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Missouri	The Science of Alloys for the 21st Century: Hume Rothery I	The Science of Alloys for the 21st Century: Hume Rothery II	The Science of Alloys for the 21st Century: Hume Rothery III	The Science of Alloys for the 21st Century: Hume-Rothery IV	Interfacial Dislocation: J Van der Merwe Anniversary I	Interfacial Dislocation: J Van der Merwe Anniversary II	Interfacial Dislocation: J Van der Merwe Anniversary III
Meramac	Crystallography & Diffraction Tutorials I, II, & III	Electron Backscatter Diffraction I	Electron Backscatter Diffraction II	Electron Backscatter Diffraction III	Electron Backscatter Diffraction IV	Microcharact-erization & Microtexture I	Microcharact-erization & Microtexture II
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Field	Advances in Interconnect & Packaging Materials I	Advances in Interconnect & Packaging Materials II	Advances in Interconnect & Packaging Materials III	Metastability in Bulk & Thin Film Materials I	Metastability in Bulk & Thin Film Materials II	GA: Ferrous Metallurgy	
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Technical Program

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2000 TMS Fall Technical Program

+ Indicates Student Paper

Crystallography and Diffraction Tutorials

Sponsored by: ASM-MSCTS Structures Committee
Presented by: Professor Marc DeGraef, Professor David Laughlin, Professor Mike McHenry; Carnegie Mellon University, Pittsburgh, PA USA

Monday AM Room: Meramac
October 9, 2000 Location: Regal Riverfront Hotel

8:00 AM
Basic Crystallography: *M. De Graef*

In the first part of this three-part tutorial we will introduce the concept of real space crystal lattices. We will define the 7 crystal systems and 14 Bravais lattices, and show how one can perform simple geometrical computations, using a formalism which is independent of the shape of the unit cell. We will in particular focus on the computation of interatomic distances and bond angles. Then we will define reciprocal space, a convenient tool for the description of diffraction phenomena. We will consider the basic geometrical characteristics of reciprocal space through a real-time computer simulation. Finally, we will conclude this part with an introduction to symmetry; we will define symmetry elements (translation, rotation, mirror, inversion, screw axis, glide plane, ...) and briefly cover the concepts of point groups and space groups.

9:15 AM Break

9:25 AM
Important Crystal Structures and Structure Types: *M. E. McHenry*

The second part of this tutorial will make use of the concepts introduced in the first part and cover in detail how a crystal structure can be described in a succinct way. Then we will begin with the basic structure types (bcc, fcc, hcp, ...), and derive a whole series of structures from the basic types; we will use lattice and interstitial substitutions to define technologically important crystal structures for both alloys and oxides. After a brief description of the space filling principles we will conclude this part with an overview of Frank-Kasper and related structures and quasi-crystals.

10:40 AM Break

10:50 AM
Basic Diffraction and the Use of the International Crystallographic Tables: *D. E. Laughlin*

The third and last part of the tutorial will cover the fundamentals of diffraction: Bragg's Law, which deals with the geometry of the diffraction process, and the Structure Factor, which forms the basis for intensity calculations. Examples will be drawn from the structure types introduced in the previous part. We will introduce the basic experimental configurations for recording x-ray diffraction patterns, and illustrate neutron and electron diffraction. We will demonstrate how screw axes and glide planes affect the diffraction patterns. We will use the diamond structure as an example. Then we will introduce the International Crystallographic tables and show how to use the special conditions. Again the diamond structure will be used, since its diffraction patterns have missing reflections due to the Bravais lattice, the presence of screw axes and glide planes, and the special positions of the atoms.

Advances in Interconnect & Packaging Materials - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Thin Films & Interfaces Committee
Program Organizers: Frank G. Shi, University of California-Irvine, Department of Chemical Engineering & Materials Science, Irvine, CA 92697 USA; Bin Zhao, Conexant System Inc., Advanced Process Technology, Newport Beach, CA 92660 USA

Monday AM Room: Field
October 9, 2000 Location: Regal Riverfront Hotel

Session Chairs: Frank Shi, University of California, Dept. of Chem. and Biochem. and Matls. Sci., Irvine, CA 92697 USA; Bin Zhao, Conexant Systems, Newport Beach, CA 92660 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Material Challenges and Opportunities for Monolithic Three-Dimensional Integration in Microelectronics: *Rafael Reif*[1]; A. Fan[1]; A. Rahman[1]; [1]Massachusetts Institute of Technology, Elect. Eng. and Comp. Sci. Depts., 60 Vassar St., Bldg. 39-527, Cambridge, MA 02139 USA

Three-dimensional (3-D) integration represents a new paradigm shift in process architecture and system architecture design. In 3-D integration, multiple device layers, each with its own set of multilevel interconnects, can be bonded together to form a multi-strata structure. Electrical interconnections between device layers can be created using vertical metal vias. The existence of both horizontal and vertical metal lines promises significant reductions in interconnect delay, and gives the designer full utilization of the third dimension in device placement and routing. Moreover, by fabricating each device layer using a different technology (e.g., logic, memory, analog, MEMS, etc.), the monolithic integration of a high-performance "system-on-a-chip" appears feasible. This paper addresses the fabrication challenges and material issues involved in the development of 3-D integration.

9:25 AM Invited
Structure and Property Characterization of Low-k Dielectric Porous Thin Films: Eric K. Lin[1]; Hae-jeong Lee[1]; Howard Wang[1]; *Wen-li Wu*[1]; [1]NIST, 100 Bureau Dr., Stop 8541, Gaithersburg, MD 20899-8541 USA

A novel methodology based on a combination of small angle neutron scattering, high-resolution x-ray reflectivity and ion scattering has been developed to measure the on-wafer structural properties of low k thin films. The average pore size, pore connectivity, film thickness, matrix material density, coefficient of thermal expansion and moisture uptake can be measured and the results have been delivered to SEMATECH to aid industry in the selection of candidate materials and processes to be used in next generation integrated circuits.

9:50 AM Invited
Low Dielectric Constant Materials: *H. Treichel*[1]; [1]Lam Research Corporation, 4650 Cushing Pkwy., Fremont, CA 94538-6401 USA

The more advanced an integrated circuit becomes, the more stringent are the demands for certain properties of a dielectric or insulating material. In addition, it is essential that the layer maintain its specific electrical, physical and chemical properties after incorporation in the device structure and during subsequent processing. Due to temperature budget constraints and the accelerated decrease of feature sizes below 0.25 μm one can no longer rely on traditional choices but has to search for alternatives, both for low and high permittivity replacements. In this article we survey currently used low dielectric constant materials and future trends for microelectronic applications.

10:15 AM Break

10:25 AM Invited
Barrier Layers for Cu ULSI Metallization: *Yosi Shacham-Diamand*[1]; [1]Tel-Aviv University, Dept. of Phys. Electro., Rm. 234, Fac. of Eng., Ramat-Aviv, Tel-Aviv 69978 Israel

The essential features of ULSI technology are characterized with rapid scaling of the critical dimensions, where the state of the art today is 0.18 µm in production. Other important features are the increasing complexity of the interconnect network that link the transistors and the introduction of copper as the main metallization material. Copper is being introduced today for ULSI metallization. It offers higher electro-migration resistance and better conductivity than aluminum. The embedded copper lines (e.g. Damascene) process flow variations are fully planar and are most suitable for ULSI. The Dual-Damascene process has significantly less process steps than conventional aluminum metallization, hence it offers a significant cost reduction and possible better yield and productivity. However, copper can contaminate the silicon substrate, introduces generation-recombination levels within the silicon energy-gap, and increases junctions' leakage and threshold voltage instabilities. To solve this problem special barrier layers that are integrated around the wires isolate the copper. The barrier layers thickness should be as thin as possible to maximize the copper advantage in conductivity and reliability over aluminum. However, reducing the barrier thickness may compromise its integrity. For example, as the chips critical dimensions are getting to the 0.1 µm range, the barrier layers thickness should be ~5-10 nm. In the talk we will review the "ideal" barrier properties and discuss the possibility to prepare such material. We will also describe the status of the most successful barrier layers today. Finally, the field of electroplated barrier layers, which is investigated at Tel-Aviv University, will be described.

10:50 AM Invited
Material and Process Challenges in 100-nm Interconnects Module Technology and Beyond: *Takayuki Ohba*[1]; [1]Fujitsu Limited, Ulsi Dev. Div., 1500 Tado-cho, Kuwana-Gun, Mie 511-0192 Japan

In order to minimize manufacturing cost related to the wiring process and to realize fabrication of ULSI at 100-nm node and beyond, several Cu/Low-k wiring technologies have been proposed. The evidential criteria for choosing the most probable one are the physical and material properties of the process and materials (e.g., step-coverage and resistivity) and the requirements for manufacturing (e.g., process complexity, reliability, throughput, ramp-up speed, and total cost). The development of module processes (e.g. processing from Low-k dielectrics to metal CMP) with proven equipment and materials is an appropriate approach and has a high potential in overcoming those difficulties. In this paper, a predictive module structure of 100-nm node ULSI circuits and Cu/Low-k multilevel metallization module will be discussed.

11:15 AM Invited
Room Temperature Recrystallization and Texture Development in Electroplated and Sputter-Deposited Copper Films: *M. E. Gross*[1]; [1]Silicon Electronics Research Laboratory, Bell Labs, Lucent Technologies, 600 Mountain Ave., Room 1D-343, Murray Hill, NJ 07974 USA

Cu interconnects for advanced integrated circuits are today being fabricated by electroplating Cu into dual damascene structures consisting of trenches and vias etched in SiO2. A thin (<1,000A) sputtered Cu film over a refractory metal-based diffusion barrier serves as the cathode for plating. The texture of the barrier layer as well as the topography of the damascene structure both influence the texture of the electroplated (EP) Cu. A new sidewall texture component was identified in damascene Cu samples. Following plating, the electroplated Cu undergoes recrystallization at room temperature that advantageously produces large grains for improved electromigration resistance. The low stacking fault energy of Cu and a high concentration of incoherent twin boundaries in the electroplated film are implicated in facilitating the room temperature recrystallization. Interestingly, the stored energy in the EP film can, at room temperature, induce grain growth in sputtered Cu layers twice as thick. Comparing the recrystallization rates and crystallographic textures of different combinations of sputtered/EP Cu bilayers provides additional insights into the mechanism of recrystallization. In this talk, I will review recent results on various aspects of the texture, microstructure, and room temperature recrystallization of electroplated Cu in blanket and damascene substrates, with a consideration of the underlying mechanisms.

11:40 AM
The Material and Electrical Properties of Electoless Ag(W) Thin Film: *Yosi Shacham-Diamand*[1]; [1]Tel-Aviv University, Dept. of Phys. Electro., Room 234, Ramat-Aviv, Tel-Aviv 69978 Israel

In this study we present the results of electroless deposition of silver (Ag) and silver tungsten (Ag(W)) layers on Si, intended for application in microelectronics and MEMS technology. Silver has excellent resistivity but its thin film properties and its vulnerability to corrosion may cause a problem. In this work we present a novel Ag(W) type of layers that has improved thin film properties, such as resistivity and surface roughness, and can serve as both barrier layer and capping layer for corrosion protection of the Ag thin films. The thin film composition was studied as a function of the deposition parameters. We found the presence of tungsten, up to 3.1 a/o, and oxygen, up to 8 a/o in addition to the silver atoms. We also studied the thin film morphology using Atomic Force Microscopy (AFM) and Scanning Tunnel Microscopy (STM) imaging of the surface after each process step. Finally, we discuss the possible mechanisms for the deposition of Ag(W).

