central University

The copper/solder/nickel sandwich structure was one of the most common solder joint configurations for electronic packages. In the past, many studies just focused on the unilateral reaction of solders with Cu or Ni, and didn't take into consideration the possible interaction of the two interfaces. In this study, the interaction between Cu and Ni across the Sn3.5Ag (wt.%) solder was investigated. The Sn3.5Ag spheres used in this study were 760, 500, or 300 microns in diameter. As a result, solder joints with the same configuration but three different volumes were studied. The as-reflowed joints were subjected to high temperature storage at 160°C. The results revealed the Cu6Sn5 and the Ni-bearing Cu6Sn5 were the predominant reaction products on the Cu-side and the Ni-side of the joints, respectively. Their growth and morphologies were found to be strongly dependent on the solder volume. The kinetics and the underpinning mechanism will be presented.

3:05 PM Break

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Electrical Charateristics for Sn-Ag-Cu Solder Bump with Ti/Ni/Cu Under-Bump-Metallization after Environmental Tests: T. I. Shih[1]; S. Y. Tsai[2]; G. J. Chiou[1]; *Jenq-Gong Duh*[1]; [1]National Tsing Hua University; [2]Ming Hsin University of Science and Technology

Lead free solder bump has been widely used in current flip chip technology (FCT) due to the environment issue. Solder joints after various environmental tests were employed to investigate the interfacial reaction between the Ti/Ni/Cu under-bump metallization (UBM) and Sn-Ag-Cu solders. The environmental tests included highly accelerate stress test (HTST), temperature cycling test (TCT), and high temperature storage test (HTSL). An SEM internal probing system was introduced to evaluate the electric characteristics in the intermetallic compounds after different test conditions. The electric data would be correlated to microstructural evolution due to the interfacial reaction between solder and UBM. In addition, a REBECA-3D software was also used to simulate the corresponding strain and stress distribution in the joints subjected to environmental tests.

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Joule Heating Effect in Flip-Chip Solder Joints for Various Dimension of Al Traces: *Sheng-Hsiang Chiu*[1]; Chih Chen[1]; S. S. Lin[2]; C. M. Chou[2]; Y. C. Liu[2]; K. H. Chen[2]; [1]National Chiao Tung University; [2]MEGIC Technology

The purpose of this paper is to investigate the thermal characteristic of solder bumps with different dimension of Al trace under current stressing in flip-chip package. The thermal characteristic of flip-chip solder joints under different current stressing conditions was measured by infrared technique. The measured temperature increase due to Joule heating, and we also discussed the temperature increase in different dimension of Al trace under current stressing. Because the Al traces on the chip side have much higher electrical resistance than the total electrical resistance of the flip chip solder joints and the Cu pad on the substrate. The Al trace on the chip side is the major heat source during current stressing. According the results from infrared technique, it was found that the solder joint of with longer Al trace has Joule heating effect than that of solder joint with shorter Al trace.

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Morphology and Growth Pattern Transition of IMCs between Cu and Sn3.5Ag Containing Small Amount of Additives: *Feng Gao*[1]; Tadashi Takemoto[1]; Hiroshi Nishikawa[1]; [1]Osaka University

The morphology and the grain growth pattern of IMCs formed between Cu substrate and Sn3.5Ag solder doped with small amount of additives (0.1mass%), say, Ni or Co, was investigated. The faceted-shape IMCs were observed for outer region of (Cu, Ni)6Sn5 or (Cu, Co)6Sn5 IMCs. However, the rounded-shape IMCs were identified for inner region of (Cu, Ni)6Sn5 or (Cu, Co)6Sn5 IMCs. Based on the thermodynamic calculation, the enthalpy change for the outer region IMCs was significantly elevated due to the high concentration of Ni or Co additives. Consequently the increased Jackson parameter value was even larger than 2 to ensure the formation of faceted-shape crystal. The abnormal grain growth (AGG) was demonstrated from the evolution of IMC grain size distribution. With the extended reflow time, the abnormal large grain size of outer region of (Cu, Ni)6Sn5 or (Cu, Co)6Sn5 IMCs was generated.

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A Study on the Rework Lead-Free Solder Components: *Huann-Wu Chiang*[1]; Ming-Chuan Chen[1]; Jeffrey Lee[2]; [1]I-Shou University; [2]Advanced Semiconductor Engineering, Inc.

A rework process will be performed on low-profile quad flat package (LQFP) components with various terminal finishes by using Sn-3.0Ag-0.5Cu lead free solder wire on Cu/Ni/Au PCB (printed circuit board). After 3 time rework processes, 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 EPMA (energy probe micro analysis) to observe metallurgical evolution in the interface and solder buck itself. Pull test will be performed on the TCT samples. The relationship among interfacial microstructure, the terminal finish and the joint strength will then be analyzed and discussed. Samples by normal SMT process will also be analyzed for comparison.

Magnesium Technology 2006: Alloy Development 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

Wednesday PM — Room: 6B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Gordon Dunlop, Advanced Magnesium Technologies; Kwang S. Shin, Seoul National University

2:00 PM

AM-Lite a New Magnesium Diecasting Alloy for Decorative Applications: Trevor Abbott[1]; Morris Murray[1]; *Gordon Dunlop*[1]; [1]Advanced Magnesium Technologies Pty Ltd

The recently developed magnesium diecasting alloy, AM-lite, has attributes that address many of the issues that restrict the growth of applications for the well known magnesium diecasting alloy, AZ91D. As well as being capable of replacing AZ91D, AM-lite can also replace zinc and aluminium diecasting alloys and plastics in many applications. The most important features of AM-lite relate to its diecastability, its much improved as-cast surface, ability to be electroplated, oxidation and corrosion resistance, improved mechanical properties and recyclability. Improved fluidity of the alloy leads to an ability to reliably diecast very thin sections with

reduced dimensions of runners and overflows. The as-cast surface is much smoother than AZ91D resulting in a reduced need for buffing or polishing as preparation for surface finishing operations. The surface chemistry allows high quality electroplating at a similar cost as for zinc diecastings. Diecast AM-lite has a much higher linear elastic limit than AZ91D and this provides a higher design strength. Because of its resistance to oxidation, the alloy has improved recyclability giving it a potential for in-cell recycling in many diecasting operations.

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Effect of Ca Addition on the Microstructure Evolution of AZ31 Alloy During Thermomechanical Processing: *Elhachmi Essadiqi*[1]; Jian Li[1]; C. Galvani[1]; P. Liu[1]; R. Varano[2]; S. Yue[2]; Ravi Verma[3]; [1]MTL CANMET; [2]McGill University; [3]General Motors R&D Center

The use of Magnesium alloys in auto industry has been limited to diecast parts due to their low ductility and fracture toughness. Grain refinement in wrought magnesium alloy can be produced by thermal-mechanical processing to increase the ductility, fracture toughness and the strength. In this study, the microstructure of two AZ31 alloys during extrusion and hot rolling, one with Ca and one without Ca addition, were compared. The magnesium with small amount of Ca addition produced finer grain size. The grain refinement is attributed to the precipitation of small Ca-rich particles, which reduce the grain boundary mobility and stabilize the fine-grained microstructure during extrusion and subsequent hot rolling.

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Effect of Ca on Microstructure and Mechanical Properties of Mg-Li-Al Alloy: Hong Bin Li[1]; Guang Chun Yao[1]; *Yi Han Liu*[1]; [1]Northeastern University

Mg-9mass%Li-2mass%Zn(-Ca) alloys were cast and rolled to sheet at room temperature. Effect of Ca on microstructure and mechanical properties of the sheet is studied in this study. Uniaxial tension tests are carried out at room temperature. The rise of tensile strengthen and elongation of sheets is 19% and 6%, respectively, when Ca content being 0.1mass%. While elongation descends largely with Ca content rise. Ca can refine grains and the effect of thinning is the evident test while Ca content being 0.1mass%. It is clarified that adsorption of Ca on the grain boundary makes grains refined and that it changes the mechanical properties of sheets.

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Effect of Ca/Y on Tensile Properties and Damping Capacity of As-Cast Mg-0.6Zr Alloy: *Chuming Liu*[1]; Ren Feng Ji[1]; Haitao Zhou[1]; [1]Central South University

The effect of Ca/Y on tensile properties and damping capacity of Mg-0.6Zr alloy are investigated. It is found that the 0.3%Ca, 0.3%Ca +0.1%Y added into Mg-0.6Zr alloy respectively leads to both grains refinement and improvement of tensile properties which refers to grain strengthening and solution strengthening. In contrast, the damping capacities of the modified alloys measured by DMA decreased slightly. When strain is below 0.014, Q-1 decreases rapidly. While strain is over 0.014, Q-1 decreases slightly. This indicates that it is dependent on strain amplitude. The decrease of damping capacity is due to the hindrance to the motion of dislocations with the element Ca/Y and the increasing number of grain boundaries. The most interesting result is that the addition of Ca/Y suspends the damping peak(P1) of Mg-0.6Zr at 75°C. The damping mechanism of the tested alloys could be analyzed by G-L dislocation damping model.

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Effects of Alloying Elements on Mechanical Properties of Mg-Al-X Alloys at Elevated Temperatures: *Kwang Seon Shin*[1]; Woo Chul Cho[1]; Hwa Chul Jung[1]; [1]Seoul National University

Various new magnesium alloys have been developed in recent years for elevated temperature applications. In the present study, the effects of various alloying elements on mechanical properties of Mg-Al-X alloys were investigated at elevated temperatures. Various alloying elements including Ca, Sr, Mm (Misch metal), etc. were added to Mg-Al alloys in order to either introduce the thermally stable precipitates at grain boundaries as well as in the grain interior or suppress the formation of the Mg17Al12 phase and thus improve the high temperature mechanical properties of magnesium alloys. The specimens were produced on a 320 ton high pressure die casting machine. The microsructures of the specimens were examined by optical and scanning electron microscopy. In addition, the existence and distribution of second phases were also examined by X-ray and EPMA. The high temperature mechanical properties of the die-cast specimens were examined in the temperature range of 150°C to 200°C.

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Hot Deformation Behaviors of Mg-6Zn-2Nd-0.5Zr Alloy: *Haitao Zhou*[1]; Zhendong Zhang[1]; Chuming Liu[1]; [1]Central South University

Deformation behaviors of Mg-6Zn-2Nd-0.5Zr alloy during hot compression at temperature range from 543 K to 693 K and strain rate from 0.001s-1 to 1 s-1 are investigated. Flow softening is found to occur and all flow curves exhibit a peak with characteristics of dynamic recrystallization and significantly change with temperature and strain rate. As a result of DRX, the grains are greatly refined through dynamic recrystallization, and the mean size of the recrystallized grain decreases with the decrease of temperature or the increase of Z parameter, while the reciprocal of the recrystallized grain has a good linear relationship with the natural logarithm of Z value. After comparsion of DRX grain size, it is found 593K is the best deformation temperature. After hot extrusion at 593 K, the yield strength and tensile strength of extruded Mg-6Zn-2Nd-0.5Zr alloy are 320MPa and 370MPa respectively, which are higher that of ZK60 alloy.

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Influence of Li and Y Alloying Additions on Microstructural Evolution and Texture of Magnesium Alloys: *Luke William Fox Mackenzie*[1]; Mihriban Pekguleryuz[1]; Ravi Verma[2]; [1]McGill University; [2]General Motors

The influence of alloying additions, Li and Y, on the microstructural and texture evolution of pure magnesium and AZ31 alloys, during thermomechanical processing, has been investigated. Alloy coupons were cast in copper moulds and homogenized at temperatures between 380 and 450°C. As-cast and homogenized microstructures were then characterized, and the homogenized specimens were rolled up to 60% at temperatures between 25 and 400°C. Selected specimens were subsequently annealed at 400°C. Optical microscopy, scanning electron microscopy and electron backscattered diffraction (EBSD) were used to characterize the microstructures and textures of the rolled and annealed alloys. The influence of Li and Y alloying additions on the microstructural evolution and texture of magnesium alloys has been demonstrated.

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Magnesium Alloys with Magnetic Properties: *Martin Bosse*[1]; Friedrich-Wilhelm Bach[1]; [1]University Hanover

Magnesium is suited especially as construction material for highly accelerated components. In order to increase its field of application, magnesium alloys shall additionally be equipped with ferro-magnetic properties. The main objective of the presented project founded by the German Research Foundation (DFG) is to develop a composite material made of magnetic particles and a magnesium base alloy. The die casting process is used to insert magnetic powders in the outer surfaces of the component. Hence, it is possible to use this part of the component as a storage zone for magnetic encoding. A second goad is the characterization of magnesium alloys which are modified by additions of cobalt in combination with Rare Earth (RE) metals. Previous research at IW has shown that intermetallic phases with magnetic properties can be precipitated controllably. These phases open the possibility to use the reverse magnetostriction effect to measure the mechanical load in the component.

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The Microstructures and Tensile Properties of AZ61-RE Alloy: *Haitao Zhou*[1]; Zijuan Liu[1]; [1]Central South University

The microstructure and tensile properties of AZ61 and AZ60-RE alloys are investigated. It is found that RE brought about precipitation of a new Al11RE3 phase besides ß phase. After hot extrusion, both alloys are refined due to dynamic recrystallization. However, a very finer grain with size of 6um is obtained in AZ61-RE. This suggests that RE had a great effect on grain refining during dynamic recrystallization. Tensile tests at room temperature suggest that the strength of hot-extruded AZ61-RE is higher than that of AZ61-RE alloy, whilst ductility of hot-extruded AZ61-RE is a little lower. The improvement of tensile properties of AZ61-RE alloy by RE addition is attributed to very finer grains.

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High-Strength Mg-Zn-RE Alloys with Long Period Order Structure: *Yoshihito Kawamura*[1]; Shintaro Yoshimoto[1]; Michiaki Yamasaki[1]; [1]Kumamoto University

Ingot metallurgy (I/M) alloys with a long period order (LPO) phase had excellent yield strength above 400 MPa, ultimate tensile strength of 450 MPa or more, and elongation of 5% or more. Magnesium alloys in which LPO phase was formed were limited to magnesium with Zn and RE elements added. RE elements forming LPO phase were limited to seven: Y, Gd, Tb, Dy, Ho, Er, and Tm. The Mg-Zn-RE alloys with LPO phase can be divided into two types. Type I is Mg-Zn-(Y, Dy, Ho and Er) alloys, in which LPO phase was formed in an as-cast state. Type II is Mg-Zn-(Gd, Tb and Tm) alloys. In the type II, LPO phase which did not exist in an as-cast state was formed by annealing at 773 K for 5 hours or more. The LPO Mg-Zn-RE alloys are expected to yield a new concept of strengthening in magnesium alloys.

Magnesium Technology 2006: Wrought Alloys and Forming Processes III

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

Wednesday PM — Room: 6A
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Alan A. Luo, General Motors Corporation; Mihriban O. Pekguleryuz, McGill University

2:00 PM

Low-Temperature Superplasticity of Bulk Microcrystalline Magnesium Alloys: *Vladimir N. Chuvil'deev*[1]; Mikhail Yu. Gryaznov[1]; Anatoly N. Sysoev[1]; Vladimir I. Kopylov[2]; [1]Physical-Technical Research Institute of Nizhny Novgorod State University; [2]Physical-Technical Institute of National Academy of Science

New microcrystalline ZK60, AZ31, and AZ91 magnesium alloys are processed by equal - channel angular pressing (ECAP). Mean grain size of microcrystalline magnesium alloys is equal to 1 micron. The record low-temperature superplastic characteristics are obtained in microcrystalline alloys: ZK60 (elongation to failure is equal to 810%), AZ31 (400%), and AZ91 (380%) at temperature of 250°C and strain rate of 0.003 1/s. Moreover, ECAP technology increases the room temperature elongation of commercial cast magnesium alloys up to 3 times without decreasing tensile strength. The model of superplastic flow rheology of microcrystalline magnesium alloys is developed; it is shown that non-equilibrium state of grain boundaries is the main factor influencing the deformation behavior of microcrystalline magnesium alloys at low-temperature superplasticity. The authors thank International Scientific and Technical Center (Grant #2809) and U.S. CRDF (Grant Y2-P-01-04) for financial and technical support.

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Low Temperature Hydrostatic Extrusion of Magnesium Alloys: *Jan Bohlen*[1]; Jacek Swiostek[1]; Heinz Günter Brokmeier[1]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre

Semi-finished components from magnesium wrought alloys like extruded profiles are in the focus of present studies in order to improve the properties of final structural parts. The parameter settings during extrusion, e.g. the extrusion temperature, play an important role for the development of the microstructure and therefore the resulting mechanical properties of the extruded component. In this paper hydrostatic extrusion trials using several magnesium alloys will be shown. The extrusion temperatures were varied in a range beginning as low as 100°C which resulted in a better homogeneity of the microstructure compared to trials at 200 – 300°C. A very fine grain size leads to improved mechanical properties. The microstructure and texture of extruded rods will be shown and compared to the mechanical properties. The influence of the extrusion temperature and the mechanisms that play a role during the development of the microstructure will be discussed.