General Abstracts: Non-Ferrous and Powder Metallurgy

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Monday AM — Room: Jefferson A
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chair: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

8:30 AM
Mechanical Properties of an Induction Slag Melted Ti-6Al-4V Alloy: *Karol K. Schrems*[1]; Alan D. Hartman[1]; Jeffrey A. Hawk[1]; [1]U.S. Department of Energy, Matls. Conserv. Div., Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

The Albany Research Center (ARC) of the Department of Energy is investigating a means to form useful wrought products by direct and continuous casting of titanium bars using cold-wall induction melting. An investigation to compare the mechanical properties of a commercial Ti-6Al-4V ingot with a cold-wall induction slag melted Ti-6Al-4V ingot was performed to determine if the slag additions used to improve the surface finish adversely affect ingot quality, i.e., microstructure and mechanical properties. Room temperature tensile, Charpy, and fatigue tests were performed. The experimental alloy was found to have slightly higher strengths, and slightly lower ductilities, impact energies, and fatigue life than the commercial Ti-6Al-4V ingot, although all values were found to be similar to typical handbook properties. Slag additions do not appear to detrimentally affect the mechanical properties of Ti-6Al-4V ingots prepared in a cold-wall induction slag furnace.

8:50 AM
The Effects of Cold Rolling Strain and Annealing Schedule on the Volume Fraction of Texture Components of AA3105 DC Hot Band and AA3015 SC Hot Band: *Jiantao Liu*[1]; Yansheng Liu[1]; Wenchang Liu[1]; Xiyu Wen[1]; James G. Morris[1]; [1]University of Kentucky, Dept. of Chem. and Matls. Eng., 175 Anderson Hall, Light Mets. Rsch. Labs., Lexington, KY 40506-0046 USA

The minimization of earing behavior depends on the balance between deformation texture components and recrystallization texture components. In this paper, both AA 3105 DC hot band and AA 3015 SC hot band are used for cold rolling and annealing. The effects of strain, annealing temperature and annealing time on the volume fraction of the deformation texture components and the recrystallization texture components are investigated using an orthogonal design method. The phenomenon of different softening texture components evolution between DC material and SC material is discussed. Based on the analysis of the experimental results, the optimum cold rolling and annealing schedules for minimizing earing behavior are proposed for both materials.

9:10 AM
Microstructural Study of the Isothermal Martensitic Transformation in Cast and Wrought Co-Based Alloys: *Armando J. Saldivar*[1]; [1]University of Wisconsin-Milwaukee, Matls. Dept., 3200 N. Cramer St., Milwaukee, WI 53211 USA

The Co-based alloys have been widely used for biomedical applications, in this work it was found that the cast and wrought versions of these alloys exhibit important differences in the response to the isothermal aging heat treatment. This comparative metallographic study by optical and scanning electronic microscopy shows how the segregation of alloying elements has a remarkable effect on the microstructural features and kinetics of the isothermal martensitic transformation. The variations in morphology and

the kinetics of transformation are discussed in terms of alloying element segregation and stacking fault energy.

9:30 AM +
The Potential for Paint-Bake Aging in Enhanced-Ductility Al Alloys I-Artificial Aging Studies: *Jennifer A. Hamilton*[1]; Dustin C. Balderach[1]; Eric M. Taleff[1]; [1]The University of Texas at Austin, Texas Matls. Instit., C 2200, Austin, TX 78712-1063 USA

Enhanced tensile ductility (>100%) has been produced in a variety of Al-Mg alloys under warm-forming conditions as a result of solute-drag creep. Ternary alloying additions, such as Zn, enable these materials to be age hardened without adversely affecting the enhanced ductilities obtained at elevated temperatures. The potential for age hardening during the paint-bake cycle in automobile manufacturing has been investigated in these alloys through a series of artificial aging experiments. Various Al-Mg-X ternary alloys were artificially aged at a characteristic paint-bake temperature then mechanically tested. Testing consisted of Vickers hardness for all conditions and tensile tests for a few selected conditions. A significant paint-bake response with high strength was found.

9:50 AM +
The Potential for Paint-Bake Aging in Enhanced-Ductility Al Alloys II-Natural Aging Effects: *Dustin C. Balderach*[1]; Jennifer A. Hamilton[1]; Eric M. Taleff[1]; [1]The University of Texas at Austin, Texas Materials Institute, Austin, TX 78712-1063 USA

Several Al-Mg-X alloys have been studied which exhibit enhanced tensile ductility under warm-forming conditions and can be subsequently age hardened. The potential paint-bake hardenability of these alloys has been further studied through combinations of artificial and natural aging treatments. The effects of natural aging treatments on Vickers hardness, both before and after artificial aging treatments, are investigated. Extended natural aging times of many months were applied to investigate the stability of several heat-treated conditions. Aging times and temperatures relevant to the automobile paint-bake cycle and possible automobile service conditions were used for determining test conditions. In addition to Vickers hardness tests, tensile tests were performed on selected samples. A good potential for paint-bake hardening was found for several alloys.

10:10 AM
Application of Recycled Metal Powder for Machine Parts Production: *Alexey V. Sverdlin*[1]; Tatiana L. Loumacheva[2]; Alexey A. Melnikov[3]; Kristina Sarbaeva[3]; Anna Kirillova[3]; [1]Bradley University, Indust. and Manufact. Eng. and Tech., 1501 W. Bradley Ave., Peoria, IL 61625 USA; [2]Bradley University, Dept. of Mktg., Peoria, IL 61625 USA; [3]Samara State Aerospace University, Dept. of Matls. Sci., Moskovskoe shosse 34a, Samara 443086 Russia

The recovery of waste material after grinding, stamping and forging operations is being developed as a potential source of iron powder for powder metallurgy (PM). Such slurry is not processed by metallurgical conversion and causes environmental conditions to worsen. After a method of recovering slurry was developed, research found that the recovered powder is suitable for use in structural and other high-density parts. A team of researchers from the USA and Russia has developed the basis for the manufacturing process, produced components from recovered powder and is now looking to refine the process. Therefore, the project stands to benefit the environment as well as the PM community.

10:30 AM
Modeling and Simulation of Polymer Removal from PIM Compact by Thermal Debinding: *Shengjie Ying*[1]; Y. C. Lam[1]; S. C.M. Yu[1]; K. C. Tam[1]; [1]Nanyang Technological University, Sch. of Mech. and Prod. Eng., 50 Nanyang Ave., Singapore 639798 Singapore

Powder injection molding is an important net-shape manufacturing process. Thermal debinding is a common methodology for the final removal of residual polymer from a PIM compact. This process is an intricate combination of evaporation, liquid and gas migration, pyrosis of polymer, and heat transfer in porous media. Numerical simulation of the process based on an integrated mathematical model for mass and heat transfer in porous media is proposed. The mechanisms of mass transport, i.e., liquid flow, gas flow, vapor diffusion and convection, as well as the phase transitions of polymer, and their interactions, are included in the model. The macroscopic partial differential equations are formulated by volume averaging of the microscopic conservation laws, and are solved numerically. Polymer residue, pressure and temperature distributions are predicted. The importance of the various mass transfer mechanisms is evaluated. The effects of key mass transfer parameters on thermal debinding are discussed.

10:50 AM
Determination of Sintering Activation Energy for Powder Injection Molded 316L Stainless Steel Parts: *Zhen Yun Liu*[1]; Ngiap Hiang Loh[1]; Khiam Aik Khor[1]; Shu Beng Tor[1]; [1]Nanyang Technological University, Sch. of Mech. and Product. Eng., 50 Nanyang Ave., 639798 Singapore

The sintering of powder injection molded 316L stainless steel has been investigated at constant heating rate of 2.5°C to 15°C/min. Linear shrinkage was recorded in-situ by a dilatometric test machine. The activation energies for relative density ranging from 0.6 to 0.8 were calculated. To achieve the same density, the sintering temperature should be shifted to higher with increasing heating rate during heating up. The results showed that the activation energies for the intermediate stage sintering of powder injection molded 316L stainless steel were in the range of 272.2 to 368.8kJ/mol for different densities. At low densities, the sintering is dominated by volume diffusion mechanism. As more grain boundaries formed at higher density, grain boundary diffusion will also contribute to the densification of the compact. The possibility of the high activation energies for powder injection molded compacts was explained by particle surface impurities after debinding.

11:10 AM
Hot Pressing of Ti-Al-Si Alloy Powder: *Yuri M. Lytvynenko*[1]; Leonid D. Kulak[1]; [1]Institute for Problems of Materials Science, 3 Krzhizhanovsky St., Kyiv 03142 Ukraine

New titanium based materials with natural strengthening via the precipitates of hardening phases at the eutectic crystallization can be manufactured by both usual casting technology and granulating technology with following compacting. The method of hot pressing the grains allows to obtain the parts with more advanced level of the mechanical properties as compare with cast ones because the cast genesis defects are removed. The spherical powders of hypoeutectic and eutectic Ti-Al-Si alloy with different contents of alloying elements were compacted. The pressing was performed at the temperatures of 1000, 1250, 1350, 1450 and 1500°C during exposures from 10 to 15 min. and the pressures of 25 and 33 MPa. The microstructure and mechanical properties at the temperatures of 700, 800, 900 and 1000°C were investigated.

11:30 AM
Effect of Radiation Heating on the Surface Structure and Properties of SiC-Si3N4 Materials: Oleg N. Grigoriev[1]; Svetlana V. Kucheriava[1]; *Yuri M. Lytvynenko*[1]; [1]Institute for Problems of Materials Science, 3 Krzhizhanovsky Str., Kyiv 03142 Ukraine

It is known that the oxide layers on a surface of ceramic materials improve both a strength and corrosion resistance. A forming of the oxide layers structure as a result of heating by the concentrated solar radiation as well as an influence the layers on the physical and chemical properties of SiC-Si3N4 ceramics is interested. The materials have stability to oxidation up to a level of heat flow 6000 kW/m2. Under the radiation heating effect a high-temperature erosion takes place. The general behavior of the influence of the local heat topology on the ceramic surface is revealed. The area of effect of the solar heat spot can be sectioned into zones having temperature fields which resulting to formation of frames sharply distinguished from the outer radius of the light spot to the center of irradiated area. The structure of these zones varies with both the entered components in SiC-Si3N4 matrix and a mode of irradiation of the materials. A corrosion resistance, strength and hardness of the ceramics are investigated.

Materials Issues in Nuclear Waste Management: Waste Form Development and Characterization - I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Nuclear Materials Committee
Program Organizers: Thad M. Adams, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Robert Sindelar, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Patrick R. Taylor, University of Idaho, Department of Metals & Mining Engineering, Moscow, ID 83843-3024 USA

Monday AM — Room: Clark
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chair: Robert L. Sindelar, Westinghouse Savannah River Company, Aiken, SC 29808 USA

8:30 AM Opening Remarks: Dr. Thad M. Adams

8:35 AM Invited
Radiation Effects in Nuclear Waste Materials and Their Consequences: *L. M. Wang*[1]; R. C. Ewing[1]; [1]University of Michigan, Dept. of Nuclear Eng. and Radio. Sci., 1301 Beal Ave., Ann Arbor, MI 48109-2104 USA

Radioactive decay and the associated radiation fields in the repository for radioactive waste over geologic time scale may lead to important

changes of the near-field materials (waste forms and back-fill) that may affect the release rate of radionuclides. For example, radiation-induced amorphization may drastically increase the leaching rate of the ceramic waste form. Extensive studies on radiation effects im complex ceramics have been carried out to evaluate the long-term radiation stability of potential nuclear waste materials (i.e., waste form, back-fill and materials for chemical separation). The materials studied include various compositions of structure-types which include: olivine, spinel, zircon, monazite, mica and zeolite. The microstructural changes induced by ion and electron beam irradiations have been followed by in situ transmission electron microscopy (TEM), and the results were compared with that from natural analogues which have suffered alpha decay damage during the geological times. Most complex ceramic materials were found to be susceptible to radiation-induced amorphization. In general, critical amorphization dose increases with the irradiation temperature at a rate determined by the competition between kinetics of amorphization and recrystallization. Mica and zeolites are extremely sensitive to ionizing irradiation. For zeolites, amorphization dose decreased at elevated temperatures due to the thermal instability of the material. However, as indicated by ion exchange experiments, amorphization of zeolites may reduce the release rate of radionuclides trapped in the structure. Other effects studied include radiation-induced chemical disordering, decomposition, nanocrystallization as well as cavity formation.

9:10 AM

Developing Separations Technologies and Waste Forms for the Accelerator Transmutation of Waste (ATW): *James J. Laidler*[1]; *Sean M. McDeavitt*[1]; Mark A. Williamson[1]; James L. Willit[1]; Daniel P. Abraham[1]; Karthick V. Gourishankar[1]; [1]Argonne National Laboratory, CMT-205, 9700 S. Cass Ave., Argonne, IL 60439 USA

The Accelerator Transmutation of Waste (ATW) concept is being developed in a six-year, science-based technology development program at Los Alamos National Laboratory, Argonne National Laboratory, and other contributing labs, universities, and companies. The basic premise of the ATW concept is that long-lived fission products and transuranic (TRU) elements in spent nuclear fuel, such as technetium and plutonium, may be destroyed in an accelerator-driven subcritical system to eliminate long-term toxicities and facilitate the storage of high-level radioactive waste. An integral part of this program is the development of chemical separations processes to extract long-lived radionuclides from commercial spent nuclear fuel for transmutation and then to recycle the fuel from the accelerator-driven system to maximize the TRU destruction. The processes under development include an aqueous process to remove uranium from oxide spent fuel, a pyrochemical process to prepare the concentrated TRU oxides for fuel fabrication, and a post-irradiation pyrochemical process to recycle the remnant TRU species back into the ATW accelerator-driven system. These chemical processes involve radioactive waste streams, and so waste form materials are also being defined. This paper summarizes the state of the baseline and optional process flowsheets and the materials issues being examined for the ATW waste form definition.

9:30 AM

The Development, Processing and Qualification of Ceramic and Metal Waste Forms from the Electrometallurgical Treatment of Spent Nuclear Fuel: *Sean M. McDeavitt*[1]; Kenneth M. Goff[2]; Daniel P. Abraham[1]; William L. Ebert[1]; Mark C. Hash[1]; Stephen G. Johnson[2]; Dennis D. Keiser[2]; Dusan Lexa[1]; Thomas P. O'Holleran[2]; Michael K. Richmann[1]; Michael F. Simpson[2]; Brian R. Westphal[2]; [1]Argonne National Laboratory, CMT-205, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Argonne National Laboratory West, NT-AW 774, P.O. Box 2528, Idaho Falls, ID 83403-2528 USA

Electrometallurgical treatment was successfully demonstrated by Argonne National Laboratory to be a viable technology for the conversion of sodium-bonded metallic nuclear fuel into stable waste form materials and uranium metal. In this process, uranium from the spent fuel is electrorefined in a molten chloride salt electrolyte. The radioactive waste constituents that must be immobilized after electrorefining include salt-borne chlorides in the electrolyte and metallic fuel pin constituents that are electrochemically noble in the electrorefiner. The processing methods and behavior characteristics for ceramic and metal waste forms were developed and are continuing to be qualified for high level waste production and repository disposal. The salt-borne wastes are immobilized into a zeolite 4A; the salt-loaded zeolite is mixed with glass; and the composite mixture is consolidated at 850°C. The zeolite transforms to sodalite in situ and the final ceramic waste form is a composite of sodalite, glass, and minor oxide phases. The metal waste form is a stainless steel-15 wt% zirconium alloy that is produced by alloying the remnant metals at 1600°C. Both waste form materials have been subjected to rigorous testing programs to characterize their behavior in preparation for the formal waste form qualification process.

9:50 AM

Vitrification with a Plasma Assisted Cyclone Reactor: *Patrick R. Taylor*[1]; [1]University of Idaho, Plasma Process. Lab., P.O. Box 443024, Moscow, ID 83844-3024 USA

A non-transferred arc thermal plasma assisted cyclone reactor has been designed, built and operated to evlauate it's use in vitrification of wastes. Simulated waste (liquid, slurry or fine particulate) is injected near the plasma flame, where nearly instantaneous dehydration and calcination reactions happen. The closed top cyclone flow sends the solid particulates to the wall, where they are captured by a thin film of molten glasss that is injected as a powder at a position lower in the reactor. The molten glass is then collected in a crucible. Example results are given for three types of waste streams.

10:10 AM

Development of a Metalic Spent Fuel Form: *T. M. Adams*[1]; H. B. Peacock[1]; D. L. Fisher[1]; A. J. Duncan[1]; N. C. Iyer[1]; [1]Westinghouse Savannah River Company, Savannah River Tech. Ctr., Bldg. 773-41A, Rm. 151, Aiken, SC 29808 USA

Abstract text unavailable.

10:30 AM Break

10:40 AM

The Effect of Temperature and Composition on Spinel Equilibrium in High-Level Waste Glass: *Benjamin K. Wilson*[1]; Brandon R. Wilson[1]; Trevor J. Plaisted[1]; Pavel Hrma[1]; [1]Pacific Northwest National Laboratory, K6-24, P.O. Box 999, Richland, WA 99352 USA

The equilibrium concentration (C_o) of spinel was measured in 16 high-level waste (HLW) glasses as a function of temperature (T). The glasses were formulated by a one-at-a-time composition variation of Al_20_3, Cr_20_3, Fe_20_3, Li_20, MgO, Na_20, and NiO. The ideal solution relationship between C_o and T was used to fit data. The coefficients of this relationship and the temperature at which C_o= constant were expressed as functions of glass composition using first-order approximation. All varied components had an effect on liquidus temperature (T_L), but only NiO and Fe_20_3 had a significant impact on spinel concentration below T_L. Since the HLW glass is constrained to form a limited or zero fraction of spinel in the melter, these relationships can be used for optimized formulation of HLW glasses.

11:00 AM

Characterization of Iron Phosphate Waste Forms: *Patrick R. Taylor*[1]; Wanqing Huang[1]; [1]University of Idaho, Plasma Process. Lab., P.O. Box 443024, Moscow, ID 83844-3024 USA

Iron phosphate glasses are promising host matrices for vitrifying actinide rich nuclear wastes materials because of its high solubility of actinide oxides, high chemical durability, and relatively low melting temperatures. An iron phosphate waste form was developed at the University of Missouri-Rolla. The structure and property of the waste form were well characterized by modern materials techniques. However, to scale the process and advance it to commercial maturability, it is necessary to understand the processing parameters, such as the partitioning of waste elements, the viscosity and density of iron phosphate melt at high temperatures. Viscosity of 40Fe2O3-60P2O5 mol% iron phosphate melts at 1000-1200°C was measured by the rotating-spindle method. It was discovered that the fluid has non-Newtonian behavior. Experiments were performed to study rheological properties following the Standard Test methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer (ASTM D 2196-86). The partitioning behavior of waste elements in the iron phosphate glass was studied by melting in high temperature furnace and ICP analysis. Density of the iron phosphate melt at 1200°C was measured by the improved Archimedean method.

11:20 AM

Characterization of N-Reactor Fuel Element SFEC5, 4378 at Argonne National Laboratory: *T. S. Bray*[1]; M. Goldberg[1]; R. V. Strain[1]; H. Tsai[1]; J. Y. Park[1]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

Twelve specimens from N-Reactor Mark IV-E fuel element SFEC5,4378 were characterized at Argonne National Laboratory's Alpha-Gamma Hot Cell Facility in support of the National Spent Nuclear Fuel Program for the Release Rate Testing Program. The samples originated approximately two inches from the element end and approximately one inch above a cladding failure. The characterization included as-received examination, metallography, scanning electron microscope analysis, and electron microprobe analysis. Visual examinations revealed the presence of cracking (radial, axial, and circumferential) in the fuel matrix. Metallography and scanning electron microscope examination results revealed apparent interaction along some cracked regions, fuel matrix inclusions and oxidation, tight fuel-clad interfaces, and phase discontinuities. Inclusions in the fuel matrix include carbides, oxides, and possibly hydrides. One sample revealed the presence of a crack propagating into the cladding. The microprobe exami-

nation evaluating the distribution of nuclides at the fuel-clad interface did not reveal quantifiable fuel-cladding interactions.

11:40 AM
Characterization of the Glass Being Produced at Savannah River Site for Immobilization and Disposal of Its Radioactive High Level Waste: *N. E. Bibler*[1]; T. L. Fellinger[1]; J. W. Ray[1]; [1]Westinghouse Savannah River Company, Savannah River Tech. Ctr. and Defense Waste Process. Facility, Aiken, SC 29808 USA

The radioactive high level waste (HLW) sludge slurries at Savannah River Site (SRS) are currently being processed and immobilized into a borosilicate glass in the Defense Waste Processing Facility (DWPF) at SRS. The glass is prepared at the DWPF by mixing the treated sludge slurry with glass formers, melting the mixture in a Joule heated melter at 1150°C, and then pouring the molten glass into stainless steel canisters for eventual final disposal in a geologic disposal. Currently more than 500,000 gallons of HLW have been immobilized into more than 600 stainless steel canisters for disposal. These canisters are currently being stored in an interim facility at SRS. This paper will discuss the properties of the glass. There are its radioactive and nonradioactive composition, its microstructure based on examination by scanning electron microscopy, and its leachability in water. Evaluation of these properties is based on characterization of actual radioactive samples of glass obtained from the pour stream of the DWPF melter during its operation. Pertinent data on the effects of alpha, gamma, and beta radiation on these properties will also be briefly reviewed and discussed. Finally, how these properties relate to current acceptance criteria for the geologic repository will be discussed.