2:40 PM

The Temperature Dependent Role of Twinning in the Ductility of Magnesium Alloy Sheet: *Ashutosh Jain*[1]; Sean R. Agnew[1]; [1]University of Virginia

A polycrystal plasticity simulation code was used to model experimental compression test data obtained from AZ31 sheet over the temperature range T = 300-473 K. The resulting model predicts the deformation behavior, including strength and strain anisotropy, as well as deformation texture evolution. The predicted relative activities of deformation modes suggest that excessive twinning is detrimental to ductility, as it causes rapid hardening to stress levels which result in failure. Thermally activated deformation modes, prismatic and pyramidal slip, compete with twinning and promote ductility. Twinning also has a significant impact on the flow stress during a change in strain path, such as compression along the rolling direction followed by compression along the normal direction. Such non-monotonic strain paths are ubiquitous in practical metal forming, which provides an incentive to model their impact. Accurate models could assist forming process designers, and ultimately promote the use of wrought magnesium.

3:00 PM

Microstructure Evolution during Hot Rolling of Magnesium Alloy AZ31 Strip: *Mahmoud Shehata*[1]; Jon Carter[1]; Claude Galvani[2]; Amjad Javaid[2]; Elhachmi Essadiqi[2]; Ravi Verma[1]; [1]General Motors R&D; [2]CANMET-MTL

A series of quenching experiments was designed to follow the evolution of microstructure during the hot rolling of a 5 mm strip into 1.5 mm sheet. The hot rolling schedule consists of three passes (30% reduction each) at 350 C, where the material was reheated between rolling passes so that each reduction pass started at 350 C. Samples were quenched after the passes, and after the reheats so as to monitor the dynamic recrystallization during reduction and the static recrystallization during reheat. Metallography of the quenched samples showed that the large cast grain structure is broken down by segmentation of the cast grain through localized deformation in twin bands, where dynamic recrystallization occurs in these bands as well as in the grain boundaries (necklacing). Quantitative metallography by image analysis was performed to quantify the extent of static and dynamic recrystallization at each processing step.

3:20 PM

Microstructure and Texture Evolution during the Uniaxial Tensile Testing of AM30 Alloy: *Lan Jiang*[1]; Stephane Godet[1]; John J. Jonas[1]; Alan Luo[1]; Anil Sachdev[1]; [1]McGill University

The evolution of microstructure and texture were examined in an AM30 alloy during uniaxial tensile testing under different conditions. This study is part of a systematic analysis of the formability of Mg alloy tubes during warm hydroforming. Samples were cut from extruded AM30 tubes along the extrusion direction. Microstructural and EBSD examination show that double twinning exerts an important influence on both flow and fracture at temperatures below 200°C. Dynamic recrystallization (DRX) was observed even at 150°C. However, it never goes to completion under any set of conditions. The microstructures near fracture surfaces indicate that cracks are readily formed next to twins at high strain rates; such twinning-induced cracks are evident at temperatures up to 250°C. Partial recrystallization also exacerbates the tendency for cavitation. No significant change in the texture occurred during straining along the extrusion direction.

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Numerical Modelling of Large Strain Deformation Phenomena in HCP Metals: *Julie Lévesque*[1]; Kaan A. Inal[1]; Kenneth W. Neale[1]; Alan A. Luo[2]; Raja K. Mishra[2]; [1]University of Sherbrooke; [2]General Motors Corporation

In this paper, a new constitutive framework based on a rate-dependent crystal plasticity theory is presented to simulate large strain deformation phenomena in HCP metals. In this new model the principal deformation mechanisms considered are crystallographic slip and deformation twinning. The new framework is incorporated into in-house finite element

(FE) codes and simulations are performed using two approaches. In the first approach, the Taylor theory of crystal plasticity is adopted to model the behavior of the polycrystalline material. In the second approach, each grain is represented individually using one or more finite elements. Using these two approaches, various large strain phenomena for magnesium alloys at different temperatures are investigated. These include behavior in tension and compression as well as localized deformation failures. In certain cases comparisons are made with experimental results. Limitations of the current modelling approaches will also be discussed.

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Microstructure, Mechanical Properties and Bendability of AM60 and AZ61 Magnesium Alloy Tubes: Yingxin Wang[1]; Xiaoqin Zeng[1]; Wenjiang Ding[1]; *Alan A. Luo*[2]; Anil K. Sachdev[2]; [1]Shanghai Jiaotong University; [2]General Motors

Microstructure, mechanical properties and bendability of AM60 and AZ61 magnesium alloy tubes have been investigated. AM60 magnesium alloy tube is extruded with extrusion-temperatures of 643K and 693K and average strain rates of $0.1s^{-1}$ and $0.6s^{-1}$ successfully and the extrusion-temperature is 683K and average strain rate is $0.1s^{-1}$ for AZ61 magnesium alloy tube. Lower extrusion-temperature leads to higher yield strength for AM60 magnesium alloy tube and the strain rate has no effect on the mechanical properties. Meanwhile, extrudability of AM60 alloy is better than that of AZ61 alloy. The results of bendability of AM60 and AZ61 magnesium alloy tubes show that the bendability of AM60 magnesium alloy tube is better than that of AZ61 magnesium alloy tube and lower yield strength results in the well bendability for the two magnesium alloy tubes. Furthermore, compressive stress leads to the more twinnings formed during the bending process than tensile stress.

4:40 PM
Warm Formability and Plastic Anisotropy of AZ31B Mg Sheets: *W. Bang*[1]; H. J. Sung[1]; I. J. Kim[1]; Dong-Kyun Choo[1]; Woo Jin Park[1]; In-Ho Jung[1]; Sangho Ahn[1]; [1]Research Institute of Industrial Science and Technology

Due to the limited ductility and the extensive springback at room temperature, warm forming is considered indispensable for the processing of Mg sheets. In this study, the warm formability of Mg sheets has been evaluated, having different fabricating routes such as strip cast and coil rolled, DC cast and sheet rolled, DC cast and extruded. A series of mechanical tests has been conducted to characterize the deformation behaviors of AZ31 sheets at elevated temperatures. Deep drawing tests have also been conducted using the universal drawing machine equipped with an warm toolset. Transition of drawability and failure mode was investigated in terms of measured mechanical properties, i.e. plastic anisotropy.

5:00 PM
High Internal Pressure Forming of Magnesium Tubes: *Adi Ben-Artzy*[1]; A. Spinat[2]; O. Dahan[2]; K. Siegert[3]; S. Jager[3]; Klaus Bernd Mueller[4]; T. Altan[5]; [1]Rotem Ind; [2]Magtech-Magnesium Technologies; [3]Stuttgart University; [4]Technische University; [5]Ohio State University

High-strength steels, aluminum, and polymers are already being used to reduce the weight of various components in many fields, but much additional reduction could be achieved by greater use of low-density magnesium (Mg) and its alloys. Lightweight components will improve ease of use, performance and structure of many applications, especially where mobility is essential. In transportation (bicycle, vehicle, spacecraft etc.) applications - for example, the steel in a chassis is strong and relatively cheap but is also very heavy. A reduction in vehicle weight will reduce fuel consumption and emission without degrading performance. Magnesium is an attractive material, primarily because of its light weight (Mg density ~ 1.7 g/cm^3) - it is 36% lighter per unit volume than Al and 78% lighter than iron (Fe). When alloyed, it has the highest strength-to-weight ratio of all the structural metals, about 65% of that of Aluminum. Furthermore, Mg is the eighth most abundant element; seawater, the main source of supply, contains 0.13% Mg, which represents a virtually unlimited supply. The objective of this research was to develop a method for the manufacturing of lightweight components from magnesium alloys, for all stages of manufacturing up to the semi-finished part, where the rest of the finishing can be done in conventional methods. Internal High Pressure forming (IHP-forming) is a well-known technique for the production of structure parts in the automotive industry. Especially in the body structure, weight saving is an important design issue, which can be realized by load optimized design and the use of lightweight materials, such as aluminum and magnesium. However, magnesium has a hexagonal lattice structure and shows a low formability at room temperature, as well as different tensile- and compression properties. Beside the strength properties, the formability of the extrusions plays a key role for potential applications. First investigations on hot extruded magnesium tubes show that a maximum hoop strain of 20% can be realized under plane strain condition, if the forming temperature is increased to 350°C. However, the tube does not expand uniformly, which leads to a non-uniform wall thickness reduction of the IHP-formed part. The reasons for the non-uniform expansion might be found in the hot extrusion technique (indirect hot extrusion, direct hot extrusion with moving or fixed mandrel) as well as in differences of the grain size of hot extruded tubes. Some complex magnesium technological demonstrators were formed by Internal High Pressure forming, using hot in-direct extruded Mg-tubes. These tubes where formed in certain parameters and showed a good formability in radial direction, as well as high strength properties, which are required for structure parts.

Materials Design Approaches and Experiences II: New Tools

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

Wednesday PM — Room: 202B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Gregory B. Olson, Northwestern University

2:00 PM Invited
Integration of Computational Tools for Predicting Thermodynamics and Precipitate Microstructure Evolution: T. Wang[1]; Y. Wang[1]; C. Ravi[1]; S. Y. Hu[1]; J. X. Zhang[1]; S. H. Zhou[1]; Christopher M. Wolverton[2]; Zi-Kui Liu[1]; *Long Qing Chen*[1]; [1]Pennsylvania State University; [2]Ford Motor Company

This presentation will report our recent progresses in establishing a MATerials Computation And Simulation Environment (MATCASE) that integrates first-principles calculations, computational thermodynamics (CALPHAD), phase-field models, and object-oriented finite element computation of macroscopic properties. In particular, it will be shown that it has now become a routine practice to rely on first-principles calculations to provide some of the necessary data for constructing thermodynamic, kinetic, and lattice parameter databases using the CALPHAD approach. These databases are necessary for the inputs to phase-field simulations of precipitate microstructure evolution. The advantages and limitations of various models to incorporate CALPHAD thermodynamics within the phase-field formulation will be discussed. Examples to be presented include precipitation reactions in binary and ternary Ni-base superalloys as well as Al-alloys.

2:30 PM Invited
Software Implementation of Computational Materials Dynamics for Accelerated Design of Hierarchical Materials: *Herng-Jeng Jou*[1]; Gregory Olson[2]; [1]QuesTek Innovations LLC; [2]Northwestern University/ QuesTek Innovations LLC

Computational materials design and accelerated insertion of materials (AIM) rely on a set of interacting mechanistic process/structure and structure/property models. This presentation will discuss the recent development of PrecipiCalc™ and the Computational Materials Dynamics (CMD) software platform, which provide robust and efficient implementation of accelerated material design. The PrecipiCalc software provides detailed high fidelity precipitation kinetic simulations for material process optimization, as successfully demonstrated under the DARPA AIM initiative on

Ni-based superalloys. Integration with underlying CALPHAD-based tools and with a high-level optimization engine, such as iSIGHT, are critical software components and are fully implemented in the PrecipiCalc and CMD system. In addition, a graphical user interface of the CMD system was developed to unify the operation and accelerate the testing/development of the software tools. Examples of accelerated material developments with these software tools will be discussed.

3:00 PM Invited
Modeling the Material Properties and Behaviour of Multicomponent Alloys: Nigel John Saunders[1]; Zhanli Guo[2]; Alfred Peter Miodownik[2]; *Jean-Philippe Schille*[2]; [1]Thermotech Ltd; [2]Sente Software Ltd.

This presentation describes the development of a multi-platform software programme called JMatPro for calculating the properties and behaviour of multi-component alloys. These properties are wide ranging, including: •Thermo-physical and physical properties (from room temperature to the liquid state). •Temperature dependent mechanical properties up to the liquid state. •TTT/CCT diagrams of steels, Al-alloys, Ni-based superalloys, Ti-alloys. •Physical and mechanical properties of steels as a function of time and temperature during quenching. A feature of the new programme is that the calculations are based on sound physical principles rather than purely statistical methods. Thus many of the shortcomings of methods such as regression analysis can be overcome. It allows sensitivity to microstructure to be included for many of the properties and also means that the true inter-relationship between properties can be developed, for example in the modelling of creep and precipitation hardening.

3:30 PM Invited
Development of Modeling Tools for the Prediction of Materials Properties: *Fan Zhang*[1]; S.-L. Chen[1]; K.-S. Wu[1]; Y. Yang[1]; W. Cao[2]; Y. A. Chang[2]; [1]CompuTherm LLC; [2]University of Wisconsin-Madison

Materials with desired properties are constantly needed in today's world. To achieve desired microstructures and mechanical properties, materials must be improved by adjusting alloy chemistry and processing conditions. Traditionally, these improvements have been made by a slow and labor intensive series of experiments. Today, different modeling tools have been developed that allow the simulation of material properties. Implementation of such tools has been proved to be useful in materials development and improvement and has resulted in significant cost savings through the elimination of shop/laboratory trials and tests. In this talk, we will present the simulation tools developed at CompuTherm with respect to both thermodynamic and kinetic modeling aspects. We will discuss the features of Pandat for multi-component phase equilibrium calculation. We will also show how PanEngine, the calculation engine of Pandat, can be connected with kinetic models for the kinetic simulation. Examples for industrial applications will be demonstrated.

4:00 PM Break

4:15 PM Panel Discussion
State-of-the-Art Modeling and Future Needs: Moderated by M. Fahrmann, Special Metals Corporation

Materials in Clean Power Systems: Applications, Corrosion, and Protection: Interconnection and Sealing in Fuel Cells III

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

Wednesday PM Room: 212B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Lichun Leigh Chen, Engineered Materials Solutions Inc; Jin Yong Kim, Pacific Northwest National Laboratory

2:00 PM
Development of a Compliant Seal for Planar SOFCs: *K. Scott Weil*[1]; Brian J. Koeppel[1]; [1]Pacific Northwest National Laboratory

A critical issue in developing high performance planar solid oxide fuel cell (pSOFC) stacks is appropriate seal design. To date, essentially two standard methods of sealing are used by stack developers: (1) rigid seals (e.g. glass) or (2) compressive seals (typically mica based). Each method has its own set of advantages and design constraints. We are developing an alternative approach that conceptually combines advantages of both techiques, including hermeticity, mechanical integrity, and minimization of interfacial stresses in either of the joint substrate materials, particulary the ceramic. The new sealing concept relies on a plastically deformable metal seal; one that offers a quasi-dynamic mechanical response in that it is adherent to both sealing surfaces. We will present recent experimental data on one version of this seal, as well as results from modeling of a full-scale design.

2:25 PM
Effect of Cathode and Electrolyte Transport Properties on Chromium Poisoning in Solid Oxide Fuel Cells: *Jeffrey W. Fergus*[1]; [1]Auburn University

A major degradation mechanism in solid oxide fuel cells (SOFC) is poisoning of the cathode by chromium from volatilization of the interconnect materials. The chromium deposition is generally considered to occur by an electrochemical reaction, which can only occur where both ions and electrons are available. For a purely ionic conducting electrolyte and a purely electronic conducting cathode, such an electrochemical reaction can only occur at the three-phase gas-electrolyte-electrode interface. However, the introduction of ionic conductivity into the cathode or electronic conductivity into the electrolyte can allow deposition to occur away from this three-phase interface, and thus alter its effect on the fuel cell performance. In this paper, the chromium poisoning of SOFC cathodes will be reviewed, with a focus on the effects of the transport properties of the cathode and electrolyte materials.

2:50 PM
Oxidation Resistance and Mechanical Properties of Experimental Low Coefficient of Thermal Expansion (CTE) Ni-Base Alloys: *David E. Alman*[1]; Paul D. Jablonski[1]; [1]U.S. Department of Energy

Energy generation systems must operate at higher temperatures and pressures in order to achieve increased efficiency. This may require the utilization of high temperature, high strength Ni-base alloys. However, the high thermal expansion coefficients (CTE) of commercially available Ni-base alloys relative to low cost ferritic steels, may make it difficult to employ Ni-base alloys in hot sections. Utilizing prior work by Yamamoto et al[1] and the ThermoCalc phase prediction software, a series of Ni-based alloys were developed at the Albany Research Center based on the composition range of Ni-(18-25)Mo-(8-15)Cr-1Ti-0.5Mn, with CTEs similar to ferritic steels. The oxidation behavior was measured at 800C in moist air. The tensile properties were measured at 25C and 750C after aging at 750C for upwards of 1000 hours. The results were compared to Haynes 230 and Haynes 242. [1]Yamamoto et al. Energy and Technology, 21, 2002.