Rate Processes in Plastic Deformation II: Towards an Unified Theory of Deformation - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA; Thomas R. Bieler, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; Bimal Kad, University of California, AMES Laboratory, La Jolla, CA 92093-0411 USA; Farghalli A. Mohamed, University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697 USA

Monday AM — Room: Lewis
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chairs: Prof. William D. Nix, Stanford University, Dept. of Matls. Sci., Stanford, CA 94305-2205 USA; Prof. Farghalli A. Mohamed, University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA

8:30 AM Opening Remarks

Constitutive Modeling of Materials

8:40 AM Keynote
An Examination of the Constitutive Equation for Elevated Temperature Plasticity: *Amiya K. Mukherjee*[1]; [1]University of California, Chem. Eng. and Matls. Sci. Depts., One Shields Ave., Davis, CA 95616 USA

The constitutive equation that links the stress-strain rate-grain size-temperature relation (Mukherjee-Bird-Dorn, MBD correlation) was presented in 1969 to describe elevated temperature crystalline plasticity. This equation has held up well during the intervening quarter of a century. It has been applied to metals, alloys, intermetallics, ceramics and tectonic systems and it has worked equally well. In the case of particle-dispersed systems as well as metal matrix composites, the introduction of the concept of a threshold stress was a substantial improvement in creep studies. One of the significant applications of the MBD relation has been in superplasticity. The concept of scaling with either temperature or with strain rate, inherent in this relationship, seems to be obeyed as long as the rate controlling mechanism is unchanged. The application of this relation to high strain rate superplasticity and also to low temperature superplasticity will be illustrated. It will be shown that superplasticity of nanocrystalline metals and alloys follow the general trend of the constitutive relation with important differences in the level of stress and strain hardening rates. (Current Grant #NSF-DMR-9903321).

9:10 AM Keynote
Impression Creep and Other Localized Deformation Processes: *James C.M. Li*[1]; [1]University of Rochester, Dept. of Mech. Eng., 500 Wilson Blvd., Rochester, NY 14627 USA

Impression creep is a localized creep test. Unlike conical and pyramidal indenters, a cylindrical indenter with a flat end can produce steady state creep showing a constant penetration velocity under a constant load so the stress and temperature dependences of such velocity can be easily obtained. When the stress dependence is linear, a viscosity can be calculated and in the cases studied, it agrees with other measurements. Both power law and hyperbolic sine law have been observed. Creep studies using this technique are reviewed together with other localized tests such as impression fatigue which is the propagation of a plastic zone under a stress concentration similar to ordinary fatigue in which the stress concentration comes from the tip of a crack. The two fatigue behaviors are similar even though one has a crack and the other has no crack. Some recent work on indentation fatigue of brittle materials is reviewed together with impression recovery and stress relaxation. Work supported by NSF through DMR 9623808 monitored by Dr. Bruce McDonald.

9:40 AM Invited
High-Strain Rate Deformation Mechanisms: Experiments, Modeling, and Verification: *George (Rusty) T. Gray*[1]; [1]Los Alamos National Laboratory, MST-8, MailStop G755, Los Alamos, NM 87545 USA

The deformation substructures observed in many materials subjected to high-strain rate or shock loading are similar to those formed quasi-statically at low temperature. The suppression of thermally-activated dislocation processes in this regime, added to the strain-rate sensitivity of the yield stress exhibited by many materials, can lead to stresses high enough to nucleate and grow deformation twins even in high stacking fault FCC metals. In addition, due to the subsonic restriction on dislocation motion, high rate deformation, in particular shock wave, leads to dislocation generation rates producing enhanced hardening in many materials compared to quasi-static deformation to an equivalent strain. In this talk examples of the deformation substructure evolution and the accompanying mechanical behavior observed in a range of materials at high strain rate will be presented. The process of validating constitutive materials models using Taylor cylinder impact testing is discussed with special emphasis on the influence of texture on plastic yielding and test-sample shape evolution as a function of impact loading conditions and microstructure.

10:00 AM Break

10:15 AM Keynote
Constitutive Analysis in Hot Working: *H. J. McQueen*[1]; N. D. Ryan[1]; [1]Concordia University, Mech. Eng. Dept., 1455 Blvd. Maisonneuve W., Montreal H3G 1M8 Canada

Constitutive equations including an Arrhenius term have been commonly applied to steels with the objective of calculation of rolling and forging forces. The function relating stress and strain rate is usually the hyperbolic sine since the power and exponential laws lose linearity at high and low stresses respectively. In austenitic steels, the equations have been used primarily for the peak stress (strain) associated with dynamic recrystallization (DRX) but also for the critical and steady state stresses (strains) for nucleation and first wave completion of DRX. Since the peak strain is raised by the presence of solutes and fine particles, the stress is raised more than by simple strain hardening increase, thus causing a marked rise in activation energy in alloy steels. In contrast, large carbides, inclusions or segregates, if hard, may lower the peak strain as a result of particle stimulated nucleation. Due to the linear relation between stress and strain at the peak, flow curves can be calculated from the constitutive data with only one additional constant. Peak pass stresses can also be calculated from a sinh constitutive equation determined in multistage torsion simulations of rolling schedules. Comparison is made between C, HSLA, tool and stainless steels and to ferritic steels which usually do not exhibit DRX. Parallels to the effects of impurities and dispersoids on Al alloys are briefly discussed.

10:45 AM Invited
Assessment of Damage and Life Prediction of Austenitic Stainless Steel Under High Temperature Fatigue-Creep Interaction Condition: *Soo Woo Nam*[1]; [1]Korea Advanced Institute of Science and Technology, Depts. of Matls. Sci. and Eng., 373-1 Kusong-dong Yusong-gu, Taejon 305-701 Korea

It has been well known that the high temperature low-cycle fatigue (LCF) life is reduced by the imposed creep deformation due to the "creep-fatigue interaction". LCF test is conducted by strain controlled mode with or without strain hold in tension and/or compression. During the hold, the peak stress is relaxed to give creep damage that is responsible for the reduction of the fatigue life. Depend on the metallurgical structures and the thermodynamic stability of the structure, the time dependent damage mechanisms by creep are found to be different for different materials and test

conditions. Among those high temperature materials, austenitic stainless steels have very clear grain boundary surrounding thermodynamically stable single phase or alpha phase. On the other hand, ferritic steels such as Cr-Mo-V rotar steels do not have clear grain boundary and carbides are dispersed in the thermodynamically unstable matrix of tempered martensite. If fatigue and creep deformations are applied in these alloys damages induced by the deformations will be initiated at the worst unstable structure or having the highest potential energy region. In case of austenitic stainless steel, the interface between matrix and carbides at the grain boundary is the most favorable place where cavities are nucleated and growing under tensile strain hold or due to creep effect. A new creep-fatigue life prediction model in terms of nucleation (due to the plastic deformation by fatigue) and growth (due to the creep deformation during hold time) of grain boundary cavities is introduced. From the life prediction equation of creep-fatigue interaction, a new Creep-Fatigue Damage Function is proposed. This damage function is a combination of the terms such as plastic strain range, test temperature, grain boundary diffusivity and duration of hold time in which the creep deformation is taking place. And this damage function is proven to be generally applicable to the materials in which failure is controlled by the grain boundary cavitational damage. Additionally, using this damage function, all the Coffin-Manson plots at the various levels of tensile hold time, temperature and strain range can be normalized to make one master curve, that is a normalized Coffin-Manson relation. For the above life prediction model, it is found that grain boundary cavity is found to be nucleated at the carbide and the number of cavities are measured to be proportional to the density of the grain boundary carbide. This means that, to extend the creep-fatigue life or to develop better high temperature material, the number of grain boundary carbide has to be reduced. Future investigation will be focused on the subject of alloy development which has less harmful carbide and lower carbide density. For the ferritic steels, because of the unstable microstructure of the matrix, surface cracks are the main damage initiated by the LCF. These surface cracks are growing through inside of grain (most unstable or high energy region) to result transgranular fracture. In this case there have been proposed many models of life prediction, however, no model has the expression of the generalized physical meaning for the crack growth. To derive a unified life prediction model for these ferritic alloys, those variables of strain range, tensile hold time, relaxed stress and test temperature are related to form a generalized equation. Future investigation is to be concentrated on the normalization of the generalized life prediction equation of the rotor steels.

11:05 AM

Factors Contributing to Matrix Strengthening in the Creep Behavior of Aluminum-Based Composites: *Bing Q. Han*[1]; Terence G. Langdon[1]; [1]McGill University, Depts. of Metal. Eng., 3610 University St., W. H. Wong Bldg., Montreal, Quebec H3A 2B2 Canada

There have been several recent investigations of the creep behavior of discontinuously-reinforced aluminum matrix composites with at elevated temperatures. In many cases, the results reveal a difference in the strain rates at the same effective stress between the composites and the matrix materials. This difference has been attributed to the occurrence of load transfer based on the modified shear-lag theory. The creep behavior was analyzed for several aluminum matrix composites and it is shown that load transfer cannot account fully for the difference in the strain rates between the composites and the matrix materials. This paper shows that substructure strengthening also plays an important role in determining the strain rates during creep deformation.

11:25 AM

The Influence of Prior Strain Rate on Stress Relaxation in Solder Alloys: Michael W. Woodmansee[1]; *Richard W. Neu*[1]; [1]Georgia Institute of Technology, The George W. Woodruff Sch. of Mech. Eng., Atlanta, GA 30332-0405 USA

New experiments were performed on several solder alloys (60Sn-40Pb, 96Sn-4Ag, and 96.2Sn-2.5Ag-0.8Cu-0.5Sb) that exhibit Class M behavior to shed light on the influence of the strain rate immediately before a strain hold during which stress relaxation is recorded. In these experiments, stress relaxation is measured immediately after three different applied strain rates (10-3 s-1, 10-4 s-1, and 10-5 s-1) at a homologous temperature between 0.60 and 0.67, depending on the alloy. The response of all three alloys are similar. The higher strain rates during prior deformation result in not only greater total stress relaxation but also a lower relaxed stress. Implications for describing this behavior with phenomenological constitutive models are discussed. However, most advanced unified creep-plasticity phenomenological models do not capture this behavior well. A reason for their failure can be argued based on the role of dislocation substructures. Consequently, a substructure-based deformation model will likely be needed to capture these observed prior strain rate effects.

State of the Art in Cast MMC's: Processing of Metal Matrix Composites - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing and Manufacturing Division, Jt. Composite Materials Committee, Solidification Committee
Program Organizer: Pradeep Rohatgi, University of Wisconsin, Materials Department, Milwaukee, WI 53211 USA

Monday AM — Room: Laclade
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chair: Pradeep Rohatgi, University of Wisconsin, Matls. Dept., Milwaukee, WI 53211 USA;M. Makhlouf, Worchester Polytechnic Institute, Dept. of Mech. Eng., Worchester, MA USA

8:30 AM

Reactive Infiltration of TiO_2 Preforms with Molten Al: *Iulian Gheorghe*[1]; Henry J. Rack[1]; [1]Clemson University, Cer. and Matls. Eng. Depts., 204 Olin Hall, Clemson, SC 29634 USA

The kinetics of the reaction between TiO_2 and molten aluminum was investigated. Pressure-casting technique was employed to infiltrate preforms of TiO_2 whiskers of an average length of 6μm and mean diameter of 0.7μm with molten aluminum, pressures of 1500 psi applied on the melt being sufficient to completely infiltrate the preform. By varying the initial preform temperature from 850°C to 450°C, the temperature of the melt from 850°C to 675°C as well as the post-infiltration cooling speed, large variations in the extent of the reaction between TiO_2 and Al were produced. The range of microstructure extended from fully reacted composites Al_2O_3 reinforced Al+Al_3Ti to non-reacted TiO_2/Al composites. It was found that in the initial stages of the reaction a partial substitution of Ti atoms with Al atoms on Ti_2O_3 lattice occurs. Al_2O_3 is formed upon total substitution of Ti atoms with Al atoms. The released Ti dissolves in the matrix where after reaching the critical concentration forms Al_3Ti. This presentation will summarize the reaction kinetics between molten Al and TiO as well as the effect of processing conditions (i.e., melt temperature, preform temperature and post-infiltration cooling speed) on the final microstructure of the composite. This work is supported by Dr. S. Fishman from the Office of Naval Research under contract No. 96PR07712-00.

8:50 AM +

In-Situ Processing of AlN-Al Alloy Composites: *Qingjun Zheng*[1]; R. G. Reddy[1]; Banqlu Wu[1]; [1]The University of Alabama, Dept. of Metall. and Matls. Eng., A129 Bevill Bldg., 126th Seventh Ave., Tuscaloosa, AL 35487-0202 USA

Experimental study of in-situ formation of AlN reinforced aluminum alloy composites was carried out by bubbling nitrogen gas into aluminum alloy melts. AlN particulate reinforcement was synthesized in the aluminum alloy melts at 1200°C. Mg_3N_2 was not found in the composites when the aluminum alloy contained magnesium. The experimental products were characterized using X-ray diffraction, SEM and EDX. Equilibrium compositions of the system were calculated using Gibbs energy minimization method, indicating that the AlN is thermodynamically stable in Al-N binary and Al-Mg-N ternary systems in a wide temperature range. In addition, the effect of oxygen was discussed.

9:10 AM

In Situ Processing of Cu- TiC Composites: *E. Fras*[1]; A. Janas[1]; A. Kolbus[1]; H. F. Lopez[2]; [1]University of Mining and Metallurgy, Reymonta 23, Krakow, Poland; [2]University of Wisconsin-Milwaukee, Matls. Dept., P.O. Box 784, Milwaukee, WI 53201 USA

The present work was undertaken to in-situ produce TiC particles within a Cu melt containing various amounts of Ti. In this process, a reactive gas (CH_4) was injected into the melt to promote the dissociation of this gas into elementary carbon which then reacted with the Ti of the melt to produce a fine dispersion of TiC particles. Synthesis of the TiC particles is examined in terms of the solid-liquid interface reaction, and multiple nucleation and growth events for the development of TiC in the melt. The microstructural features of the experimental materials thus produced were investigated by X-ray diffraction and optical and scanning electron microscopy (SEM). It was found that the TiC particles formed were of the order of 1-4 μm, and they usually segregated at the α-Cu dendritic boundaries. Also, X-ray diffraction results indicated the presence of Cu_3Ti. Finally, it was found that the volume fraction of reinforcement is strongly influenced by the time of synthesis and the content of Ti in the melt.

9:30 AM

A New Model of Solid Particles Segregation During Centrifugal Casting: *Ludmil B. Drenchev*[1]; Jerzy Sobczak[2]; [1]Institute of Metal Science, 67 Shipchenski Prochod St., Sofia 1578 Bulgaria; [2]Foundry Research Institute, 73 Zakopianska St., Krakow 30-418 Poland

Segregation of solid particles dispersed in liquid rotating slurry occurs owing to centrifugal force. Particles are moved either to the outer or the inner part of cylindrical vessel because of their density difference with the melt. The process of particle segregation evolves together with heat transfer process. These two processes define changes of physical properties in the liquid metal but the changes influence on evolution of the processes. There are different approaches for description of physical interactions, which take place at this complex phenomena characterizing centrifugal casting of composites. Almost all models are one-dimensional because of simplicity and because of the fact that centrifugal acceleration is much greater than gravity. But two-dimensional heat transfer effects particle distribution. Our experience shows that very often for real metal/ceramics centrifugally cast composite castings, the particle rich zone at both ends of cylinder (the upper and down, front and rear part) is more than 100% larger than the same zone at the central part of casting. This means that the real process is strongly two-dimensional. In this paper a 2-dimensional mathematical model has been developed for full analysis of solidification, heat transfer and solid particle movement in a rotating melt in cylindrical cup. Mathematical description and an explicit formula for pressure field p in inner point (r,z) of liquid were obtained: $p = p_0 + p\omega^2 (r^2-r_0^2)/2-\rho g (H-z)$. Here ρ is density of the melt, ω is rotational speed, g is gravity acceleration, r and z are cylindrical coordinates, r_0 is the radius if inner surface of rotating melt and H is length of the cup. Another basic feature of the developed model is that the Coriolis force is included in particle moving equation. This force has an essential meaning when mass of particle is big or rotational speed is very high. Some explicit solutions of moving equation are given. For the purpose of investigating the influence of operating parameters of centrifugal casting such as rotational speed, particles size, pouring temperature, initial mold temperature and initial concentration on final particle distribution, a lot of numerical experiments was carried out. The final particle distribution calculated with the help of the model reflects two-dimensionality of heat transfer process. The results of numerical simulation are compared with experimental data for centrifugally cast A356 aluminum alloy/SiC composite. The correlation between experimental and numerical results is acceptable.

9:50 AM

On the Drag Force Acting on Ceramic Particles during Processing of Cast MMCs: *George Kaptay*[1]; Katalin K. Kelemen[2]; [1]University of Miskolc, Dept. of Phys. Chem., Miskolc, Egyetemvaros 3515 Hungary; [2]University of Miskolc, Dept. of Mech., Miskolc, Egyetemvaros 3515 Hungary

Viscous drag force acting on ceramic particles during production of MMCs is one of the key forces determining the movement of the particles in liquid metals. Hence, its detailed knowledge is of major importance for modeling MMCs. The well-known Stokes equation describing the drag force acting on a spherical, perfectly wetted, single, small particle moving far from any interface with velocities ensuring low values of the Reynolds number (below 1) should be modified for the reasons than in real processing conditions: particles are not perfectly spherical; particles are not perfectly wetted by the liquid; the volume concentration of particles is relatively large; larger particles can move fast enough to create Re higher than; particles can agglomerate during their movement; particles might appear close to the walls of the crucible, or to the solidification front. In the present contribution the main emphasis will be on the theoretical analysis of the effect of the volume concentration of particles on the drag force. A new equation, being different from those existing in the literature will be presented. Our new equation has been validated against experimental results on the sedimentation velocities measured in different systems. In the second part of our paper the agglomeration of particles during their sedimentation in the melt is analyzed. While the particles move in the vertical direction, they will influence each other in a horizontal direction due to the following two forces: the interfacial force, attracting the particles from a distance; the horizontal term of the repulsing drag force, appearing in the case of asymmetrical arrangement of particles relative to each other. From the analysis of these two forces, the corresponding values of the critical concentration of the particles and their critical size can be calculated for each particular system. It has been shown that under "normal" processing conditions of MMCs the coagulation of particles can be considerably reduced if particles are larger than 10 μm in radius. For smaller particles the probability of their coagulation is considerably increased.

10:10 AM Break

10:30 AM

Synthesis of Al-TiC "In-situ" Composites via SHSB Technology: *E. Fras*[1]; A. Janas[1]; A. Kolbus[1]; H. F. Lopez[2]; [1]University of Mining and Metallurgy, Reymonta 23, Krakow Poland; [2]University of Wisconsin-Milwaukee, Matls. Dept., P.O. Box 784, Milwaukee, WI 53201 USA

In this work, Al-TiC were processed "in-situ" via a variant of the self propagating high temperature synthesis (SHS) method. This method can be carried out at lower temperatures and it is known as the SHSB method, where the B stands for bath. This method was considered for processing of TiC reinforced aluminum, where the amount of Al_4C_3 is significantly reduced. It is well known that Al_4C_3 is undesirable in these composites since it easily reacts with the environment and increases its volume to a great extent. This in turn leads to a degradation of the composite properties. Accordingly, the work discusses the method employed, as well as the exhibited microstructures and mechanical properties.

10:50 AM

Porosity in Cast Aluminum Metal Matrix Composites: *B. C. Pai*[1]; R. M. Pillai[1]; K. G. Satyanarayana[1]; [1]Regional Research Laboratory, Coun of Sci. and Indus. Res., Industrial Estate PO, Thiruvananthapuram 695 109 India

The presence of porosity, interfacial reaction between the dispersoid and the matrix and non uniform distribution of the dispersoids in the matrix are the key factors responsible for widening the scatter in the properties of cast metal matrix composites. Generally, aluminum alloy castings are prone for the presence of hydrogen gas porosity and shrinkage porosity unless proper care is taken. Further, in composite melts, the presence of second phase material alters the casting and solidification characteristics leading to higher levels of porosities in composite castings. The identified factors contributing to the higher levels of porosities in the composite castings and the possible remedial measures to be taken to reduce its effects are (I) minimizing the absorbed gases over the surface of the dispersoids by proper surface treatments, (II) reducing sucking in of air during dispersoid introduction as well as stirring through a shallow vortex and proper impeller design, (III) degassing of the melt prior to casting with enough precaution, (IV) avoiding entrapment of gases during mold filling by turbulence free filling and also providing generous air vents and (V) considering the higher viscosity of the composite melt during mold filling and ensuring suitable modifications in casting design. By exercising the above precautions, it is possible to reduce the gas content in the composite melt to an acceptable level. This paper overviews the literature presently available on the subject, compares porosity formation in reinforced and reinforced Al alloy melts and highlights some of the work carried out at the Regional Research Laboratory, Thiruvananthapuram (RRL-T) on Al metal matrix composites prepared by stir casting route with dispersoid systems like SiC_p, $graphite_p$, and short carbon fibers.

11:10 AM

On Reactions during Melting of Aluminum-Coke Composites: *Sanjit Anand*[1]; N. K. Batra[1]; [1]Indian Institute of Technology, Depts. of Matls. and Metall. Eng., Kanpur 208016 India

Metallurgical coke is produced by the destructive distillation of coal at around 1100° centigrade in absence of air. Silica in coke is transformed to silicon carbide at temperatures above 1540° centigrade. This was confirmed in the present work by measuring the loss in weight before casting of the composite. Silicon carbide might react with aluminum if silicon content of the metal is less than 3 pct. or so. The resulting $A1_4C_3$ phase is detrimental to the properties of the composites. The formation of this phase might be avoided by adding several percent of an active element such as titanium to the melt. Silicon carbide will be replaced by far less detrimental but equally effective titanium carbide particles as a reinforcement compared to the $A1_4C_3$ phase.