3:15 PM

Development of Clad Metals for Planner SOFC Interconnects: *Lichun Leigh Chen*[1]; Z. Gary Yang[2]; G. G Xia[2]; Jeff W. Stevenson[2]; [1]Engineered Materials Solutions Inc; [2]Pacific Northwest National Laboratory

Recent research and development in metallic interconnect materials for solid oxide fuel cells (SOFC) at operating temperatures in the range of 650~ 850°C have focused mainly on two major alloy groups to meet the multiple requirements. That is ferritic stainless steels, and Ni-based heat resistance alloys. Coating is also being studied to improve the material performance. In the present work, a different material option, clad metals, is explored, in order to combine the merits and overcome the shortfalls of different alloys or alloy groups. In addition, the applications of clad metal technologies can allow dealing with the dual environments of the SOFC interconnect separately at each side. Roll-bonded clad metals with a high chromium ferritic stainless steel substrate and Ni-based superalloy surface clad layers were processed and tested under the SOFC interconnect service conditions. This paper will present the details of this developing effort.

Multicomponent-Multiphase Diffusion Symposium in Honor of Mysore A. Dayananda: Surfaces and Interfaces

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

Wednesday PM
March 15, 2006

Room: 203B
Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Yong-Ho Sohn, University of Central Florida

2:00 PM Invited

Single Atom Surface Diffusion: An Atomic View: *Seong Jin Koh*[1]; [1]University of Texas at Arlington

The diffusion of single atoms on atomically flat surfaces and the effects of atom-atom interactions on single atom diffusion will be presented. The diffusion of single atoms was studied by tracing the displacement of individual atoms using a field ion microscope (FIM) and model systems of Pd and W on W(110). It was found that for both Pd and W, the adatoms make long jumps in addition to nearest-neighbor jumps. The ratio of long jumps to nearest-neighbor jumps increases as temperature goes higher, indicating that at high temperatures the long jumps would prevail. The effects of adatom interactions on single atom diffusion were studied with Pd and Ir atoms on W(110) as model systems. It was found that the adatom-adatom interactions are long-range and quite anisotropic, which drastically affects the random characteristic of single atom diffusion. Supported by the Department of Energy (DEFG02-96ER-45439).

2:30 PM

Rapid Diffusion in Grain Boundary Triple Junctions: *Alexander H. King*[1]; Mysore A. Dayananda[1]; Raghavan Narayanan[1]; Shashank Shekhar[1]; [1]Purdue University

It is well-recognized that grain boundaries act as rapid diffusion paths in polycrystalline materials, and they can dominate mass transport at lower temperatures. In recent years, it has become apparent that the triple lines at which grain boundaries meet (grain boundary triple junctions, or GBTJs) may have distinct properties and behaviors from the grain boundaries themselves. In order to investigate the kinetic effects of GBTJs, we have performed a study of the diffusion of nickel into copper, using controlled tricrystals of copper. This allows us to assess the penetration of nickel into the crystal lattice, the grain boundaries and the GBTJ, independently. The results provide a clear demonstration of enhanced diffusion along the triple junctions, along with a number of other effects. The implications for mass transport in polycrystals will be discussed. Acknowledgement: This work is supported by the US Department of Energy, under contract number DE-FG01-01ER45940.

2:55 PM Break

3:15 PM Invited

Ad- and Desorption of Oxygen at Metal-Oxide Interfaces: Two-Dimensional Modelling Approaches: *A. Oechsner*[1]; M. Stasiek[1]; J. Gracio[1]; [1]University of Aveiro

Metal/ceramic phase boundaries are of great importance for many applications in materials science technology, e.g. to thin solid films or coatings. They are also included in microminiature electronic devices, e.g. MOSFETs. The presence of solute atoms at internal metal-oxide interfaces influences the physical properties of the interfaces and this, in turn, may affect the bulk properties. Therefore, it is an important task to predict and measure accurately the level of equilibrium solute-atom segregation at internal interfaces. In the presented work, the segregation of oxygen at Ag/MgO interfaces is numerically simulated whereas a general segregation kinetic which incorporates adsorption and desorption of oxygen is considered. The solution of the coupled system of partial differential equations is based on a two-dimensional finite element scheme for arbitrary oxide distribution. Based on model oxide distributions, the influence of the oxide distribution is numerically investigated and compared with the solution for equidistant arrangements.

3:45 PM

Interface Chemistry of Fe/Al-Si Bimetallic Joints: *Myriam Sacerdote-Peronnet*[1]; Jean-Claude Viala[1]; [1]University of Lyon

This contribution deals with the chemical reactivity in bimetallic couples resulting from the association of molten Al-Si alloys and ferrous base metals. The applied background concerns the development of various metallic products for the automotive and aeronautic industries.The interface reactivity of the Fe/Al-Si system was studied by two complementary approaches: experimental determination of the phase equilibria in the ternary system at 730°C (isothermal reaction-diffusion and thermal analysis), characterization of the reaction zones formed at the interfaces, depending on the temperature and the silicon content of the aluminium alloy. From the results obtained, a refined Al-Fe-Si section at 730°C and the reaction scheme describing the crystallisation sequence of Al-Fe-Si liquids below 730°C are proposed. We also determined the growth kinetics and mechanism at these interfaces. The diffusion path concept was used to relate the phase diagram of the relevant systems with the reaction layers observed at the interfaces.

4:10 PM

Interface Diffusion Phenomena in Advanced Materials: *Yury R. Kolobov*[1]; Maxim B. Ivanov[1]; Ilya V. Ratochka[2]; [1]Belgorod State University; [2]Institute of Strength Physics and Materials Science SB RAS

The characteristic regularities of diffusion and diffusion-controlled processes (grain boundary sliding, dislocation accommodation and grain boundary migration) in polycrystalline metals and alloys with bcc and fcc crystal lattice during annealing and simultaneous activity of temperature and loading during creep were considered. The particular features of the effect of grain boundary sliding activation by directed diffusion fluxes of atoms along grain boundaries are discussed. The interaction and interference of diffusion processes - grain boundary sliding and migration - as the factors determining the development of plastic deformation at conditions considered are analyzed. The determining role of diffusion-controlled processes on grain boundaries in grain boundary sliding development during creep and superplastic flow of nanostructured metals and alloys is proved. The regularities of low temperature or/and high-strain rate superplasticity manifestation in the materials indicated were investigated.

4:35 PM Concluding Comments

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials V: Phase Simulation and Interface Reactions in Solder Joints

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

Wednesday PM — Room: 213B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: C. Robert Kao, National Central University; James W. Morris, University of California, Berkeley

2:00 PM Invited
Melting Point Lowering of the Sn-Sb Alloys Caused by Substrate Dissolution: *Sinn-Wen Chen*[1]; Po-Yin Chen[1]; Chao-Hong Wang[1]; [1]National Tsing Hua University

Sn-Sb alloys are used for the soldering of ceramic devices. The liquidus and solidus temperatures of the Sn-5wt%Sb alloy are 244°C and 240°C, respectively. However, in the step soldering, the Sn-Sb solder melts at temperature lower than 232°C. Dissolution of the Ag and Cu substrates into the molten Sn-Sb alloys are determined by metallographical examinations and atomic absorption. The melting points of the ternary Sn-Sb-Cu and Sn-Sb-Ag alloys are determined using differential thermal analyzer. It is found that the substrate dissolution into the Sn-Sb alloy in the first reflowing process is the primary cause for the melting point lowering phenomenon. During the first reflow, the substrate either Cu or Ag dissolves into the molten solder, and the binary Sn-Sb alloy becomes a ternary melt. The ternary melts are with lowering melting points. Preliminary liquidus projections of the ternary systems are constructed to illustrate the solidifications of the ternary melts.

2:35 PM Invited
Numerical Prediction of Fraction of Eutectic Phase in Sn-Ag-Cu Soldering: *Machiko Ode*[1]; Minoru Ueshima[2]; Taichi Abe[1]; Hideyuki Murakami[1]; Hidehiro Onodera[1]; [1]National Institute for Materials Science; [2]Senju Metal Industry Company, Ltd

A combination of macro-scale solidification simulation and phase-field calculation is employed to predict fraction of the eutectic phase in Sn-4.0mass%Ag-XCu solder alloys (X=0.5-1.1mass%). The solidification simulation incorporates the cooling rate in the phase-field simulation. We assume the residual liquid solidifies as eutectic phase when the driving force for the nucleation of Sn6Cu5 amounts to a critical value. The driving force is estimated as the free energy difference between that of Sn6Cu5 and the undercooled melt adjacent to the primary beta-Sn phase. The driving force criteria are determined based on the force in 4.0mass%Ag-1.1mass%Cu alloy. The obtained ratio of eutectic phase shows good agreement with the experimental data.

3:10 PM
Lead-Free Soldering: The Meaning of Formation Enthalpies of Liquid and Solid Alloys: *Hans Flandorfer*[1]; Herbert Ipser[1]; [1]University of Wien

Besides its melting temperature, the solidification behavior of a solder and its interfacial reactions with substrate materials are the most important criteria for joining and the reliability of solder joints. Thus, for a systematic design of new solder materials, a comprehensive knowledge of intermetallic phases, phase relations, thermochemical properties, and diffusion is indispensable. Usually, solders are binary or higher-order alloy systems whereas substrate materials are a single component, but very often also binary or higher-order systems. The intermetallic system formed after joining is therefore of ternary or higher-order. Phase diagrams of such complex systems cannot be treated experimentally only but thermodynamic calculations according to the well established CALPHAD method are necessary. However, their quality depends strongly on experimental data like e.g., the enthalpy of formation of solid and liquid alloys. Furthermore, the formation enthalpy plays an important role for the theoretical prediction of diffusion behavior and various surface phenomena.

3:35 PM
Investigation of the Phase Equilibria of the Sn-Cu-Au Ternary, the Sn-Cu-Au-Ag Quaternary System, and Interfacial Reactions in Sn-Cu/Au Couples: *Yee-Wen Yen*[1]; Hsien-Ming Hsiao[1]; Chiapyng Lee[1]; Yu Tseng[1]; Yu-Lin Kuo[2]; [1]National Taiwan University of Science and Technology; [2]Case Western Reserve University

Sn-Ag-Cu and Sn-Cu alloys are the commercial Pb-free solders and widely used in electronic industries. The Au is commonly used in FC technology, TAB as UBM and substrate materials in PCB. In this study, the phase equilubria of the Sn-Cu-Au ternary, Sn-Ag-Cu-Au quaternary system, and interfacial reactions in Sn-Cu/Au were experimentally investigated. Results indicate that there exists a complete solid solubility between AuSn and Cu6Sn5 at 200°C and three ternary intermetallic compounds are found at 200°C. Three IMCs, AuSn, AuSn2, AuSn4 are found in all couples, and (Au,Cu)Sn/(CuxAu1-x)6Sn5 is found in all Sn-Cu/Au couples except the Sn/Au couple. Total thicknesses of reaction layers increase with higher temperature, longer reaction time and lower content of Cu, and the growth mechanism can be described by using the parabolic law. In addition, with increasing reaction time, the IMC AuSn4 disappears gradually, and turns into (Au,Cu)Sn/(CuxAu1-x)6Sn5.

4:00 PM Break

4:10 PM Invited
Growth and Reconfiguration of Interfacial Intermetallics in Solder Joints: *John W. Morris*[1]; Tae-Kyu Lee[1]; Kyu-Oh Lee[1]; [1]University of California

The bonding of microelectronic solders to the conrtacts they joined is usually accomplished by the formation of intermetallics between the Sn constituent of the solder and the various metallic constituents of the contact, primarily Cu and Ni. The nature and morphology of the intermetallic is affected by the composition of the solder and the contacts, by the temperature and duration of the reflow and by the temperature and time in subsequent service. The present talk will focus on some anomalous features and effects of intermetallic growth: exaggerated intermetallic development in asymmetric joints, anomalous precipitation in the bulk and massive reconfiguration if intermetallics during service.

4:45 PM
Effect of Co Addition to Sn-Ag Solder on Interfacial Reaction and Joint Strength of Solder with Cu: *Hiroshi Nishikawa*[1]; Akira Komatsu[1]; Tadashi Takemoto[1]; [1]Osaka University

The reaction between Sn-Ag solder added Co and Cu substrate was investigated in order to clarify the effect of the addition of Co to Sn-Ag solder on the formation of intermetallic compound at the interface and the joint strength of the solder with Cu. Sn-3.5mass%Ag-xCo solders (x = 0, 0.1, 0.3, 0.5, 1.0mass%) was specially prepared. As a result, it was shown that the thickness of the intermetallic compound (IMC) at the interface was the thinnest in binary Sn-3.5Ag solder just after reflow process at 250°C for 60s. During aging process at 150°C for 504h, in the case of solder added Co, the growth rate of the IMC layer is lower compared to the case of Sn-Ag solder. On the other hand, during aging process, the degradation of the joint strength for Co added solders is smaller than that for binary Sn-3.5Ag solder.

5:10 PM
Effect of Ni Particles and Nano-Sized Ni_3Sn_4 Powders Addition on the Interfacial Reactions between Sn-Ag-Ni Solders and Cu Substrate: Hsiang-Yi Lee[1]; *Li-Yin Hsiao*[1]; Jenq-Gong Duh[1]; Su-Yueh Tsai[1]; [1]National Tsing Hua University

Ni and nano-sized Ni_3Sn_4 powders were incorporated into Sn and Ag powders to form 95.5Sn-3.5Ag-1.0Ni solders by mechanical alloying. The morphologies of IMC at Ni-doped and Ni_3Sn_4-doped solders/Cu interfaces after reflows at 240°C were observed. $(Cu,Ni)_6Sn_5$ IMC in Ni_3Sn_4-doped solder appeared pebble-shape, while scallop-shape $(Cu,Ni)_6Sn_5$ was

revealed in Ni-doped solder. Due to the difference in thickness of IMC, the reaction was much rapid in the Ni_3Sn_4-doped solder. By quantitative analysis. The variations of Ni concentration in $(Cu,Ni)_6Sn_5$ were evaluated. The elemental redistributions of $(Cu,Ni)_6Sn_5$ were discussed and correlated to Ni-Cu-Sn ternary equilibrium.

5:35 PM

Growth of Bulk Cu-Sn Compound by Liquid Phase Electroepitaxy: *Yao-Chun Chuang*[1]; Cheng-Yi Liu[1]; [1]National Central University

Cu-Sn compounds phases, Cu3Sn and Cu6Sn5, play very important roles for the reliability of Sn-based solders jointing with Cu pads. Yet, the physical properties of Cu-Sn compounds are still not very well understood! In the talk, we will report the fabrication of bulk Cu-Sn compounds by using liquid phase electroepitaxy (LPEE) process. Under different process temperatures, Cu3Sn and Cu6Sn5 compounds can be grown, respectively. Also, from X-ray analysis, we found that LPEE-grown Cu3Sn and Cu6Sn5 compounds are nearly single crystal and have the prefer orientation along the direction of the electrical current. The plane of LPEE-grown Cu-Sn compounds, which parallel with the direction of electrical current, was found to have the largest d spacing and the lowest planar density. Besides, physical properties of Cu-Sn compounds, CTE, and electrical resistivity, will be reported in this talk.

Phase Transformations in Magnetic Materials: Information Storage

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

Wednesday PM
March 15, 2006

Room: 213A
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Kazuhiro Hono, National Institute for Materials Science; Yasukazu Murakami, Tohoku University

2:00 PM Invited

A1 to L10 Transformation in FePt Nanoparticles: *Sara Majetich*[1]; Yi Ding[1]; [1]Carnegie Mellon University

FePt nanoparticle arrays have been proposed for the next generation of data storage media, as made the coercivity is low. When the particles are annealed to induce the transformation, they sinter. Here we describe the mechanism of the A1 to L10 transformation in nanoparticles, in order to understand why the phase transformation is much more challenging in nanoparticle arrays than in thin films. We investigated the switching field distributions in samples with and without sintering. The switching field distributions are related to the volume distributions and show that the larger particles are, on average, more likely to have transformed. By repeating this process for particles of different initial sizes, we demonstrate the importance of grain boundary diffusion and the limitation of the nucleation site density.