The Mechanisms of the Massive Transformation - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Phase Transformations Committee
Program Organizers: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; V. K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Monday AM Room: Mississippi
October 9, 2000 Location: Regal Riverfront Hotel

Session Chair: Mats Hillert, Royal Institute of Technology, Dept. of Phys. Metall. and Cer., Stockholm 10044 Sweden

8:30 AM Introductory Remarks: V. K. Vasudevan, University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221 USA

8:35 AM
History of and Perspective on the Massive Transformation: *Thaddeus B. Massalski*[1]; [1]Carnegie Mellon University, Mat. Sci. and Eng. Depts., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The historical aspects leading to the discovery and studies of the massive transformation mode will be reviewed briefly, and followed by an outline of the major challenging concepts that have emerged. The questions of current interest continue to be related to the presence or absence of lattice orientation relationships between the parent and product phases during both nucleation and growth, the formation of the product phase in two-phase fields, the nature of the interface during growth, and the general definition of what constitutes a massive transformation.

9:15 AM
Overview of Central Issues in the Massive Transformation: *Hubert I. Aaronson*[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

The central issues of this symposium are: (i) can the massive transformation take place at temperatures below To in a two-phase field, and (ii) does transformation crystallography play a role equivalent to that exerted during precipitation? A consensus is now developing that the massive transformation can take place below To in its two-phase region when precipitation can be wholly suppressed. Concerning crystallography, repeated appearances of irrational orientation relationships and irrational habit planes bounding interfaces in which no interfacial structure can be discerned with TEM continues to provide strong support for the view that massive: matrix boundaries are incoherent. On the other hand, nucleation theory, new results to be reported in this symposium by Howe and by Nie and Muddle, and recently developed views (with Howe and Hirth) on interphase boundary structure are offered as further support of a dominant role for crystallography in the massive transformation.

9:55 AM
Structure and Migration Mechanism of Irrational Interphase Boundaries Formed during Grain Boundary Precipitation: *Tadashi Furuhara*[1]; Tadashi Maki[1]; [1]Kyoto University, Dept. Matl. Sci. and Eng., Yoshida-honmachi, Sakyo-ku, Kyoto 606-8501 Japan

The interphase boundary structures of grain boundary precipitates in bcc/hcp (Ti-X) and fcc/bcc (Ni-Cr) systems will be discussed. In those systems, the grain boundary precipitate has a nearly rational orientation relationship with one of the adjacent matrix grains but an irrational relationship with the other matrix grain. On both sides of the grain boundary, regardless of the presence of near rational orientation relationship, the precipitate/matrix interphase boundaries exhibit planar facets that often contain ledges and/or regularly aligned dislocations. The geometrical analysis of the faceted interphase boundary in the fcc/bcc case confirms that the observed facet planes contain higher densities of coherent atom pairs for either near-rational or irrational orientation relationships. Those results implies strongly that grain boundary precipitates (and also massive phase) form partially coherent interphase boundaries with both of the adjacent matrix grains and grow by means of ledge mechanisms.

10:35 AM Break

10:40 AM
Critical Limit for Massive Transformation: *Mats Hillert*[1]; [1]Royal Institute of Technology, Dept. of Phys. Metall. and Cer., Stockholm SE-10044 Sweden

The effect of an alloying element on an allotropic transformation under partitionless conditions depends on two factors, at least. The first one is the thermodynamic effects of the alloying element on the relative stability of the two phases. The second one is the interaction between the alloying element and the migrating phase interface, which depends on the structure of the interface. The transformation will be massive if the interface is essentially incoherent. The natural limit for the occurrence of a massive transformation would be the phase boundary of the new phase if the atomic mobilities are high enough for local equilibrium to be established between the two phases across the interface. It will be the T0 line if there is no redistribution between the elements. I principle, anything in between could occur depending on the actual mobilities inside the interface and in front of it and on the tendency of the alloying element to segregate to the interface. Since there is no direct information on these quantities except for the volume diffusivities, it is important to study the limit experimentally and to compare with predictions from theoretical models. The development of models and some recent experimental results will be described.

11:20 AM
On the Question of Massive Transformations within Their Two-Phase Region: *Masato Enomoto*[1]; [1]Ibaraki University, Dept. of Matls. Sci., 4-12-1 Nakanarusawa, Hitachi 316-8511 Japan

The kinetics of austenite to ferrite transformation in low carbon iron alloys during continuous cooling are studied by computer simulation incorporating the influence of boundary mobility and solute drag on the boundary motion. The finite boundary mobility and solute drag by alloying element both reduce the composition region in which the product phase has the same solute concentration as the bulk matrix phase. The consumption of driving force by diffusion within the boundary due to these two effects and the degree to which ferrite transformation is controlled by diffusion of solute in austenite in the (α+γ) two-phase region is discussed.

The Science of Alloys for the 21st Century: A Hume-Rothery Symposium Celebration - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, and the University of California Lawrence Livermore National Laboratory

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

Monday AM Room: Missouri
October 9, 2000 Location: Regal Riverfront Hotel

Session Chair: Lyle H. Schwartz, AFOSR/NA (Rm 732), Aerospace and Materials Science, Arlington, VA 22203-1977 USA

8:30 AM Opening Remarks

8:35 AM Invited
History of the William Hume-Rothery Award: *Karl A. Gschneidner*[1]; James T. Waber[2]; [1]Iowa State University, Ames Lab., Dept. of Matls. Sci. and Eng., 255 Spedding, Ames, IA 50011-3020 USA; [2]Retired, 2651 Caminato Carlitos, Sante Fe, NM 87505 USA

Shortly after the death of Prof. William Hume-Rothery in 1968, several members of The Metallurgical Society (TMS) of American Institute of Mining, Metallurgical and Petroleum Engineers' (AIME) Committee on Alloy Phases (CAP) thought it would be appropriate for a major metallurgical society to establish an award in honor of Prof. Hume-Rothery for his visionary research on the understanding of the fundamentals of the science of metals and alloy formation. Since Hume-Rothery was a member of the Institute of Metals, the CAP members felt that this society might be interested in establishing such an award. However, the Institute of Metals was not responsive to CAP's proposal, so the members explored this possibility with the Board of Directors of TMS. Eventually TMS approved the establishment of this award. The first person to receive this award was

Prof. Paul A. Beck in 1974. Initially the winners received the award, which consisted of a plaque with the likeness of Prof. William Hume-Rothery, at the awards banquet at TMS's annual meeting. In the meanwhile CAP was successful in establishing a special symposium in which the awardee would present the plenary address (in addition to receiving the plaque at the awards dinner), and other scientists active in his/her area of research would present invited papers. Adries R. Meidema was the first Hume-Rothery awardee to be so honored in 1981. This tradition has continued to today, and is one of the highlights of TMS'S annual meetings.

8:50 AM Invited
William Hume-Rothery: His Life and Science: *David G. Pettifor*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK
Abstract text unavailable.

9:15 AM Invited
Materials Research Sponsored by the Air Force Office of Scientific Research: *Lyle H. Schwartz*[1]; [1]Air Force Office of Scientific Research, Aero. and Matls. Sci. Div., Rm. 732, 801 N. Randolph St., Arlington, VA 22203-1977 USA

Materials have always been and will continue to be an enabling technology for the aerospace industry. The AFOSR has played a major role for the last fifty years in facilitating the development and insertion of new materials into aerospace application. The end of the Cold War has accelerated the changes required to continue to satisfy these needs in the new world of rapid deployment, space operations, and affordability driven decision making. In this presentation, I will briefly describe the AFOSR and its current materials research program. Then, focusing my attention on structural materials, I will discuss the needs for the future and conclude with a description of our new emphasis, Materials Engineering for Affordable New Systems, or MEANS. MEANS will focus our attention on the scientific understanding, measurement capability and computational resources required to support the engineering development of new materials.

9:40 AM Invited
Hume-Rothery Rules Revisited: *Thaddeus B. Massalski*[1]; [1]Carnegie Mellon University, Mat. Sci. and Eng. Depts., Forbes Ave., Pittsburgh, PA 15213 USA

Some historical aspects of the discovery and formulation of the Hume-Rothery rules will be reviewed briefly and followed by a discussion of the current views with regard to the electron concentration rule. The importance of the e/a concept has withstood the test of time and has served as a basis for many interpretations of various observed behaviors and properties in alloys. In the Hume-Rothery phases, based on the noble metals Cu, Ag and Au, evidence for the importance of e/a is quite striking, both in the cubic phases and the hexagonal phases. However, the theoretical basis for the e/a rule has seen many interpretations which will be reviewed.

10:20 AM Break

11:00 AM Invited
High-Temperature Reaction Calorimetry of Alloys and Related Materials: *Ole J. Kleppa*[1]; Susan V. Meschel[1]; Qiti Guo[1]; [1]The James Franck Institute, 5640 Ellis Ave., Chicago, IL 60637 USA

This paper will discuss some of the important developments that have occurred during the past 50 years in the area of high-temperature reaction calorimetry. These developments include the general adoption of microcalorimetry for work at elevated temperatures, and also its many applications in such areas as: liquid metal solution calorimetry; liquid + liquid mixing experiments; the new development of oxide melt solution calorimetry; the new technique of solute-solvent drop calorimetry; and finally, the solid + solid direct synthesis calorimetry of intermetallics and related compounds at very high temperature. As a result of these developments, some systematic pictures of the experimental information on the thermochemistry of binary alloys are gradually emerging. Some examples of these systematic pictures will be presented.

Advances in Interconnect & Packaging Materials - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Thin Films & Interfaces Committee
Program Organizers: Frank G. Shi, University of California-Irvine, Department of Chemical Engineering & Materials Science, Irvine, CA 92697 USA; Bin Zhao, Conexant System, Inc., Advanced Process Technology, Newport Beach, CA 92660 USA

Monday PM Room: Field
October 9, 2000 Location: Regal Riverfront Hotel

Session Chairs: Frank Shi, University of California, Dept. of Chem. and Biochem. and Matls. Sci., Irvine, CA 92697 USA; A. Shapiro, Newport Communication, Irvine, CA USA

2:00 PM Keynote
R & D Perspectives of Electrically Conductive Adhesives: *James E. Morris*[1]; [1]State University of New York at Binghamton, Dept. of Elect. Eng., T. J. Watson Sch. of Eng. and Appl. Sci., Binghamton, NY 13902-6000 USA

The term electrically conductive adhesives (ECAs) includes both isotropic and anisotropic conductive adhesives (ICAs and ACAs). The latter term typically implies paste formulations, but also includes the film format, referred to as either anisotropic conductive film (ACF) or z-axis film (ZAF). So the ECA category of materials actually includes some significantly different technologies. This review will not attempt to be comprehensive. Instead, the ECA field will be used as an example of how a technology develops through concept to problem solving, and on to technological optimization. As the technology progresses, the research focuses on the smaller problems, and on improved characterization for the marketplace. The view of the technology also changes. The initial impetus for ECA development for solder replacement was environmental, but the driver has now changed to performance and new applications. These points will be covered in the paper, to expand upon the basic review of the material principles and properties.