2:35 PM Invited

Atomic Ordering in FePt Nano-Particles: Mihaela Tanase[1]; Timothy Klemmer[2]; Jian-Gang Zhu[1]; *David E. Laughlin*[1]; [1]Carnegie Mellon University; [2]Seagate Research

FePt nano-particles are being studied world wide for their potential use as ulta-high density media. The ordering process takes the FCC phase (A1,cF4) into the L10 phase (tP2). We present our recent results on the atomic ordering process in nano-particles of FePt. Of particlular interest is the effect of size and shape of the particles on the ordering kinetics and the maximum atomic order parameter obtained. Our investigations include high resolution TEM, selected area electron diffraction and nanobeam electron diffraction. This work is supported by Seagate Research, through the Data Storage Systems Center of Carnegie Mellon.

3:10 PM Invited

Formation of fct-FePt Thin Films with High Coercivity at Low Deposition Temperature: *Jun Ding*[1]; Zeliang Zhao[1]; Jingsheng Chen[2]; [1]National University of Singapore; [2]Data Storage Institute

FePt with the fct-phase is very promising as the next generation of high-density magnetic recording media, because of its large magneto-crystalline anisotropy, high coercivity and good chemical stability. However, one of the major problems need to be solved is the high formation temperature of the fct-phase. In general, the phase transformation from soft-magnetic fcc-phase to hard-magnetic fct-phase requires a relatively high temperature (> 600°C). The high formation may lead in formation of large grain and damage of substrate. Therefore, many research groups in the world are investigating how to form fct-phase at low temperature with promising structural and magnetic properties. In our work, we have successfully employed thin Ag layers to promote the formation of the hard-magnetic fct-phase. Nanostructured FePt thin films deposited on MgO at 400°C have shown very promising properties for perpendicular magnetic recording, such as high coercivity of >30 kOe and large perpendicular anisotropy.

3:45 PM

Microstructure Evolution in Fe-Pt Thin Films during Post-Deposition Ordering: *Andreas Kulovits*[1]; Bryan Webler[2]; Anirudha R. Deshpande[1]; Paul Ohodnicki[2]; Jorg Michael Wiezorek[1]; [1]University of Pittsburgh; [2]Carnegie Mellon University

The tetragonal L1o-ordered intermetallic phase FePt is offers attractive uniaxial hard-ferromagnetic properties for permanent magnet applications. FePt thin films produced by magnetron sputtering without substrate heating usually consist of the disordered FCC-solid solution, stable at higher temperatures and metastable at room temperature, rather than the L10-phase. Hence, post-deposition annealing procedures inducing the ordering transformation have to be used. Optimization of processing strategies for FePt based thin films, including alloying schemes, requires a basic understanding of their microstructural response to annealing. Here combinations of X-ray diffraction (XRD), including texture measurements, and imaging and analytical techniques of transmission electron microscopy (TEM) have been used to characterize as-deposited and annealed films in order to study the microstructural responses. The magnetic properties and domain structure of the FePt thin films have been monitored by VSM and MFM experiments.

4:05 PM Break

4:15 PM Invited

Low-Temperature Chemical Ordering in Fe-Pt Nanoparticles by Sb Doping: Qingyu Yan[1]; Taegyun Kim[1]; Arup Purkayastha[1]; Y. Xu[2]; Mutsuhiro Shima[1]; Richard Gambino[3]; *Ganapathiraman Ramanath*[1]; [1]Rensselaer Polytechnic Institute; [2]University of Minnesota; [3]Stony Brook University

Obtaining the ordered tetragonal L1-sub-0 phase at low temperatures is crucial to achieving high magnetic coercivity in nanoparticles to harness them for ultra-high-density memory devices. This talk demonstrates that Sb doping stabilizes the ordered structure in Fe-Pt nanoparticles, yielding higher magnetic coercivity at lower temperatures, than previously reported. Even as-synthesized 8-nm-diameter nanoparticle assemblies with XSb=0.23 and XSb=0.14 are ferromagnetic at room temperature with coercivity Hc ~120 mT and 10 mT, respectively. Upon annealing to temperatures as low as ~300°C, nanoparticles with XSb = 0.14 show Hc >500 mT, without particle coalescence. The coercivity is >10-times greater than previously reported for similarly-sized FePt nanoparticles and annealing temperature. XRD and TEM analyses suggest that ordering at low temperatures is due to a lattice shuffling effect caused by Sb during annealing. The ordering mechanism and implications of our results on the development of new high-coercivity nanostructures will be discussed.

4:50 PM

Numerical Simulations of Infrared Processing of FePt Nanoparticle Films: *Adrian S. Sabau*[1]; Ronald D. Ott[1]; Ralph B. Dinwiddie[1]; Puja Kadolkar[1]; Craig Alan Blue[1]; [1]Oak Ridge National Laboratory

In order to anneal FePt nanoparticle films, a plasma arc lamp was used in this study. The processing aimed at reaching a peak target temperature for multiple pulses of 550C. Numerical simulations of the heat transfer

and infrared measurements for the PTP were carried out to determine the operating power levels for the plasma arc lamp. Infrared measurements were conducted to obtain experimental data for the surface temperature of the FePt nanofilm. Parameters needed for the heat transfer model were identified based on the experimental temperature results. Following the model validation, several numerical simulations were carried out to identify the power levels for each pulse. It was shown that the FePt nanoparticle films were successfully processed using the power levels provided by the heat transfer analysis.

5:15 PM
Pulsed-Thermal-Processing of FePt Thin Films: Amanda C. Cole[1]; *Gregory B. Thompson*[1]; J. W. Harrell[1]; Ronald Ott[2]; [1]University of Alabama; [2]Oak Ridge National Laboratory

The magnetic anisotropic $L1_0$ FePt phase is a candidate material for next generation magnetic storage. When FePt is sputter deposited, it adopts a metastable A1 phase that is superparamagnetic. Conventional annealing at 600°C will phase transform FePt into $L1_0$ but result in grain coarsening and the loss of the narrow granular size distribution. We report pulsed-thermal-processing using a high density infrared plasma arc light source at exposure times of 100 to 250 ms to chemically order 20 nm and 100 nm thick FePt films on Si substrates. Upon ordering, no grain growth in the 100 nm thick films was observed. As the thin film thickness decreased, grain growth became more prevalent for similar processing conditions. Depending on the time, temperature and number of pulses, we are able to control the texture evolution of the film. XRD, TEM and magnetometry characterization have been performed and will be reported for each process.

5:40 PM
The Mechanism of (001) Texture Evolution in FePt Thin Film during Post-Annealing: *Jae-Song Kim*[1]; Yang-Mo Koo[1]; Byeong-Joo Lee[1]; Seong-Rae Lee[2]; [1]POSTECH; [2]Korea University

We discuss the origin of the (001) texture evolution of FePt thin films during post-annealing, considering order/disorder transformation strain. Strain energies corresponding to transformation strain and in-plane strain were calculated according to crystals having various normal orientations, using Molecular Statics (MS) with Modified Embedded Atom Method (MEAM). In order to confirm the calculated results, residual stress of the FePt thin films was measured with the $\sin^2\varphi$ technique modified by the present author. The theoretic calculations revealed that the transformation strain stabilizes the crystal grains with the (001) crystallographic orientation relative to the surface of the film under in-plane tensile strain and the free normal stress, which was consistent with experimental results from the residual stress measurements. From this, it is suggested that transformation strain generated in the early stages of post-annealing plays an important role in the texture evolution of FePt thin film.

Point Defects in Materials: Atomic Kinetics Processes - Joint Session with Computational Thermodynamics and Phase Transformations

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

Wednesday PM — Room: 210B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Adri C. van Duin, California Institute of Technology; Mark D. Asta, University of California

See Computational Thermodynamics and Phase Transformation Symposium on page 264 for presentations.

Recycling - General Sessions: General 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 International

Wednesday PM — Room: 8B
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Christopher M. Keller, Aleris International Inc

2:00 PM Introductory Comments

2:05 PM Invited
Immobilization Process via Solidification of Hazardous Heavy Metals for Recycling Industry Waste: *Ji-Whan Ahn*[1]; Kwnag-Suk You[1]; Seung-Hyeon Ahn[2]; Hwan Kim[2]; [1]Korea Institute of Geoscience and Mineral Resources; [2]Seoul University

The purpose of this study was to investigate the immobilization as a host for metallic pollutants (Cr, Cu, Pb, and Zn) by ettringite phase. Ettringite phase is produced through hydration reaction of calcium aluminate with gypsum. From the XRD patterns, it is known that the distance between spaces in 010 planes in ettringite crystal increase with an increase of ionic radius of heavy metal except Pb; no big difference is shown. For this reason, the d-value of 010 planes in ettringite crystal increased with the substitution of Cr, Cu and Zn having a big Al ironic radius. But in case of Pb, because the substitution occurred at calcium site, it does not influence to increase d-value of 010 planes.

2:30 PM
Effect of Magnetic Separation in Removal of Heavy Metals in Municipal Solid Waste Incineration Bottom Ash: *Nam-Il Um*[1]; Gwang-Suk You[1]; Gi-Chun Han[1]; Ji-Whan Ahn[1]; Hee-Chan Cho[1]; [1]Korea Institute of Geoscience and Mineral Resources

Incineration is a commonly used solid waste treatment in many countries and typically 20% of the mass remains as a bottom ash collected as fly ash. Although incineration has merits such as heat recovery, a small volume compared to the volume of waste incinerated, the management of residues have been become a main issue as criteria for use of the residues or landfilling. MSWI bottom ash have been considered as aggregate substitute because MSWI bottom ash consists of slag, glasses, ceramics mainly. And in several countries such as the Netherlands, Denmark, France, MSWI bottom ash have been used as road materials after separating ferrous metals. Separation of ferrous metals from MSWI bottom ash may not only give economical advantage but also removal of heavy metals. Therefore, in this study, the relationship between recovery of ferrous metals and separation of heavy metals from MSWI bottom ash was investigated.

2:55 PM
Repeatedly Utilization of Carbon Fiber Chemistry Copper Plating Waste Liquid: *Tianjiao Luo*[1]; Guangchun Yao[1]; Linli Wu[1]; Xiaoming Zhang[1]; [1]Northeastern University

How to reuse the waste liquid directly was researched in this paper. Firstly, the waste liquid should be filtrated to remove the impurity; then some of H2O2 solution was dripped into the waste liquid to pretreat it; finally, CuSO4 solution was added into the treated waste liquid and the pH was adjusted to about 12, then some HCHO solution was added into it, in which the treated carbon fiber can be plated. The morphology and composition of coatings were investigated by means of Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy. The results indicated that, as the new plating solution, and the pretreated solution is stable, and the plating rate is still about 0.8μm/h, and the copper coating is even and combines closely with carbon fiber. According to this way, the waste liquid can be reuse 5~7 times.

3:20 PM
Recycling of Sludge Derived from Cutting and Polishing Natural Stones in Ceramic Formulations: Paula Costa Torres[1]; Rodrigues Salvador Manjate[1]; Sandra Fernandes Quaresma[1]; *José Maria da Fonte Ferreira*[1]; [1]University of Aveiro

The aim of the present work was to study the incorporation of industrial sludges derived from cutting and polishing natural stones (slate, granite, and quartzite) as main components in stoneware compositions and to evaluate the impact on the final properties of the products. The materials were characterized in terms of particles size distribution, chemical (XRF) and mineralogical (XRD) compositions, and thermal behaviour (DTA, AT, Dilatometry) and density. Formulations containing 70 wt.% of residues and 30 wt.% of a plastic ball clay were prepared. The plasticity of all the pastes tested was characterised. The sintered samples were characterised for shrinkage, crystalline phase formation, water absorption, flexural strength, density and microstructure (SEM). The final properties obtained (flexural strength >35 MPa, water absorption <3%) are typical of stoneware products.

3:45 PM Break

4:00 PM
Zinc Vapor Treatment for Precious Metals Recovery: *Masao Miyake*[1]; Junichi Itoh[1]; Masafumi Maeda[1]; [1]University of Tokyo

Compound formations of precious metals by zinc vapor treatment were investigated to develop a new recovery process of precious metals from scraps. Precious metals were exposed to zinc vapor at constant temperatures of 673-973 K, and compound layers were formed on the surface of the precious metal. The growth rates of the layers followed the parabolic low, indicating that volume diffusion controlled the growth of the layers. Interdiffusion coefficients in the compound phases were derived from concentration-penetration profiles.

4:25 PM
A New Recycle Process of Uranium from Spent Fuel by Selective Sulfurization: *Nobuaki Sato*[1]; Genki Shinohara[1]; Osamu Tochiyama[1]; [1]Tohoku University

For the recovery of uranium from spent nuclear fuel, sulfide reprocessing process, which consists of voloxidation, selective sulfurization, and magnetic separation or selective dissolution steps, are proposed. In this paper selective sulfurization of rare-earth oxides in the presence of uranium oxides was studied by thermogravimetry and X-ray diffractometry. In case of the mixture with UO2, the rare-earth oxides R2O3 (R=Nd, Eu, etc.) were preferentially sulfurized by CS2 forming oxysulfides and sulfides, while UO2 remained at temperatures lower than 500°C. When R2O3 was mixed with U3O8, first, the reduction of U3O8 to UO2 occurred at low temperatures followed by the sulfurization of R2O3. The sulfurization of UO2 solid solution containing rare-earths was also investigated. The magetic separation and selective dissolution processes were discussed by the fundamental experiments using the simulated mixture of uranium and rare-earths oxides.

4:50 PM
Recycling of Oxidic Waste from the Ferroalloy Production: Markus Hochenhofer[1]; Helmut Antrekowitsch[2]; *Michael Potesser*[2]; Wolfgang Labenbacher[1]; [1]Christian-Doppler Laboratory for Secondar Metallurgy of Non-Ferrous Metals; [2]University of Leoben

Due to the economic aspects and the environmental regulations recycling of slag and waste from the ferroalloy production is a very important point for the companies. The treatment of slags depends on the composition especially on the valuable metals and impurities. Furthermore the aim is the use of these materials in the industry e.g. cement industry, pig-iron production etc. Therefore the requirements for these applications should be reached be metallurgical processing. The treatment of the slag with a high V-, Mo- and P-content by using a reducing metal bath is one alternative to recover the elements from the materials. Besides phase analysis thermodynamic calculations were done to determine the process parameters. Additionally the Christian Doppler Laboratory for Secondary Metallurgy of Nonferrous Metals at the Institute of Nonferrous Metallurgy at the University of Leoben carried out investigations with different slag compositions to verify the thermodynamic calculations.

5:15 PM Concluding Comments

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

Wednesday PM — Room: 6D
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Murat Tiryakioglu, Robert Morris University

2:00 PM Invited
Modeling of Precipitate Free Zone Developing upon Heat Treatment in Industrial Aluminum Casting: *Charles A. Gandin*[1]; Alain Jacot[1]; [1]Ecole des Mines de Paris

The formation of precipitate free zone (PFZ) upon heat treatment of industrial aluminum alloys is known to be detrimental to fracture toughness. The width of the PFZ depends on the relative importance of solute diffusion in the vicinity of primary particles as compared with the potency of precipitates to nucleate and grow in the matrix phase. A model to predict the PFZ is presented. It is based on the coupling between a Precipitate Size Distribution method and a Pseudo-Front Tracking method. Coupling with equilibrium thermodynamic calculations is achieved through Thermo-Calc. As a result of the interaction between precipitation and long range diffusion, the PFZ is predicted together with profiles of the compositions of species, the density and the volume fraction of the precipitates. The simulation results are compared with experimental data collected in the literature for an industrial homogenization heat treatment of a 3003 aluminum alloy.

2:25 PM Invited
Modeling the Heat Treatment of Age-Hardenable Cast Aluminum Alloys: *Richard Dean Sisson*[1]; Shuhui Ma[1]; Md. Maniruzzaman[1]; [1]Worcester Polytechnic Institute

The effects of solutionizing times and quenching rates on the microstructure and mechanical properties of age-hardenable cast aluminum alloys (i.e. 319 and A356) has been experimentally investigated with Jominy End Quench Bars and theoretically analyzed using Quench Factor Analysis. The results indicate that the solutionizing time for metal mold cast alloys can be reduced from 12 hours to less than 4 hours depending on the casting microstructure and secondary dendrite arm spacing. The Jominy End Quench experiments revealed that these alloys are not very quench sensitive with alloy A356 being more quench sensitive than 319. Quench Factor Analysis proved to applicable to these cast alloys and a good predictor of hardness. The results are discussed in terms of alloy microstructure including Silicon spheroidation and hardening precipitate development.

2:50 PM
Optimization of Load Design and Thermal Recipe Design for Time and Energy Efficient Heat Treatment Processes: *Yao Zhou*[1]; Jinwu Kang[1]; Yiming Rong[1]; [1]Worcester Polytechnic Institute

Aluminum alloy solutionizing is a time and energy consuming process. Based on the process model built in our previous research, the processes can be simulated, evaluated, and optimized. This research examined two possibilities to reduce cycle time and energy consumption: load design and thermal recipe design. Load arrangement can be quantitatively assessed and determined among several designs to achieve shorter cycle time and lower energy consumption. Thermal recipe can be such designed that the furnace is first heated over the soaking temperature so as to heat the load faster, and then furnace is brought back to the soaking temperature at a proper time so the load finally rises to the soaking temperature

without overheat. This "proper time" is critical and is calculated and controlled by the algorithms. Comparative study shows that the proposed optimizations in load design and thermal recipe design yield significant saving on both time and energy.

3:15 PM

Effect of Various Casting Parameters on Air Gap Formation and Interfacial Heat Transfer Coefficient for Various Aluminum Alloys Cast against Different Metal Molds: *Stavros A. Argyropoulos*[1]; Horazio G. Carletti[1]; Basil L. Coates[1]; [1]University of Toronto

This paper will be focused on the effects of surface roughness and temperature and metallostatic head on the heat transfer coefficient at the metal mold interface. The experimental work was carried out in a special apparatus developed to measure heat transfer at the metal mold interface. This apparatus was instrumented with two types of sensors, thermocouples and LVDT's(Linear Variable Differential Transformer). The monitoring of the two types of sensors was carried out simultaneously during solidification of various aluminum alloys. These alloys were cast against different metal molds. Inverse heat transfer analysis was used to estimate the heat transfer coefficient and the heat flux at the metal mold interface. In general, an increase in mold surface roughness results in a decrease in the heat transfer coefficient at the metal mold interface. On the other hand, an increase in metal temperature results in an increase in the heat transfer coefficient.

3:40 PM Break

3:55 PM

Numerical Modeling of the Heat Treatment with Residual Heat of Aluminum-Alloy Castings: *He Liang*[1]; Kang Jinwu[1]; Huang Tianyou[1]; [1]Tsinghua University

Aluminum-alloy castings are frequently used by the automotive industry in both as-cast and heat-treated conditions. The purpose of heat treatment is to obtain a better combination of strength and ductility, but the heat treatment demands a very large cost of electric energy. The heat treatment with residual heat of aluminum-alloy castings, immediately after their solidification, can reduce reheating, cycle times and energy consumption. A three-dimensional model for the solidification, solution and artificial aging is presented in this study. This model predicts the temperature distribution during the whole heat treatment procedure and can do help on the design of the heat treatment parameters. The model is used for the 319 aluminum-alloy castings and the simulation results are in good agreement with experimental results.

4:20 PM

Thermal Contact in Permanent Molding of Aluminum Alloys: *John Trevor Berry*[1]; Rogelio Luck[1]; Robert D. Pehlke[2]; August Johnson[1]; Jeffrey Weathers[1]; [1]Mississippi State University; [2]University of Michigan

The problem of adequately describing the conditions of thermal contact in casting processes involving metallic molds, especially where a protective coating is in place, has been investigated almost continually over the last quarter century. A recent investigation has coupled both analytical and computational techniques with careful measurement in order to obtain reliable data describing the time/temperature dependant heat transfer coefficients concerned and their relation to current coating practice. Additionally, the invasive nature of temperature sensing devices in the mold and how true time-temperature data might be recovered for use in inverse analysis has been studied. (The work has been supported by the US Dept. of Energy through the Cast Metals Coalition and by Mississippi State's Center for Advanced Vehicular Systems).

4:45 PM

Modeling the Quench Sensitivity of an Al-7wt.%Si-0.6wt.%Mg Alloy: *Murat Tiryakioglu*[1]; Ralph Shuey[2]; [1]Robert Morris University; [2]Alcoa Technical Center

The quench sensitivity of a cast Al-7wt.%Si-0.6wt.%Mg alloy was characterized by tensile tests and scanning electron microscopy. Samples were cooled from the solution treatment temperature by interrupted and delayed quenches. Mg2Si was found to nucleate on Si particles as well as in the matrix. The quench sensitivity was modeled by double-C curves following the quench factor analysis methodology.

Solidification Modelling and Microstructure Formation: A Symposium in Honor of Prof. John Hunt: Solidification Processing and Thermophysical Properties

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

Wednesday PM — Room: 6C
March 15, 2006 — Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: P. Grant, University of Oxford; L. Katgerman, Delft University of Technology

2:00 PM Invited

Evaluation of Kinetic Competition during Solidification: *John H. Perepezko*[1]; Kjetil Hildal[1]; [1]University of Wisconsin

The evolution of an as-cast microstructure is related to the dynamic kinetic competition during solidification. Microstructure morphology transitions from cells to dendrites and the columnar to equiaxed grain transition reflect a kinetics competition that is motivated by a changing interface velocity and interface undercooling. Similarly, the conversion between metastable and stable phase products and the crystal to glass reaction are related directly to a changing melt undercooling. While growth kinetics transitions tend to be sharply defined, nucleation controlled transitions occur over a range. Traditionally, solidification transitions have been examined by multiple directional freezing runs, but with a wedge mold castings can be subjected to a range of solidification rates differing by orders of magnitude. With microstructure analysis, temperature measurements and heat transfer analysis, wedge casting can be used to explore a spectrum of solidification microstructures and provide new insight on the processing conditions as illustrated for bulk glass formation.

2:25 PM

Issues in the Computational Modeling of Solidification and Casting Processes: *Mark Cross*[1]; Nick Croft[1]; Diane McBride[1]; Alison Jayne Williams[1]; Kyriacos A. Pericleous[2]; [1]University of Wales Swansea; [2]University of Greenwich

Solidification modelling of metals casting, whether for shape, (semi-) continuous or in the manufacture of thin strip is now a mature activity. There are now a wide range of software tools addressing all aspects of this family of processes. Indeed, the main challenge here is perceived as being in capturing the micro-structure within the context of a macro-scale process, the area precisely where John Hunt made many of his achievements over his career. In this paper we will focus upon a range of challenges that still remain to enable effective and comprehensive simulation at the macro-scale to ensure the arising computational models are appropriate hosts for capturing micro-structural behaviour. These challenges will include: 1) macro-segregation for multi-component alloys; 2) mould filling and simultaneous solidification in very complex geometries; 3) closely coupled thermo-fluid-structure interaction in the metal-mould context; 4) simulation issues for operating at multiple length and time scales.

2:50 PM

Fractional Latent Heat Data from Heat Flux Differential Scanning Calorimetry: *Adrian S. Sabau*[1]; Wallace D. Porter[1]; [1]Oak Ridge National Laboratory

Differential Scanning Calorimetry (DSC) measurements are used to estimate the fractional latent heat release during phase changes. There are instrument temperature lags due to the temperature measurement at a different location than that of the sample and reference materials. Recently, Dong and Hunt (2001) showed that significant improvement in estimating the fractional latent heat can be obtained when a detailed simulations of the heat transfer within the instrument are performed. The Netzsch DSC 404C instrument, with a high accuracy heat capacity sensor, is considered in this study. This instrument had a different configuration that

that studied by Dong and Hunt (2001). The applicability of Dong and Hunt's approach to this instrument is investigated. It was found that the DSC instrument can be described by numerous parameters. It was difficult to identify model parameter. Numerical simulation results are presented and compared with experimental results for the fractional latent heat.

3:10 PM

Determination of Solute Diffusion Coefficient by Droplet Migration Method: *Shan Liu*[1]; Jing Teng[1]; [1]Iowa State University

Liquid droplets can migrate through a solid matrix in a temperature gradient field. Watson and Hunt employed this method to obtain the solute diffusion coefficient (D_L) in Al-Cu alloys by interrupted experiments in order to determine the position of a band of droplets. In this study, we used transparent SCN base alloys and obtained the solute diffusion coefficient by quantifying the migration behavior of an individual droplet. We find that there is a characteristic migration curve for an alloy at each temperature gradient that all the droplets follow, indicating that the instant velocity of droplets only depends on the position in the temperature field and is independent of the droplet size and shape. The diffusion coefficient is determined through the this characteristic curve, which gives D_L in the SCN-salol system as 0.65×10^{-9} m^2/s and D_L in the SCN-camphor system as 0.26×10^{-9} m^2/s.

3:30 PM Invited

The Contribution of John Hunt to Measurement of Solid/Liquid Interfacial Energies in Metallic Systems: *Howard Jones*[1]; [1]University of Sheffield

Solid-liquid interfacial free energy σ_{SL} plays a key role in a wide range of metallurgical phenomena. Pioneer estimates by Turnbull et al in the 1950's from undercooling measurements were primarily for pure metals on the assumption of homogeneous nucleation and estimates from dihedral angle measurements require an independent measurement or estimate of σ for at least one of the other interfaces involved. Gündüz and Hunt in 1985 reported the first direct measurements of σ_{SL} for three eutectic alloy systems from the profiles of grain boundary grooves stabilised by a known radial temperature gradient, embodying a methodology applied prior to 1985 only to transparent systems or pure metals at or near to ambient temperatures. The significance of these and subsequent measurements and their application to solidification theory will be critically evaluated.

3:55 PM Break

4:10 PM

Measurement of Solid-Liquid Interface Energies in Ternary Alloys: *Annemarie Bulla*[1]; Carlos Carreno-Bodensiek[1]; Bjoern Pustal[1]; Ralf Berger[1]; Andreas Bührig-Polaczek[1]; Andreas Ludwig[2]; [1]RWTH Aachen University; [2]University of Leoben

The solid-liquid interface energy plays a major role in phase transformations. It has a strong influence on solidification morphologies and the final grain structure. The "grain boundary groove in an applied temperature gradient" method developed by J. Hunt/ Oxford was found to be suitable for measuring the solid-liquid interface energy in metal alloy systems. A radial heat flow apparatus was assembled as described by Hunt and Gündüz. This apparatus ensures a stable temperature gradient for hours and thus allow the sample to equilibrate. After rapid quenching, the local curvature of the groove can be analysed. From the graphically measured boundary groove information and the simulated local temperatures, the solid-liquid interface energy can be evaluated indirectly using the Gibbs-Thomson equation. Values for the solid-liquid interface energy and the Gibbs-Thomson coefficient have been obtained for the Al-Cu-Ag ternary eutectic system. The relationship between concentration and solid-liquid interface energy was also investigated.

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Microstructure and Creep Resistance of Lead-Free Solder under Different Solidification Conditions: Jin Liang[1]; N. Dariavach[1]; *Dongkai Shangguan*[2]; [1]EMC; [2]Flextronics

Mechanical, metallurgical, thermal and environmental factors all affect the service reliability of solder joints, and are under extensive study for preparation of the transition from Sn-Pb eutectic soldering to lead-free soldering in the electronic industry. However, there is a general lack of understanding about the effects of solidification conditions on the microstructures and mechanical behavior of lead-free solder alloys. This study attempts to examine the creep resistance of the Sn-Ag-Cu eutectic alloy with a variety of solidification conditions with cooling rates ranging from10+2C/sec to 10-2C/sec. Although real soldering processes have limited solidification rate variations, an understanding of the mechanical property change with microstructures, which are determined by the solidification conditions, should shed some light on the fundamental deformation and fracture mechanisms of lead-free solders.

4:50 PM

Preparation of AZ91D Slurries for Semi-Solid Forming Using Precipitates and Cast Properties: *Ha Heon Phil*[1]; Byun Ji Young[1]; [1]Korea Institute of Science and Technology

During the solidification of the AZ91D-alloys, the Al8(Mn,Fe)5 phase is generally precipitated in the melt in advance of the precipitation of the primary a-Mg. The basic principle for manufacturing AZ91D-alloy slurries for semi solid forming is to use the Al8(Mn,Fe)5 precipitates as the heterogeneous nucleation sites for primary a-Mg phases. Al8(Mn,Fe)5 precipitate is the effective heterogeneous nucleation site for the primary a-Mg phase. It was also observed that increase of the Mn content in the melt and the cooling rate below to the solid/liquid two-phase region resulted in smaller and more globular primary a-Mg due to the increase of heterogeneous nucleation sites. The cooling rate below to the solid/liquid two-phase region, Mn content in AZ91D alloy, and the holding time and temperature affected on the quality of slurry. After annealing rheo-cast samples, mechanical properties were examined.

5:10 PM

Solid-Liquid Interfacial Energy of Tin in the Cd-Sn Eutectic System: *Buket Saatçi*[1]; Sevgi Çimen[1]; Hakan Pamuk[1]; Mehmet Gündüz[1]; [1]Erciyes University

When the stabilized solid-liquid interface intersects with a planar grain boundary in a temperature gradient, G a shape of the grain boundary groove is formed. The equilibrated groove shapes were directly observed after annealing the sample at just above the eutectic temperature for about 8 days. The thermal conductivities of the solid phase, Ks and the liquid phase Kl for the groove shapes were measured. From the observed groove shapes the Gibbs-Thomson coefficients have been obtained for the solid Sn in Cd-Sn liquid solition by using the measured G, Ks and Kl with a numerical method. The solid-liquid interfacial energy, between solid Sn and Cd-Sn liquid solition has been determined from the Gibbs-Thomson equation. The grain boundary energy for the same material has been callucted from the observed groove shapes.

5:30 PM

Measurement of Solid-Liquid Interfacial Energy for Solid Zn in Equilibrium with the Zn-Mg Eutectic Liquid: *Mustafa Erol*[1]; Kazim Keslioglu[1]; Necmettin Marasli[1]; [1]Erciyes University

The equilibrated grain boundary groove shapes for solid Zn in equilibrium with the Zn-Mg eutectic liquid were observed on rapid quenched samples. The Gibbs-Thomson coefficient for the solid Zn have been determined to be $(10.64 \pm 0.43) \times 10^{-8}$ Km from the observed grain boundary groove shapes with present numerical model and the solid-liquid interfacial energy for the solid Zn in equilibrium with the Zn-Mg eutectic liquid has been obtained to be $(89.16 \pm 8.02) \times 10^{-3}$ Jm^{-2} from the Gibbs-Thomson equation. The grain boundary energy for the solid Zn has been calculated to be $(172.97 \pm 20.76) \times 10^{-3}$ Jm^{-2} from the observed grain boundary groove shapes. The thermal conductivity of the solid phase and thermal conductivity ratio of liquid phase to solid phase for Zn-0.15wt.%Mg alloy have also been measured.

Surfaces and Interfaces in Nanostructured Materials II: Coatings, Films, Multi-Layers and Arrays

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

Wednesday PM Room: 209
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chair: Sudipta Seal, University of Central Florida

2:00 PM
Deposition, Characterization and Performance Evaluation of Nano-Layered Superlattice Nitride Coatings: *Qi Yang*[1]; Linruo Zhao[1]; [1]National Research Council Canada

Nano-layered superlattice nitride coatings with two or multi- layer constituents were deposited by unbalanced reactive magnetron sputtering technique using metallic targets. The modulation periods of the superlattices were measured according to the reflection peak positions in their low-angle X-ray reflectivity spectra using the modified Bragg law, a technique proven to be accurate by TEM analysis. Superlattice structures can lead to a remarkable higher hardness than monolayered nitride coatings; and hardness enhancement is closely related to the superlattice layer constituents, modulation period and coating crystallographic orientation. Post-annealing tests indicate that nitride superlattices have a high thermal stability. Pin-on-disc dry sliding test shows that the nitride superlattices exhibit lower friction coefficients and markedly higher wear resistances than commercial TiN hard coating. These tribological properties make these superlattice coatings a strong candidate for surface protection against wear in engineering applications.

2:20 PM
Nanoscale Coatings for Surface Modification of Carbon Structures: *Rajasekhar V. Pulikollu*[1]; Pratik Joshi[1]; Sharmila M. Mukhopadhyay[1]; [1]Wright State University

For nanoscale materials to be implemented in useful devices, control of surface functionality for appropriate bonding or dispersion is very important. It is important that the surface modification is limited to the top few atomic layers and does not degrade the underlying structure. In this project, microwave plasma coatings having thickness less than 5 nm can be applied successfully to modify complex and uneven carbon structures like carbon foam and nanofibers. This talk will focus on silica-like oxide coating that has been successfully developed for enhancing the bond between carbon reinforcements and matrix materials in composites. Characterization techniques such as XPS, SEM, TEM, AFM and mechanical testing are used to study the chemistry, morphology, structure and effectiveness of these coatings. Additionally, the influence of these coatings on nucleation and growth of nanotubes on larger carbon structures (to produce multiscale, multifunctional materials) will be presented.