3:00 PM Invited
Reworkable Underfills: Materials, Processes, and Reliability: *L. Nguyen*[1]; [1]National Semiconductor Corporation, P.O. Box 58090, M/S 19-100, Santa Clara, CA 95052-8090 USA

Commercial underfill systems have been mainly based on epoxy chemistries. The relatively high Tg, good adhesion, and toughness of the materials meet the most stringent flip chip, and more recently chip scale packages, performance requirements. Furthermore, the low viscosity resins can result in good wicking for fast capillary flow and good wetting of surfaces. However, the high crosslink density of the epoxy also makes reworking of the die or package difficult. Although the materials can typically soften at high temperatures allowing the defective chip or package to be removed, it is difficult to clean the polymer residues without damaging the integrity of the substrate. As a result, a reworkable version of underfills developed for captive use at some OEMs has focused thus far only on ceramic-based substrates. Within the last few years, reworkable underfills have seen a surge in interest due to the growth in both flip chip and chip scale packages. This paper will discuss recent developments in reworkable underfills. The various approaches will be discussed in terms of materials, processes, and reliability performance.

3:25 PM
Role of Underfill Constraints on Thermo-Mechanical Behavior of Flip Chip Solder Joints: *Indranath Dutta*[1]; Ashok Gopinath[1]; Charles Marshall[1]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943 USA

The thermal fatigue life of flip chip solder joints is known to increase substantially due to the presence of an underfill encapsulant between the chip and board. This enhancement is generally attributed to load sharing by the encapsulant. In this study, we investigate the impact of the constraint imposed on the solder joint by the surrounding encapsulant, and its impact on the deformation of the joint using both experimental and numerical approaches. The effect of underfill properties on the imposed constraint has been parametrically studied, as has the impact of underfill defects. The studies are based on a unit-cell approach, where the behavior of one eutectic Pb-Sn solder ball, representing the joint farthest from the neutral point, along with the surrounding encapsulant, has been studied in detail under thermal cycling conditions. The analytical studies are based on finite element modeling, whereas the experimental studies are based on thermal

cycling in a bimetallic load frame which subjects the joint to varying shear strains depending upon the temperature. The results suggest that optimization of encapsulant properties may result in significant hydrostatic constraints on the solder ball, and thereby improve joint life.

3:45 PM Break

3:55 PM Invited
Stress Testing of a Recrystallizing CaO-B2O3-SiO2 Glass Ceramic with Ag Electrodes for High Frequency Electronic Packaging: *Andrew A. Shapiro*[1]; Martha L. Mecartney[1]; [1]University of California at Irvine, Depts. of Chem. and Biochem. Eng. and Matls. Sci. and Eng., Irvine, CA 92697 USA

Recrystallizing CaO-B2O3-SiO2 glass-ceramics are attractive for use in microelectronics packaging since they can be processed at low temperatures (<950°(C) and the resultant material has a low loss tangent. This paper reports on the stability of the Ag electrodes with respect to diffusion in an electric field. Planar multilayer recrystallizing glass-ceramic/Ag samples were prepared and fired at temperatures from 800-950°C. Buried capacitor Planar multilayer recrystallizing glass-ceramic/Ag samples were prepared and fired at temperatures from 800-950°C. Buried capacitor structures with Ag plates 10mm in diameter with a separation distance of 0.1 mm were created. These were stressed with voltages from 50V to 3kV at temperatures from 25°C to 200°C to induce failure. Failure modes observed ranged from increased leakage to catastrophic events. A population was analyzed for mean time to failure characteristics. SEM and EDX were used to try to identify leakage paths and movement of the Ag through the glass-ceramic. An activation energy was determined for the most common failure mode.

4:20 PM Invited
Applications of Thermodynamics in the Study of Interfacial Reactions in Microelectronic Package: C. E. Ho[1]; B. L. Shiau[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Chem. Eng., Chung-Li, Taiwan 32054 China

The Au/Ni is one of the most common surface finishes for the solder-ball pads in the Ball-Grid-Array (BGA) packages. The first layer of the surface finish, which is to be in direct contact with the solder, is a Au layer of about 1 micron thick. Beneath the Au layer is the Ni layer, whose thickness is about 7 micron. After the first reflow, all the Au would leave the interface, and formed many particles of the AuNiSn ternary intermetallic compound inside the solder joint. After the subsequent solid-state aging, most of the AuNiSn intermetallic would come back to the interface. This process repeated itself for additional reflow-aging cycles. In this study, we will use thermodynamic data to rationalize this intriguing behavior. We will also apply thermodynamics to explain a few other phenomena in this system, which are also very interesting.

4:45 PM
Phase Equlibria and Transformations in Cu-Ge Alloys: *Sumanth Jagga*[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

Alloys based on the Cu-Ge system, especially those on the ε_1-Cu_3Ge compound, have considerable potential for application as interconnects in microelectronic devices because of their attractive electrical and barrier properties. Though the thermodynamics of this system has been assessed based on available data, knowledge of phase equilibria, structures of phases and phase transformations is far from complete. The present study was undertaken to address these aspects. Arc-melted cigars of five Cu-Ge alloys ranging in composition from 10 to 30 at.%Ge were prepared, homogenized and then subjected to a series of high temperature treatments followed by quenching in various media. Transformations and reaction temperatures were studied by DTA and the microstructures of both as-cast and heat treated samples were characterized by XRD, OM, SEM, EPMA and TEM. The kinetics and temperature dependence of transformations during continuous cooling were studied using a novel, computer-controlled in situ temperature and electrical resistivity measurement system, coupled with post-mortem analysis of the microstructures. Reaction start and finish temperatures, continuous cooling diagrams, enthalpies and driving forces associated with the $\varepsilon/\varepsilon_2$ to ε_1 phase transformation were determined for alloys containing 20 to 30 at.%Ge. New results related to the electrical resistivity characteristics, structures of the high-temperature phases and the nature of the transformation to ε_1 were obtained. These results will be presented and discussed.

Coating and Joining of Refractory Materials - Current Issues - I

Sponsored by: Structural Materials Division, Refractory Metals Committee
Program Organizers: John J. Stephens, Sandia National Laboratories, Albuquerque, NM 87185-0367 USA; John A. Shields, CSM Industries, Cleveland, OH 44117 USA

Monday PM — Room: Clark
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chairs: John J. Stephens, Sandia National Laboratories, Dept. 1833, Albuquerque, NM 87185-0367; John A. Shields, CSM Industries, Research Dept., Cleveland, OH 44117 USA

2:00 PM Opening Remarks
Joining of Refractory Materials: *John Stephens*

2:10 PM
Tantalum Alloy T-111 Weld Creep Response: *Charles V. Robino*[1]; John J. Stephens[1]; Gerald W. Wellman[1]; Bruce D. Hansche[1]; Mike T. Valley[1]; [1]Sandia National Laboratories, 1833, MS0367, P.O. Box 5800, Albuquerque, NM 87185-0367 USA

Base metal and cross-weld creep tests have been conducted on T-111 alloy (Ta-8W-2Hf). In order to maximize the information obtained in these tests, an electronic speckle pattern interferometry (ESPI) technique was developed to determine the local creep strains throughout the test weldments. This technique is non contact and provides detailed displacement data over a gage area which can encompass the base metal, fusion zone, and heat-affected zone of the weldment. The technique reveals the differing creep rates of the various weld regions, and, when coupled with finite element models of the test configuration, can be used to develop creep response curves for welded structures. The distribution of hafnia precipitates along grain boundaries influences creep response, and relationships between weld microstructure and creep rate will be described. Comparisons between the weld data and literature data for T-111 base metal in various conditions will be also discussed. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000

2:40 PM
Application of Asymmetrical Four-Point Bend Test Method to Measure Shear Strength of Joints: *Ozer Unal*[1]; [1]Ames Laboratory, 207 Metals Dev., Ames, IA 50010 USA

The asymmetrical four-point bend (AFPB) test was used to measure the shear strength of monolithic SiC, SiC fiber-reinforced composite and solder joints. A 6061 Al-alloy was also tested to check the accuracy of results. Testing of monolithic SiC joints was difficult due to the presence of high stress concentration at the loading points. The AFPB test method had a better success with the composites due to their tolerance to contact forces. This test performed best with metallic materials and solder joints. The fixture dimensions of x=30 for the outer span distance and y=4 mm for the inner distance were found to be the most suitable for the test specimens used in this study. The results of experiments and stress analysis by finite element modeling (FEM) will be presented. Ames Laboratory is operated by Iowa State University for the U.S. DOE under contract No. W-7405-ENG-82.

3:10 PM
The Effect of Coating on the Oxide Scale Formation on Mo-B-Si Alloy: *J. S. Park*[1]; R. Sakidja[1]; J. Fournelle[2]; J. H. Perepezko[1]; [1]University of Wisconsin-Madison, Matls. Sci. and Eng. Depts., 1509 University Ave., Madison, WI 53706 USA; [2]University of Wisconsin-Madison, Dept. of Geo. and Geophys., 1215 W. Dayton St., Madison, WI 53706 USA

The oxidation behavior of Mo-Si-B ternary alloys and the effect of coatings have been examined. An alloy with Mo(ss) + T2 monoeutectic composition located very close to the two-phase field has been selected. Following oxidation in air at 1000-1200°C for times up to 100 hrs, the alloy forms a protective and adherent SiO2 layer on the surface. In addition, the formation of Mo-oxide as well as a mixed Mo-rich silicide is observed beneath the glassy oxide scale. The effect of selective coatings on the oxide scale formation has also been investigated. Crystalline coatings of SiO2, ZrO2, Al2O3, Y2O3, HfO2, TiO2, La2O3 were applied by spray deposition on the alloy surface. Similar oxidation tests were performed on the coated specimens. The results on the coating compatibility as well as

the coating effect on the kinetics are presented. The support of ONR (N00014-92-J-1554) and AFOSR (F49620-00-1-0077) is gratefully acknowledged.

3:40 PM Break

3:55 PM
High and Intermediate Temperature Creep Properties of the 54Fe-29Ni-17Co (Kovar) Alloy: *John J. Stephens*[1]; Jerome Rejent[1]; David T. Schmale[1]; [1]Sandia National Laboratories, Dept. 1833, MS0367, P.O. Box 5800, Albuquerque, NM 87185-0367 USA

The 54Fe-29Ni-17Co (wt.%) alloy, known commercially as Kovar, is a controlled expansion alloy, originally developed for glass to metal sealing applications, which also sees extensive application in metal/ceramic brazing. We have investigated the elevated temperature compression creep properties of annealed Kovar (1/2 hr. at 850°C) over the temperature range 350-900°C. The creep properties of this alloy can be separated into two different regimes: a high temperature power-law regime from 650-900°C, with an activation energy of ~ 61. kcal/mole, followed by an intermediate temperature regime from 350-650°C, with an activation energy of ~46. kcal/mole. The latter regime is best fit by the Garofalo sinh equation. Compressive stress-strain curves have also been generated over the temperature range 23-900°C. These results will also be presented, along with selected microstructures of the creep-deformed material. This work was supported by the US Dept. of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Department of Energy.