2:40 PM
Role of Interfaces in Determining Phase Stability in Nanoscale Multilayers: *Arda Genc*[1]; Rajarshi Banerjee[1]; Mark Yavorsky[2]; Dennis Maher[2]; Hamish L. Fraser[2]; [1]University of North Texas; [2]Ohio State University

When the layer thickness in a multilayered thin film is reduced to the nanometer scale, the interfaces between the layers dominate the free energy of the system consequently dictating the structure of the individual nanolayers. While the phase stability in these multilayers can be rationalized based on a thermodynamic model, their predictive capability depends critically on the accuracy of input computational parameters such as the interfacial energy. Accurate computation of such interfacial energies in turn depends on the detailed characterization of the atomic-scale structure and chemistry of the interfaces between the nanolayers. This presentation will focus on the application of multiple high-resolution characterization techniques, including, high-resolution TEM, electron-energy loss spectroscopy at a high spatial resolution, and, 3D atom-probe tomography, for the characterization of these interfaces at the atomic-scale.

3:00 PM
Tailoring the Texture of Magnetron Sputtered Ta Films by Self-Assembled Dendrimer Sublayers: *Xiao Li*[1]; Feng Huang[1]; Shelby F. Shuler[1]; Shane C. Street[1]; Mark L. Weaver[1]; [1]University of Alabama

In this study, we demonstrate the use of ultrathin (<5 nm in thick) self-assembled dendrimer monolayers to tailor the texture of the magnetron sputtered Tantalum (Ta) films. As characterized by X-ray diffraction (XRD) techniques, dendrimer-mediated films developed a (330) preferred orientation in the tetragonal ß-Ta phase, which is in contrast to the (002) preferred crystalline orientation in the bare Si substrates. Variation of the sputtering conditions and the generation of the dendrimers, alters the ratio of the intensity from the two peaks, yielding weak or strong (330) texture. A discussion of the nucleation and the competitive growth is conducted for the origin of the tailored Ta texture.

3:20 PM
Three Dimensional Imaging of Cracking in Berkovich Indented Thin Film Coatings: Lok-Wang Ma[1]; Damien McGrouther[1]; *Paul Munroe*[1]; Julie Cairney[1]; Mark Hoffman[1]; [1]University of New South Wales

A TiN thin film coating deposited on a ductile steel substrate, was subjected to surface deformation via nanoindentation using a Berkovich indenter. Pop-ins were observed during loading that are characteristic of the onset of cracking and the formation of shear steps at the coating-substrate interface. Focused ion beam microscopy was used to prepare and observe cross-sections through the indentation that revealed the presence of both intercolumnar and inclined cracks. However, the pyramidal nature of the indenter means that the cracks observed in these 2-D sections are strongly dependent on the location of the milled region. In this study we have used dual-beam focused ion beam instrumentation to acquire datasets used to construct 3-D visualisations of the indented regions. These images provide highly detailed images of the morphology of cracks, which were observed to be consistent with theoretical models of plastic deformation of such brittle coatings.

3:40 PM
Synthesis of Nanocrystalline Intermetallic Layer by Mechanical Attrition: *Xiao-Lei Wu*[1]; You-Shi Hong[1]; Nai-Rong Tao[2]; Xiang-Kang Meng[3]; Jian Lu[4]; Ke Lu[2]; [1]Institute of Mechanics, Chinese Academy of Sciences; [2]Institute of Metal Research, Chinese Academy of Sciences; [3]Nanjing University; [4]University of Technical of Troyes

By means of the technique of surface mechanical attrition treatment, the nanocrystalline (nc) intermetallic compound Co3Fe7 was synthesized in the surface layer of bulk cobalt. It was found that diffusion occurred during deformation, leading to the extension of solid solution and a series of phase transformation. Fe contents were significantly higher in the grain boundary and triple junction than in grain interiors. In particular, stacking faults were found to contribute diffusion significantly. The alloying on the atomic level was ascribed to deformation-induced intermixing during deformation. The superimposed effects of deformation at high strain rates on diffusion were analyzed in terms of dislocation solute-pumping mechanism and enhanced mobile vacancy concentration. The mechanism of intermetallic formation was a result of numerous nucleation events followed by limited growth.

4:00 PM
Surface Nanostructuring with Ordered Arrayed Nanoparticles of Tunable Size, Shape and Property: *Yong Lei*[1]; Gerhard H. Wilde[1]; [1]Forschungszentrum Karlsruhe GmbH

Nanoparticle arrays are fabricated on Si and Si/SiO2 substrates using nanoporous ultra-thin alumina membranes (UTAMs) as evaporation masks. Because the shape and size of the nanoparticles are controllable, it is possible to tune the properties of the nanoparticle arrays, which will be highlighted by selected examples of our recent work. Ordered CdS nanoparticle arrays on Si substrates with tunable photoluminescence properties have been synthesized. Moreover, ordered arrays of In2O3 single-crystal nanoparticles were obtained based on a controlled oxidation of In nanoparticles. All In2O3 nanoparticles are oriented in the similar lattice

direction, which offers an additional control parameter i.e. a high degree of orientational order. This behavior is thought to be related to the so-called "two-dimensional nucleation and layered growth". The methods developed here are applicable in a large range of possible processes and applications and serve to develop highly defined nanostructured surfaces that are structured in a massive parallel manner.

The Brandon Symposium: Advanced Materials and Characterization: Microstructure and Properties

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

Wednesday PM Room: 206B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Paul Wynblatt, Carnegie Mellon University; Dominique Chatain, Centre National de La Recherche Scientifique

2:00 PM Invited
Low Temperature Colossal Supersaturation (LTCSS) Surface Hardening of Stainless Steels via Ti Alloys: *Arthur H. Heuer*[1]; [1]Case Western University

Attempts to surface harden austenitic stainless steels by traditional carburization processes, without adversely affecting other useful properties, have traditionally failed due to formation of stable Cr-bearing carbides. We have demonstrated that with suitable surface activation, a temperature "window" exists where interstitial carbon diffusion can occur in the absence of substitutional diffusion of solutes such as Cr. Under such conditions, colossal supersaturation of carbon can occur (>10 atomic %), leading to impressive surface hardening and very high surface residual compressive stresses, improved fatigue resistance, and improved corrosion resistance, without serious loss of ductility.

2:25 PM Invited
Do Cavities Form during the Fracture of Silicate Glasses?: *Sheldon Wiederhorn*[1]; Jean-Pierre Guin[1]; [1]National Institute of Standards and Technology

The topography of surfaces formed by subcritical crack growth was investigated using the atomic force microscope. Our objective was to determine how well the "upper" and "lower" surfaces matched after being formed by a crack moving at a slow velocity. Specifically, were features left in the fracture surfaces of silicate glasses that would indicate cavity formation during the fracture process? Crack growth experiments were performed on silica, or soda-lime-silicate glass, in air and water over a velocity range of 10-10 m/s to 10-2 m/s. Silica glass surfaces "matched" normal to the fracture surfaces to better than 0.3 nm for the entire range of experimental variables. Soda-lime-silicate glass, surfaces "matched" to an accuracy of 0.5 nm to 0.8 nm normal to the fracture surface, depending on crack velocity. Within these limits, no evidence for cavitation was found in either glass.

2:50 PM Invited
Piezoluminescence Phenomenon on Thin ZnS:Metal Film for Smart Structure Applications: *Shuki Yeshurun*[1]; Yarden Weber[1]; Daniel Schweitzer[1]; Jo Amar[1]; Erez Hasman[2]; Haim Gurgov[2]; Hedva Bar[1]; [1]RAFAEL; [2]Technion

The phenomenon of light emission from crystals due to mechanical stimulation was known to science for centuries. This phenomenon is known as Piezoluminescence (PL). The main expectation from the PL materials is to be used for production of self-diagnostic materials and smart structures that will dynamically visualize the stress distribution by light emission. This paper gives a short theoretical background of the phenomenon and mainly presents the influence of different fabrication processes on light emission. The materials which was used in the impact experiment include ZnS:Mn deposited on glass slides and mixture of ZnS:Mn with epoxy. Characteristics of light emission such as intensity and time response to impact stimulation were examined. As for the impact intensity, the experiments show a clear relationship between the impact amplitude and the emission intensity.

3:15 PM Break

3:30 PM Invited
The Physical and Mechanical Properties of Nickel - SiC Metal Matrix Composites: *Pnina Ari-Gur*[1]; Seyed Mirmiran[2]; [1]Western Michigan University; [2]DaimlerChrysler Autogesellschaft

Nickel-matrix composites of ceramic nano particles present unique properties that offer potential applications in aerospace, automotive and electronics industries. Electrocodeposition is the least expensive fabrication method. The properties of these composite coatings depend on the electrodeposition parameters, the ceramic nanoparticles concentration in the bath solution; properties of the ceramic nanoparticles (material, size, shape, surface roughness, surface charge), properties of the substrate and the interfaces. These coatings are examined via different tools, including microhardness, MTS, AFM, SEM, TEM and XRD. The optimum properties of these composites are obtained when the ceramic nanoparticles are uniformly monodispersed in the matrix with the smallest obtainable grain size. The influence of these factors on the mechanical and physical properties of such composite coatings was studied. Properties like grain size, crystallographic texture, residual stresses and wear resistance and their relationship to processing parameters, ceramic particle size and concentration were determined and interpreted.

3:55 PM Invited
Fracture Initiation at Interfaces: *Placid Rodriguez*[1]; Vaidehi Ganesan[2]; [1]Indian Institute of Technology; [2]Indira Gandhi Centre for Atomic Research

The paper examines fracture initiation at interfaces. The nucleation of a cavity or a crack at an interface results from decohesion at the interface or fracture of a hard and brittle phase across the interface. The most common cause leading to such a decohesion or fracture is either the stress concentration due to the pile -up of dislocations at the interface or a sudden coalescence of dislocations at the head of a pile-up. Impingement by a persistent slip band, intersecting micro-shear bands and intersecting twins also lead to fracture initiation. Sudden unpinning of dislocations leading to localized shear bands is another damage mechanism. All situations lowering grain boundary strength promote intergranular cracking. There are also instances of enviornmet-assisted cracking mechanisms. The various mechanisms of fracture initiation at interfaces are discussed with specific examples from commercial and model alloy systems under both monotonic and cyclic loading conditions.

4:20 PM
Structural and Magnetic Characterization of $Li_{0.5}Fe_{2.5-X}Mn_XO_4$ Spinel Produced by Combustion Synthesis: *Prabeer Barpanda*[1]; Shantanu Behera[2]; Japes Bera[3]; Swadesh K. Pratihar[3]; Santanu Bhattacharya[3]; Rakesh Kumar Sinha[4]; [1]Rutgers University; [2]Lehigh University; [3]National Institute of Technology; [4]Tata Refractories Limited

Lithium ferrites are key materials to replace yttrium iron garnate (YIG) for applications like microwave devices and memory core. It is due to their low cost combined with high curie temperature, saturation magnetization and hysteresis properties. In the reported work, $Li_{0.5}Fe_{2.5-x}Mn_xO_4$ ($0 < x < 1$) spinel samples were prepared by a novel combustion synthesis routes taking metal nitrates as oxidizer and citric acid as fuel. The resulting product was annealed at temperature 300~900°C and the structural evolution was investigated using XRD and Raman spectroscopy. The ordered (α) and disordered (β) spinel phase was quantified using Rietveld analysis and Raman spectra. The structural ordering with temperature was observed using TEM of spinel powder samples. Additionally, the magnetic properties of manganese substituted lithium iron ferrites were measured using vibrating sample magnetometer and Mossbauer spectroscopy. The increasing manganese substitution was marked to enhance the magnetisaton and reduce the curie temperature.

4:35 PM Concluding Comments Wayne D. Kaplan

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

Wednesday PM
March 15, 2006
Room: 207A
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Subodh K. Das, Secat Inc; Gyan Jha, ARCO Aluminum Inc

2:00 PM
Aging Alloy 7085 Mold Block: Part I, Two-Step Aging: *James T. Staley*[1]; [1]North Carolina State University

Aerospace quality 7085 forgings are aged by a multi-step 72 hour aging practice. This treatment was deemed to be too expensive for forged block used for polymer molds, so Alcoa's Cleveland Works asked that we develop a shorter aging treatment. We first determined T_v (GP zones nucleate below this temperature regardless of vacancy content) and T_{cT} (GP zones are unable to nucleate above this temperature). Using these data, we selected a pre-aging temperature of 260°F and explored effects of time at this temperature prior to aging at the standard aging temperature 320°F. We found that 3 hours at this temperature provided a size distribution of GP zones that was stable at 320 F. Alcoa had determined previously that 17 hours at 320°F provided the strength and corrosion characyeristics required. We recommended that they precede this treatment by a pre-age of 3 hours at 260°F.

2:25 PM
Aging Alloy 7085 Mold Block: Part II, Continuous Non-Isothermal Aging: *James T. Staley*[1]; [1]NC State University

The acceptance of alloy 7085 by the polymer molding industry led to increased demands on the aging furnaces at Alcoa's Cleveland Works, so they asked us to develop an even shorter aging practice. We explored the feasibility of combining the stage that nucleates GP zones with the stage that transforms them into η' and η precipitates. We determined the heating rate that allowed GP zones to grow continuously at a rate sufficient to prevent reversion as the temperature increased. We also determined that the kinetics of overaging during continuous aging could be predicted by integrating effects of isothermal overaging. We determined that a linear heating rate of 25 F/hr. produced target strength in 11.25 hours and that heating rate need not be linear. Depending on the size of the load and the characteristics of the furnace, target properties can be obtained by setting the furnace air temperature above 360 F.

2:50 PM
Future Challenges for Aluminum Packaging Alloys: *Gyan Jha*[1]; Weimin Yin[2]; Randall Bowers[2]; [1]ARCO Aluminum Inc; [2]Secat Inc

The Aluminum packaging industry needs to have cost effective high performance alloys for a sustainable future. The key stakeholders at all parts of the supply chain need to work together to develop new alloys that are competitive in today's economic environment. The alloys used for new packaging need to be expanded beyond the current available selection. The opportunities and framework to create value for the supplier, customer and consumer will be discussed.

3:15 PM
Microstructure and Texture Evolution during Drawing and Ironing Rigid Container Sheet: *Gyan Jha*[1]; Weimin Yin[2]; Randall Bowers[2]; [1]ARCO Aluminum Inc; [2]Secat Inc

The structural evolution during D and I process has been studied in AA3xxx aluminum alloys. The complex changes of microstructure and texture occur in the process of drawing and ironing because of aggressive deformation and the heat generated in the process. The microstructure at different stages of the process is characterized in comparison with the as-received can sheet regarding particles, texture and dislocations. The insoluble particles and dispersoids have been determined using Scanning Electron Microscopy equipped with Energy Dispersive Spectroscopy. Omnimet Imaging Analysis System attached to Optical Microscopy is employed to measure the size and distribution of the particles and dispersoids. The micro-hardness and formability has also been evaluated at each stage and related to the evolution of texture and microstructure. A fundamental understanding of the alloy behavior during processing will help support the development of future alloys.

3:40 PM Invited
The Influence of Cold Working on Crystallographic Texture Following Solutionization and Quenching in Ti-6Al-4V Wire: *Thomas R. Bieler*[1]; Liang Zeng[2]; [1]Michigan State University; [2]Alcoa Fastening System

The evolution of microstructure and crystallographic texture of the alpha phase in titanium alloy were examined before and after the solutionization process. The Ti-6Al-4V wire was worked by extrusion, solution heat-treated and water quenched, then aged. Optical and transmission electron microscopes, as well as serial sectioning X-ray diffraction and texture were used to provide information about solutionization and variant selection kinetics and texture homogeneity. Following solutionization and quenching, the primary <10-10> fiber texture decreased at the expense of an emerging secondary fiber texture with <0001> basal plane normal aligned with the longitudinal axis. This 90º mis-oriented component increased in volume fraction with increasing solutionization temperature and time, due to a higher beta phase volume fraction. The relationship between the variant selections of alpha phase, and the effects of dislocations are discussed in the context of the known physical metallurgy of titanium and other alloys.

4:05 PM Break

4:15 PM
Solidification and Stress Modeling of DC Casting of 5xxx Aluminum Alloys: *Zhengdong Long*[1]; Qingyou Han[2]; Shridas Ningileri[3]; Subodh Das[3]; [1]University of Kentucky; [2]Oak Ridge National Laboratory; [3]Secat, Inc.

The DC casting is the predominant casting process for producing ingot for aluminum sheet. An integrated 3D Direct Chill (DC) casting model was used to simulate the heat transfer, fluid flow, solidification, and thermal stress during casting. The ingot surface temperature dependent heat transfer coefficient was determined by temperature measurements, which were performed in an industrial casting facility. The effects of cooling water flow rate, air gaps caused by mold and bottom block design were also coupled into the model. The heat flux, temperature distribution, solid fraction, and thermal gradients during the casting were analyzed. The stress evolution was compared at various locations and correlated with physical phenomena associated with the casting process.

4:40 PM
Friction Stir Processing of AA5052: Basil Darras[1]; *Marwan K. Khraisheh*[1]; [1]University of Kentucky

Recently, Friction Stir Processing has emerged as a promising new tool that can produce ultrafine and homogenized structures in sheet metals. Friction Stir Processing is a solid state processing technique that uses a rapidly rotating non-consumable high strength tool steel pin that extends from a cylindrical shoulder. The rotating pin is forced with a predetermined load into the workpiece and moved along the desired direction. Frictional heating is produced from the rubbing of the rotating shoulder with the workpiece, while the rotating pin deforms and stirs the locally heated material. It is a hot working process in which a large amount of deformation is imparted to the sheet. FS processed zone is characterized by dynamic recrystallization which results in grain refinement. In this work, the effects of various process parameters on the resulting microstructure are discussed.

The Rohatgi Honorary Symposium on Solidification Processing of Metal Matrix Composites: Advanced Applications 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

Wednesday PM Room: 207B
March 15, 2006 Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Nikhilesh Chawla, Arizona State University; B. C. Pai, Regional Research Laboratory, CSIR; Rahul R. Maharsia, Louisiana State University

2:00 PM Invited
Metal Matrix Composites–Commercial Status and Insights: *Daniel B. Miracle*[1]; [1]U.S. Air Force

Over the past four decades, metal matrix composites (MMCs) have been transformed from topic of scientific and intellectual interest to a material of broad technological significance. The worldwide MMC markets in 1999 accounted for 2500 metric tons valued at over $100M. Important MMC applications in the ground transportation, thermal management, aerospace, industrial and recreation industries have been enabled by functional properties that include high structural efficiency, excellent wear resistance, and attractive thermal and electrical characteristics. A suite of challenging technical issues has been overcome, including processing, material design and development, and characterization and control of interface properties. Research, development and transition efforts that led to the successful insertion of MMCs will be described, including insights gained into strategies for successful technology transition in the post-Cold War era. A forward look at the motivating factors and candidate approaches for the next generation of MMCs will be provided.

2:25 PM Invited
Low-Cost Cast Metal Matrix Composites for Ground-Based Vehicles: *Darrell R. Herling*[1]; [1]Pacific Northwest National Laboratory

Metal matrix composites (MMC) have found applications in many industries, from aerospace to automotive, sporting-goods and electronics packaging. Many applications have been for military components, where high performance materials are necessary to meet vigorous property challenges. Aluminum and magnesium MMC are attractive due to high specific stiffness and enhanced strength. In the case of most discontinuous reinforced aluminums, wear resistance of these materials is also increased significantly. Regardless of these benefits, high materials costs relative to conventional alloys have limited widespread application of this material family in the ground transportation industry. If the cost associated with production and shape forming methods of MMC can be reduced, this could enable widespread use of particle reinforced composite materials in the automotive and truck manufacturing industries. This paper will highlight the results of a program sponsored by the Department of Energy and USCAR that is focused on developing low-cost options for MMC components.

2:50 PM
Application of Al-B4C Metal Matrix Composites in the Nuclear Industry for Neutron Absorber Materials: *X. Grant Chen*[1]; [1]Alcan Inc.

For dry storage and transportation of nuclear fuel in the nuclear industry, the most commonly used neutron absorber are boron-containing metallic materials, in which the element boron possesses a high cross-section for absorbing thermal neutrons. Al-B4C metal matrix composites, recently developed by Alcan, are particularly suitable for manufacturing high quality neutron absorber components due to the flexibility of adding various boron concentrations, the lightweight, and superior thermal conductivity and mechanical properties. In this paper, a series of Al-B4C MMCs with different Al-matrix and B4C loads are presented for both structural and non-structural applications. The process routes for incorporating B4C into metal and associated downstream fabrication are outlined. The composites can be cast, extruded or rolled to almost any desired size and shape. The microstructure, thermal conductivity and mechanical properties of some Al-B4C products are described. Results of neutron absorptivity based on neutron transmission tests for typical Al-B4C products are presented.

3:15 PM Invited
Hybrid Aluminum Matrix Composites for Brake Applications: *Jason S. H. Lo*[1]; [1]CANMET, Natural Resources Canada

Traditionally, automotive brake rotors for both light and heavy vehicles are made with cast iron. Besides having economical advantage, cast iron rotors provide many disadvantages. Disadvantages due mainly to their weight are reduction in fuel efficiency; increase in green house gas emission; and increase in noise, vibration and hardness. Commercial aluminum composite materials with property overcoming most of the drawbacks in cast iron brake rotors has been developed and employed in light vehicles. However, such commercial composite materials suffer a major drawback of poor elevated temperature property. In this paper, the results of the work in developing a novel hybrid composite material specifically for the brake application are described. This material consists of a SiC reinforcement and a modifier to enhance the elevated temperature property of the aluminum matrix alloy. Both the fabrication technology, microstructure, mechanical and physical properties of the hybrid composite being evaluated, are reported.

3:40 PM Invited
High Energy X-Ray Diffraction Measurements and Imaging of Superconducting Mg/MgB2 Composites under Compressive Loading: *Marcus Young*[1]; John D. DeFouw[1]; Jonathan D. Almer[2]; Kamel Fezzaa[2]; Wah-Keat Lee[2]; Dean R. Haeffner[2]; David C. Dunand[1]; [1]Northwestern University; [2]Argonne National Laboratory

In this study, 140 µm diameter boron fibers were pressure infiltrated with liquid Mg metal and the Mg/B composite was subsequently heat treated at 950°C for 2.5 hours to convert B fibers into MgB2 fibers by reaction with liquid Mg. The resulting Mg-73 vol% MgB2 fiber composite was loaded in uniaxial compression, and volume-averaged lattice strains in the Mg and MgB2 phases were measured in-situ at various constant stresses by synchrotron X-ray diffraction up to 500 MPa. Load transfer was observed to occur from the Mg matrix to the MgB2 fibers. Spatially resolved measurements showed variations in load transfer at different positions within the composite for the elastic, plastic, and damage deformation regimes. Using phase-enhanced x-ray imaging, the extent of damage within the composites was also observed.

4:05 PM Break

4:20 PM Invited
Non Destructive Characterization of Metal Matrix and Advanced Composites—A Review: *Phani Surya Kiran Mylavarapu*[1]; Eyassu Woldesenbet[1]; [1]Louisiana State University

Metal matrix composites (MMC) offer wide variety of property advantages over conventional metals and alloys. MMC's can be classified into four types depending upon the type of reinforcement as MMC's reinforced with particles(PMC), MMC's reinforced with short whiskers or fibers (SFRM), MMC's reinforced with continuous fibers(CFM) and MMC's reinforced with mono filaments (MFRM). With the increasing usage of MMC's and advanced composites as realistic candidates for engineering components, quality control and failure monitoring during service in these materials have gained importance. This paper reviews the present non destructive characterization techniques used in the industry for characterizing MMC's and other advanced composites with the challenges behind them.

4:45 PM Invited
Applications of Solidification Processed Discontinuously Reinforced Aluminium Alloy Composites: *Satyabrata Das*[1]; Dehi Pada Mondal[1]; N. Ramakrishnan[1]; [1]Regional Research Laboratory

Aluminium Composites are engineered combination of aluminium alloy and reinforcing phase capable of providing tailored properties. AACs combine the favorable properties of aluminium and ceramic leading to improved physical, mechanical and tribological properties. Regional Research Laboratory, Bhopal, India has developed liquid metallurgy route to

synthesis Al composites. Developed composites are characterized in terms of microstructure, deformation behaviour, tribological properties etc. Efforts are being made to design and develop Al composite prototype components. Such components are field trialed in various sectors and encouraging results are obtained and thus found their way for commercialization. Considerable efforts have been made in transferring the aluminium composite technology developed in the laboratory to automobile industries. Prototype components such as brake drum for automobile are developed. It is found that AMMC brake drums are lighter by 60% and better braking efficiency as compared to existing one. Composite Apex Insert for mining industries is also developed.

5:10 PM Break

5:15 PM Panel Discussion
Future Directions in Micro and Nano Metal Matrix Composites: Moderated by Dr. Warren Hunt, Aluminum Consultants Group Inc

Titanium Alloys for High Temperature Applications - A Symposium Dedicated to the Memory of Dr. Martin Blackburn: Titanium Based Intermetallic Alloys for High Temperature Applications - Gamma

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

Wednesday PM
March 15, 2006
Room: 201
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Xinhua Wu, University of Birmingham; Wayne Voice, Rolls-Royce plc

2:00 PM Invited
Development of New Structural Alloys Based on β+γ TiAl: *Young-Won Kim*[1]; Dennis M. Dimiduk[2]; Christopher F. Woodward[2]; [1]UES Inc; [2]U.S. Air Force

Ongoing research is investigating a titanium-based high-temperature structural alloy system, called beta-gamma Ti, consisting of three phases (γ-TiAl + β-Ti + α2-Ti3Al). Gamma alloys (based on γ-TiAl and α2-Ti3Al)exhibit remarkable creep and oxidation resistance useful up to 900°C. Yet, gamma alloys have not been implemented for aerospace applications, primary due to their low low-temperature ductility, unconventional processing requirements and poor machinability. Experiments were initiated to develop robust beta-gamma alloys, and preliminary experiments show that such alloy compositions exist within the phase fields which are of three phases (γ + β + α) at low temperatures (<1100°C) and of two or three phases (β + one or two others) at higher temperatures. Determination of such phase fields is underway through phase/composition analyses of a few selected compositions and ThermoCal calculations and modeling. This accelerated development process will be discussed, along with the initial results of forging behavior, machinability and properties of a selected alloy.

2:30 PM
Synthesis and High Temperature Mechanical Properties of Two Ultrafine Grained Gamma-TiAl Based Alloys: Ti-47Al and Ti-45Al-5.5(Cr,Nb,B,Ta): Hongbao Yu[1]; *Deliang Zhang*[1]; Peng Cao[1]; Yuyong Chen[2]; Brian Gabbitas[1]; [1]University of Waikato; [2]Harbin Institute of Technology

In our research on titanium based materials, high quality bulk nanostructured (grain sizes <100nm) and ultrafine grained (100nm<grain sizes<1μm) TiAl intermetallic based alloys are produced using a novel powder metallurgy route which combines high energy mechanical milling of mixtures of elemental powders to produce nanostructured powders and consolidation of the powders using various thermomechanical processes including hot isostatic pressing, equal channel angular pressing and forward extrusion. The fine microstructure renders the alloys with improved formability which is critical for the applications of TiAl based alloys. To determine the capacity of the ultrafine grained TiAl based alloys for high temperature applications, their high temperature mechanical properties such as strength and ductility are studies. This paper will report findings of a study which has been focused on the synthesis and high temperature mechanical properties of two alloys: a binary alloy Ti-47Al and a complex alloy Ti-45Al-2Cr-2Nb-1B-0.5Ta (in at%).

3:00 PM Break

3:30 PM
The Effects of W Additions on the Microstructural and Mechanical Properties of Ti-48Al-2Nb-2Cr Alloys: *Dongyi Seo*[1]; Scott Bulmer[2]; H. Saari[2]; P. Au[1]; P. Patnaik[1]; [1]National Research Council of Canada; [2]Carleton University

Ti-48Al+2Nb+2Cr powders, with 0%, 0.5% and 1%W (at%), were consolidated by hot isostatic pressing (HIP). A heat treatment, which consisted of slow cooling from 1400°C in the α region to 1280°C in the α+γ region at a rate of 20°C/min, followed by air-cooling, was developed to produce fully lamellar microstructures with narrow lamellar spacing. This procedure also produced a microstructure that was free from Widmanstätten or massive γ and β particles. To stabilize the microstructure, the HIP'ed and heat-treated samples were subsequently aged in the $\alpha_2+\gamma$ region for various durations. The resulting microstructures were analyzed in terms of grain size, precipitate size, and interlamellar spacing. Hardness tests were also conducted to assess the strength of the heat-treated materials, and the results were analyzed with respect to W additions. The results from this study will be used to identify heat treatment conditions to optimize the creep resistance of these materials.

4:00 PM
Microstructure and Mechanical Properties of Rolled Sheet and Forged Block Gamma-Titanium Aluminides: *Russell J. Foon*[1]; Julius De Rojas[1]; Sun Hyung Kim[1]; Nicole Sporer[1]; Richard Clark[2]; John Ogren[1]; Omar Es-Said[1]; Kyle Mori[1]; Elizabeth Villalobos[1]; Hamid Garmestani[3]; Dongsheng Li[3]; Gopal Das[4]; [1]Loyola Marymount University; [2]College of the Canyons; [3]Georgia Institute of Technology; [4]Pratt & Whitney

γ-based inter-metallic titanium aluminides are ideal for high temperature structural applications in gas turbine engines and automotive industries. γ-TiAl based alloys have attractive properties such as low density, high modulus, high temperature strength, oxidation resistance, burn resistance, and the potential to replace heavier superalloys for the temperature range 550-850°C. Titanium aluminide samples processed from sheet and forged samples with varying compositions of Cr, Nb, Ta, B, C were heat treated above and below the alpha transus temperature. Microhardness, optical microscopy, texture (preferred crystallographic orientation) and tensile tests were carried out to characterize the material.

Ultrafine Grained Materials - Fourth International Symposium: High Temperature and Physical 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

Wednesday PM
March 15, 2006

Room: 217D
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Terence G. Langdon, University of Southern California; Yuri S. Estrin, Technical University Clausthal; Farghalli A. Mohamed, University of California; Taku Sakai, UEC Tokyo

2:00 PM Invited
Creep Behavior of an Al-7034 Alloy Processed by ECAP: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California

Processing by equal-channel angular pressing (ECAP) at a temperature of 473 K, an ultrafine grain size of ~0.3 μm was obtained in a commercial Al 7034 alloy and superplastic elongations of =1000% were achieved at high strain rates (=10^{-2} s^{-1}) when testing at 673 K. The creep behavior of the as-pressed alloy was investigated at temperatures of 473, 573 and 673 K and the relationship between strain rate and flow stress was examined. The stress exponent was calculated to determine the dominating deformation process and emphasis was placed on the strain rate range where superplastic elongations were achieved. The creep behavior of the alloy in the as-received condition was also examined for comparison.

2:20 PM Invited
Effect of Nano-Scale Dispersion Particles on Thermal Stability and Creep in Bulk Cryomilled Ultrafine Grained 5083 Al: Farghalli A. Mohamed[1]; *Manish Chauhan*[1]; Indranil Roy[1]; [1]University of California

The preparation of Ultra fine grained (UFG) 5083 Al using gas atomization followed by cryomilling leads to introducing oxides (resulting from the breakup of oxide layers that are formed around metal particles during gas atomization), and carbides, nitrides, and other impurities during cryomilling. The results of research in progress show that the presence of these nano- scale dispersion particles affects the behavior of the alloy in several ways. First, the particles enhance thermal stability by suppressing grain growth. Second, they serve as strong obstacles to the movement of dislocations, a process that provides a possible source for a threshold stress during superplastic flow. Finally, they act as sites for cavity nucleation, leading to a loss in ductility.

2:40 PM
A Quantitative Investigation of Cavity Development in an Aluminum Alloy Processed by Equal-Channel Angular Pressing: *Megumi Kawasaki*[1]; Cheng Xu[1]; Terence G. Langdon[1]; [1]University of Southern California

A commercial spray-cast aluminum 7034 alloy was processed by equal-channel angular pressing (ECAP) to produce an ultrafine grain size of ~0.3 μm. Microstructural examination showed the alloy contains $MgZn_2$ and Al_3Zr precipitates which restrict grain growth at elevated temperatures. Tensile tests were conducted on both as-received and as-pressed specimens at a temperature of 673 K and the results showed the occurrence of high superplastic elongations in the as-pressed material. There was also evidence for the development of extensive cavitation during testing. This paper describes a quantitative examination of cavity development in specimens pulled to failure in both the as-received and the as-pressed conditions. Measurements were taken to record critical parameters including the area of each cavity, the orientations of the long axes with respect to the tensile axis and the roundness coefficient which is a measure of the shape of each individual cavity.

2:55 PM
Compressive Creep in an Al-3%Mg-0.2%Sc Alloy Processed by Equal-Channel Angular Pressing: Jiri Dvorak[1]; Milan Svoboda[1]; Zenji Horita[2]; *Vaclav Sklenicka*[1]; [1]Academy of Sciences of the Czech Republic; [2]Kyushu University

Creep tests in compression on an Al-3wt%Mg-0.2wt%Sc alloy after processing through equal-channel angular pressing (ECAP) were conducted at temperature 473 K and stress in the range 15-50 MPa. The microstructure of a ternary alloy was examined by transmission electron microscopy. The present results were compared with the results of our earlier work on compressive creep of pure ECAP aluminium with the same imposed strain and the ECAP processing route. The results demonstrate that the creep strength of an Al-Mg-Sc alloy is significantly improved compared to that of pure aluminium. The higher strength observed for the ternary alloy in the high-stress region (>25 MPa) results from the synergism of solid-solution strengthening and precipitate strengthening due to Al_3Sc nanoscale precipitates. The presence of these precipitates dramatically increases the creep resistance in the low-stress region through a threshold stress for creep.

3:10 PM Invited
Creep Regularities and Mechanisms of Ultrafine Grained Ti-6Al-4V Alloy Produced by Severe Plastic Deformation: *Yury R. Kolobov*[1]; Galina P. Grabovetskaya[2]; Yuntian T. Zhu[3]; Konstanin V. Ivanov[1]; Olga V. Zabudchenko[4]; [1]Belgorod State University; [2]Institute of Strength Physics and Materials Science SB RAS; [3]Los Alamos National Laboratory; [4]Tomsk State University

A comparative analysis of plastic deformation development regularities at creep of ultrafine grained (0.2-2 μm) Ti-6Al-4V alloy processed by severe plastic deformation (SPD) and its fine grained (3-10 μm) counterpart was carried out. It is shown that the formation of ultrafine grain structure of two-phase Ti-6Al-4V alloy by SPD leads to temperature range shear of superplastic properties manifestation to lower temperatures. The influence of structural state of Ti-6Al-4V alloy on the development of grain boundary sliding (GBS) at creep has been studied. The apparent activation energy of creep was lower for the ultrafine grained Ti-6Al-4V than for its fine grained counterpart. The role of GBS and diffusion mass transfer in the development of plastic deformation at creep is being analyzed.

3:30 PM
Rapid Nanostructuring of Cementite during the Deformation of Pearlitic Steel: *Ian MacLaren*[1]; Yulia Ivanisenko[2]; Xavier Sauvage[3]; Ruslan Z. Valiev[4]; Hans-Jörg Fecht[2]; [1]University of Glasgow; [2]Forschungszentrum Karlsruhe; [3]University of Rouen; [4]Ufa State Aviation Technical University

It is now well known that the cementite component of pearlitic steel tends to be destroyed and dissolved on deformation, although many details of the process remain unclear. The present study focuses on the early stages of high-pressure torsion deformation of pearlitic steel using TEM and 3D atom probe as the primary characterisation methods. It is found that the ferrite lamellae deform much as expected by slip of lattice dislocations, resulting in the normal cell formation that is common to many ductile materials. In contrast to this, even at this early stage of the deformation, the cementite lamellae are reduced to a nanocrystalline structure. The compositional changes in the cementite as a consequence of the deformation are analysed and the processes that lead to this rapid nanostructuring are discussed.

3:45 PM
Stress Corrosion Cracks Propagation in Ultra-Fine Grain Copper Fabricated by an Equal Channel Angular Pressing: *Hiroyuki Miyamoto*[1]; Takuro Mimaki[1]; Satoshi Hashimoto[2]; Alexei Vinogradov[2]; [1]Doshisha University; [2]Osaka City University

The present study aims to assess the susceptibility of ultra-fine grain (UFG) copper produced by Equal channel angular pressing (ECAP) to stress corrosion cracking (SCC) by a ASTM plane strain fracture test with a constant load in an aqueous 1M NaNO2 solution under a constant applied potential of 100mV(Ag-AgCl). The effect of equilibrium and unequilibrium states of grain boundaries on the susceptibility of SCC has

been focused. Two copper billets of commercial purity were pressed through a ECA-die until eight passes via the so-called route B in order to obtain an UFG structure. One of them was subsequently annealed at 473K for 40 seconds in order to relieve internal stress. It was found that the crack propagation rate of ultra-fine grain copper decreased by a flash annealing. The decrease of SCC sensitivity is attributed to the change of grain boundary structures to equilibrium state during the flash annealing.

4:00 PM Break

4:10 PM Invited

Properties of Ultrafine Grained Conductors: *Ke Han*[1]; Jun Lu[1]; [1]National High Magnetic Field Laboratory

Codeformation is used to fabricate large quantities of metal matrix composite conductors which possess unique combination of mechanical and physical properties. The properties of those materials are related to the microstructure. A number of Cu matrix conductors have strength levels close to their theoretical strength because the size of the grains reaches nanometer scales. However, the electrical resistance is also increased because of the refined microstructure. The application of these materials requires detailed consideration of the mechanisms of strengthening and electron transport which are operative in materials with ultra fine scale microstructures. In addition, consideration must be given to the fatigue endurance of those composites. Low temperature superconductors are also the composite. The critical current of Nb3Sn superconductors is related to the mechanical strain to which the materials are exposed. This presentation will address the correlation between the properties (both mechanical and transport) and microstructure of those conductors.

4:30 PM

Texture Gradient in fcc Metals Deformed by ECAP as a Function of Stacking Fault Energy: *Werner Skrotzki*[1]; Burghardt Kloeden[1]; Nils Scheerbaum[1]; Carl-Georg Oertel[1]; Satyam Suwas[2]; Laszlo S. Tóth[3]; [1]TU Dresden; [2]RWTH Aachen; [3]University Metz

Different fcc metals (Al, Cu, Ni, Ag) have been deformed by equal channel angular pressing (ECAP) up to 3 passes using route A. The texture with respect to position in the deformed billet has been measured with high-energy synchrotron radiation. It is characterized by texture components typical for simple shear in the intersection plane of the square-shaped 90° bent channel. Intensities of the texture components as well as deviations from their ideal shear positions vary from the top to the bottom of the billet and with the number of passes. The change of the intensity of texture components and the texture gradient within the metals investigated will be discussed. Special emphasis will be put on the influence of stacking fault energy on texture formation during ECAP of fcc metals.

4:45 PM

Non-Equilibrium Processing Routes for Ultrafine Grained or Nanostructured Materials: *Gerhard H. Wilde*[1]; Nancy Boucharat[1]; Guru Prasad Dinda[1]; Harald Rösner[1]; Ruslan Valiev[2]; [1]Forschungszentrum Karlsruhe GmbH; [2]Ufa State Aviation Technical University

The sequential combination of different processing routes that drive a material to a different extent, - with different rates - and by different means from thermodynamic equilibrium present new and attractive processing opportunities to obtain bulk nanocrystalline or massive ultrafine grained materials that are widely unexplored. More specifically, processing routes based on rapid quenching or plastic deformation have been combined here such that the initial state is continuously energized and successively driven farer away from thermodynamic equilibrium. Here, different deformation methods with largely different strain and pressure levels have been applied on rapidly quenched metallic glasses, but also on elemental sheet samples. Both processing routes result in a nanocrystalline microstructure with remarkable properties, especially with respect to strength and hardness. The basic underlying mechanisms that lead to ultrafine grained or nanocrystalline microstructures are discussed and the current state of nanostructure control is highlighted by selected examples.

5:00 PM Question and Answer Period, Award Ceremony and Free Style Discussion: Moderated by Terence G. Langdon, University of Southern California

Wechsler Symposium on Radiation Effects, Deformation and Phase Transformations in Metals and Ceramics: Shape Memory Alloys

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

Wednesday PM
March 15, 2006

Room: 208
Location: Henry B. Gonzalez Convention Ctr.

Session Chairs: Louis K. Mansur, Oak Ridge National Laboratory; K. Linga Murty, North Carolina State University

2:00 PM

The Effect of Severe Plastic Deformation on the Recoverable Phase Transformation in High Temperature Shape Memory Alloys: *Benat Kockar*[1]; Ibrahim Karaman[1]; Jae-Il Kim[1]; [1]Texas A&M University

NiTi alloys are the most important shape memory alloys due to their superior mechanical and functional properties, however their use is restricted below 100°C. In order to extend their utility in high temperature applications, higher martensitic transformation temperatures than 100°C, lower temperature hysteresis, and better cyclic reversibility are required. The transformation temperatures of NiTi alloys can be increased by the addition of Hf and Pd, however, these additions lead to the degradation of thermal cyclic response under stress due to the decrease in critical stress for slip. In this study, severe plastic deformation via equal channel angular extrusion (ECAE) was used to increase the critical stress for slip by grain refinement down to nanometer range and by designing specific textures in NiTi and NiTi(Pd,Hf) alloys. We will present methods of engineering microstructure and texture using ECAE and resulting thermomechanical response including temperature hysteresis and enhanced cyclic reversibility.

2:20 PM

On the High Strain-Rate Response of Ni-Rich NiTi Shape Memory Alloy as a Function of Aging Temperature: *Raghavendra Adharapurapu*[1]; Kenneth Scott Vecchio[1]; [1]University of California

The influence of aging on the high strain-rate response of Ni-rich (60 wt%) NiTi shape memory alloy was studied. Aging in Ni-rich NiTi alloys leads to metastable Ti_3Ni_4 precipitates, which promotes the occurrence of R-phase. These precipitates lead to precipitation hardening, and aid the overall shape memory effect and strengthening of B2 matrix. Since precipitates in this alloy affect the shape memory properties and also the transformation temperatures, initial investigations were focused on precipitation using metallographic techniques and a TTT diagram was developed. In order to ascertain their behavior under impact loading conditions for various defense applications, the dynamic response of these alloys was studied as a function of aging and compared with the quasi-static results. A split-Hopkinson bar was utilized for high-strain rate compression tests using a pulse-shaper technique. Preliminary results indicate that a wide range in strength (between 950MPa – 2500MPa) is achievable in these materials.

2:40 PM

Dynamic Response of NiTi Shape Memory Alloys: Influence of Temperature and Strain-Rate on Tension-Compression Asymmetry: *Raghavendra Adharapurapu*[1]; Kenneth Scott Vecchio[1]; FengChun Jiang[1]; [1]University of California

Polycrystalline NiTi shape memory alloys (SMA) are known to exhibit asymmetry in tension-compression behavior at room temperature and low strain rates. In order to fully explore the extent of this asymmetry, the compressive and tensile behavior of NiTi shape memory alloy has been

examined as a function of temperature (-196ºC to 400ºC) at 10^{-3}/s and 1200/s strain rates. A Hopkinson tensile bar and a split-Hopkinson compression bar with a pulse shaper technique were utilized for dynamic tension and compression tests, respectively. The attendant asymmetry between tension and compression behavior was captured in the variation of stress-plateau and the critical stress (as obtained by 0.2% strain offset) with temperature. The critical stress exhibited a three-stage characteristic, with critical stress being higher in compression than in tension. These findings have important implications in the application of SMA towards defense and others applications where understanding the dynamic response of the SMA is essential.

3:00 PM

Neutron Diffraction Studies of Deformation in NiTiFe Shape Memory Alloys at 90 K: *Rajan Vaidyanathan*[1]; [1]University of Central Florida

NiTiFe shape memory alloys can undergo transformations between cubic, rhombohedral and monoclinic phases at low temperatures. The low hysteresis associated with the rhombohedral or R phase transformation, coupled with superior fatigue properties, makes them candidates for actuator applications at low temperatures. This talk reports on neutron diffraction measurements from NiTiFe shape memory alloys during mechanical loading at low temperatures, with the objective of probing deformation in the R phase. For this purpose, a low temperature loading capability for in situ neutron diffraction measurements was implemented on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory. The in situ diffraction measurements, during loading at 90 K, observed twinning in the R phase prior to a reversible martensitic transformation to the monoclinic phase at higher stresses. This work was supported by grants from NASA (NAG3-2751) and NSF (CAREER DMR-0239512).

3:20 PM Break

3:40 PM

Few Guidelines to Increase Actuation Stress in NiMnGa Magnetic Shape Memory Alloys (MSMAs): *Burak Basaran*[1]; Haluk Ersin Karaca[1]; Ibrahim Karaman[1]; Yuriy I. Chumlyakov[2]; Hans J. Maier[3]; [1]Texas A&M University; [2]Siberian Physical-Technical Institute; [3]University of Paderborn

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 in quest for identifying physical and microstructural parameters critical in increasing the magnetic actuation stress. The present paper will discuss few guidelines to increase the actuation stress considering the coupled effects of magnetocrystalline anisotropy energy, phase transformation temperatures and Curie temperature. Few specific results will be presented in which one order 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 instead of field induced martensite reorientation which has been the only mechanism reported to be responsible for magnetic shape memory effect.

4:00 PM

An In Situ Study of Martensitic Transformation in Shape Memory Alloys Using PEEM: *Mingdong Cai*[1]; Stephen C. Langford[1]; J. Thomas Dickinson[1]; Gang Xiong[2]; Timothy C. Droubay[2]; Alan G. Joly[2]; Wayne P. Hess[2]; [1]Washington State University; [2]Pacific Northwest National Laboratory

The thermally-induced martensitic transformation in a polycrystalline CuZnAl and NiTi thin film shape memory alloy (SMA) was probed using photoemission electron microscopy (PEEM). Ultra-violet photoelectron spectroscopy (UPS) measurements indicate that the apparent surface work function changes reversibly during transformation, presumably due to the contrasting electronic structures of the martensitic and austenitic phases. *In situ* PEEM images provide information on the spatial distribution of these phases and the microstructural evolution during transformation. The evolution of the photoemission intensities obtained from PEEM images during transformation can provide quantitative information on fractional percentages of austenite and martensite phases as the transformation proceeds. PEEM offers considerable potential for improving our understanding of martensitic transformations in shape memory alloys in real time.

4:20 PM

New Cobalt Based Ferromagnetic Shape Memory Alloys (SMAs): *Haluk Ersin Karaca*[1]; Ibrahim Karaman[1]; Yuriy Chumlyakov[2]; Hans Maier[3]; [1]Texas A&M University; [2]Siberian Physical-Technical Institute; [3]University of Paderborn

Ferromagnetic SMAs have attracted increasing interest because of the ability to obtain one order of magnitude higher recoverable magnetic field induced strain than other active materials. A recently discovered ferromagnetic shape memory CoNiAl and CoNiGa alloys have promising shape memory characteristics for conventional and magnetic shape memory applications. We have conducted extensive studies to capture several aspects of the shape memory behavior of both alloys in single and polycrystalline forms. It has been demonstrated that these alloys have low pseudoelastic stress hysteresis even at temperatures higher than 200°C, high strength for dislocation slip, large recoverable strain levels (10%), large pseudoelastic temperature window (>250°C), low stress for martensite reorientation, and stable response to cyclic deformation. They also demonstrate strong orientation dependence and tension/compression asymmetry in shape memory characteristics. Selected experimental findings on single and polycrystals that summarize these findings will be presented and the challenges will be addressed.

4:40 PM

Deformation Mechanisms in U-Nb Shape Memory Alloys: *Robert D. Field*[1]; Donald Brown[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory

Alloys with compositions in the vicinity of U-14at%Nb display the shape memory effect (SME), with the maximum recoverable strain associated with a martensitic transformation from the high temperature (bcc) gamma phase to a monoclinically distorted version of the room temperature alpha-U phase, designated as alpha". The martensitic structure, deformation mechanisms, and texture development during uniaxial straining in the SME regime have been explained in terms of a correspondence variant model which describes the martensitic twin relationships derived from gamma/alpha" orientation relationships. This model has been extended to the post-SME regime by considering the texture developed during SME deformation as a starting point for subsequent plasticity. Recent progress will be presented, with particular emphasis on TEM observations of deformation twinning mechanisms and crystallographic texture evolution as determined by in-situ neutron diffraction experiments.

5:00 PM

Deformation Behavior of U-6 Wt% Nb: *Carl M. Cady*[1]; George T. Gray[1]; Donald Brown[1]; Robert D. Field[1]; Philip K. Tubesing[1]; Denise R. Korzekwa[1]; Ellen K. Cerreta[1]; [1]Los Alamos National Laboratory

The shape memory effect is well documented in uranium-niobium alloys. The uranium-6 wt. % niobium alloy was investigated to determine the deformation behavior at different strains and loading conditions. Information will also be presented on the effect of strain rate, temperature, and loading orientation on the deformation behavior. Texture and mechanical experiments were conducted in compression, tension and shear to various strain levels to try to determine the active deformation mechanisms. This paper will present evidence as to the deformation mechanisms that occur, when they are activated, and when they saturate.