4:25 PM
Reactive Wetting in Refractory Metal/Ceramic Oxide: *Sean M. McDeavitt*[1]; Garth Billings[2]; Ernesto J. Indacochea[3]; [1]Argonne National Laboratory, CMT, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Integrated Thermal Sciences, Inc., 2228 Northpoint Pkwy., Santa Rosa, CA 95407 USA; [3]University of Illinois at Chicago, CME Dept., 842 W. Taylor St., MC-246, Chicago, IL 60607 USA

The control of interface reactions between dissimilar materials is a significant issue in diverse applications such as ceramic-metal composite design, brazing of ceramics, crucible development for melting of refractory metals. Chemical incompatibility between the metal and nonmetallic material can result in the formation of undesirable phases, due to reactions during joining. One of the activities in the advanced materials research of the Chemical Technology Division at the Argonne National Laboratory is to develop high-temperature filler metals for use in joining different ceramics or ceramics to metals. One of the operating limitations of the current ceramic/metal joints is the low melting point of the commercially available filler materials. One of the concerns with metals having very high melting points is the undesirable chemical reactions between the molten metal and the ceramic material which become more pronounced. These could ultimately degrade the ceramic and produce poor joints. We are attempting to develop refractory-based filler metals that will have significant chemical interaction with the ceramic substrate, to guarantee wetting and bonding, but will still maintain the stability of the joint. Our presentation will discuss the wetting characteristics of simple and complex oxide ceramic materials, and of some non-oxide ceramics, with refractory metals. A sessile type drop test is used under continuous heating until the peak temperature is reached. The wetting angle changes are monitored as a function of temperature. The experiments are conducted in an ultra-pure argon atmosphere.

4:55 PM
Diffusion Bonding of Tungsten and Tungsten Alloys for Fusion Applications: *Charles H. Cadden*[1]; John W. Elmer[2]; Ben C. Odegard[1]; [1]Sandia National Laboratories, P.O. Box 969, MS 9402, Livermore, CA 94551-0969 USA; [2]Lawrence Livermore National Laboratories, P.O. Box 808, Livermore, CA 94551-0808 USA

Plasma containment in fusion devices requires actively cooled components that are compatible with the reaction environment. Generally, an "armor" layer comprised of either a low Z material (carbon or beryllium) or a high Z material (tungsten) bonded to a structural member, typically a copper alloy or stainless steel heat sink. In order to obtain higher operating efficiencies, the design of future fusion devices may require the use of 900°C helium coolant, precluding the use of these heat sink materials. Due to a combination of high temperature strength, low neutron activation and good thermal conductivity, refractory metals are mentioned as candidates for both heat sink and armor applications. The current study examined the diffusion bonding of pure and alloyed tungsten using compliant interlayer materials. Results of microstructural and mechanical evaluations indicate that sound joints were fabricated at temperatures as low as 1000°C, although higher bonding temperatures resulted in improved bond strength.

Electron Backscatter Diffraction: History and Fundamentals of EBSD - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Thin Films & Interfaces Committee
Program Organizers: Adam J. Schwartz, Lawrence Livermore National Laboratory, L-355, Livermore, CA 94550 USA; Mukul Kumar, Lawrence Livermore National Laboratory, L-356, CA, 94550 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Monday PM — Room: Meramac
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chairs: Alwyn Eades, Lehigh University, Dept. of Matls. Sci. and Eng., Bethlehem, PA 18015-3195 USA; Robert A. Schwarzer, Physikalisches Institut der TU, AG textur, Clausthal-Z, Germany

2:00 PM Opening Remarks

2:15 PM Invited
Historical Development of EBSD: *David J. Dingley*[1]; [1]TSL/EDAX, 392 East 12300 South, Draper, UT 84020 USA

This presentation traces the historical development of the automated detection and indexing of electron diffraction patterns in both the scanning and transmission electron microscopes. Though electron backscatter diffraction, EBSD, is the best known of these, the procedures have been extended recently to the transmission electron microscope and a review would not be complete without including this new work.

2:45 PM Invited
Theoretical Framework for Electron Backscatter Diffraction: *Valerie Randle*[1]; [1]University of Wales Swansea, Matls. Eng. Dept., Singleton Park, Swansea SA2 8PP UK

Electron backscatter diffraction (EBSD) is the cornerstone of almost all microtexture (i.e. microstructure-linked crystallographic orientation) investigations. It is also used for phase identification and lattice strain assessment. The most attractive feature of the technique is its ability to perform concurrently rapid, (usually) automatic diffraction analysis to give crystallographic data and imaging with a spatial resolution of less than 0.5 micron, combined with the regular capabilities of a scanning electron microscope. Although an EBSD investigation can proceed totally automatically, it is desirable to have a basic understanding of the physical principles upon which the technique is based in order to facilitate set-up, troubleshooting and in general getting the best out of an investigation. This paper therefore documents fundamental information about EBSD. This includes formation and interpretation of an EBSD Kikuchi pattern, crystallographic terms of reference and descriptors of orientation and misorientation.

3:15 PM Invited
Rodrigues- Frank Mapping of EBSD Data: *Krishna Rajan*[1]; [1]Rensselaer Polytechnic Institute, Dept. of Matls. Sci. and Eng., Bldg. MRC 110, Troy, NY 12180-3590 USA

Electron BackScattered Diffraction (EBSD) as a technique has achieved a great deal of usage with the apparent ease by which one can associate grain specific diffraction information from a polycrystalline sample. It should be recognized, however, that lattice misorientation and not just lattice orientation is a key metric in describing crystallographic characteristics of a microstructure. In this lecture we discuss the utilization of a graphical representation based on misorientation space known as Rodrigues-Frank space mappings. We will point out that this approach is in fact a more suitable and in fact logical means of representing EBSD derived data. In this presentation we shall review some of its basic characteristics and geometric properties and provide some practical examples of texture analysis using this representation to demonstrate its utility.

3:45 PM Break

4:00 PM Invited
Fundamentals of Automated EBSD: *Stuart I. Wright*[1]; [1]TSL/EDAX, 392 E. 12300 S., Ste. H, Draper, UT 84020 USA

Modern automated electron backscatter diffraction (EBSD) systems are capable of rapid determination of crystallographaphic orientation by indexing diffraction patterns without any operator intervention. This presentation will review various steps of the automatic indexing procedure including image processing, band detection and automated indexing. In

addition, issues concerning uncertainties, determination of relevant structural data and calibration of the system will discussed.

4:30 PM Invited
Studies on the Accuracy of Electron Backscatter Diffraction Measurements: *Melik Cumhur Demirel*[1]; Bassem S. El-Dasher[1]; Brent L. Adams[1]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng. Depts., 5000 Forbes Ave., Pittsburgh, PA 15213-2890 USA

Automated orientation measurement on a local basis is now widely accepted for characterization of materials. The technique relies upon indexing of electron backscatter diffraction patterns in a scanning electron microscope. In order to exploit the available information, it is important to understand its limitations with respect to accuracy. Experiments were carried out to measure orientation fields from a silicon single crystal. The orientation dispersion was 1°. Disorientation correlation maps revealed anisotropy in the spatial variation in measured orientation.

5:00 PM Invited
Phase Identification in the SEM using EBSD: *Joseph R. Michael*[1]; Raymond P. Goehner[1]; [1]Sandia National Laboratories, Div. 1822 MS-1405, P.O. Box 5800, Albuquerque, NM 87185-1405 USA

Electron backscatter diffraction (EBSD) has been developed mainly as a tool for the determination of microtextures. Recently, EBSD had proven capable of identifying crystalline phases. It is now routinely possible to identify sub-micrometer sized phases in the scanning electron microscope (SEM) using EBSD. The combination of the excellent imaging capabilities of the SEM with EBSD provides a very powerful tool for materials characterization. This talk will discuss how phase identification is accomplished using EBSD, energy dispersive x-ray spectrometry and a suitable crystallographic database. Examples of phase identification will be shown from the areas of welding, alloy development, semiconductors and particle analysis. Recent work has shown that it is possible to extract unit cell information from unindexed EBSD patterns. This is important for the identification of phases that are not in the database.

General Abstracts: Mechanical Metallurgy and Composite Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Monday PM Room: Jefferson A
October 9, 2000 Location: Regal Riverfront Hotel

Session Chair: Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA

2:00 PM
Effect of Stress Level and Grain Size on the Room Temperature Creep Behaviour of Alpha Titanium Ti-1.6%wt.V Alloy: *Ameet Krishnan Aiyangar*[1]; Sreeramamuthy Ankem[1]; [1]University of Maryland, Depts. of Matls. and Nuc. Eng., Bldg. 090, Stadium Dr., College Park, MD 20742 USA

Titanium alloys are technologically important. It has been known for some time that some titanium alloys undergo creep deformation when subjected to a constant load at room temperature. In this presentation the creep deformation mechanisms as well as the effects of various stress levels and grain size on the creep behavior of this alloy will be discussed. This work is supported by the Office of Naval Research under Grant No. N0001496101819.

2:20 PM
Effect of Grain Size on Ambient Temperature Creep of Beta Ti-9.4%Mn Alloy: *Durgalakshmi Doraiswamy*[1]; Sreeramamurthy Ankem[1]; [1]University of Maryland, Matls. and Nuc. Eng. Depts., Bldg. 090, Stadium Dr., College Park, MD 20742 USA

It has been recently shown that Beta Titanium alloys can creep at ambient temperatures at 95% Yield Stress and that the extent of creep deformation depends on the stability of the beta alloy. In this presentation, the results of our recent study on the effect of grain size on the ambient temperature creep deformation, including creep deformation mechanisms, of Beta Ti-9.4%Mn alloy will be presented. This work is being supported by Office of Naval Research under Grant No. N0001496101819.

2:40 PM
Processing and Properties of Bulk Aluminum-Base Glasses: *Sheng Cheng*[1]; Somesh Mukherjee[1]; Walter W. Milligan[1]; Gary J. Shiflet[2]; [1]Michigan Technological University, Matls. Sci. and Eng. Dept., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]University of Virginia, Matls. Sci. and Eng. Dept., Charlottesville, VA 22904-4745 USA

Metallic glass powders containing 90% Al, 5% Fe and 5% Gd were prepared by atomization and rapidly compacted by powder forging. Subsequent mechanical properties and microstructures were studied. Behavior will be compared to that of similar alloys in ribbon form, as well as nanostructured metals and glassy polymers.

3:00 PM
Mathematical Models of Contact Fatigue of Hypereutektoid Low-Alloyed Steel: *Stanislav E. Kondratyuk*[1]; Bertold B. Vinokur[2]; [1]Ukrainian Academy of Science, Rsch.-Sci. Phys.-Tech. Instit. of Mets. and Alloys, 34/1 Academic Vernadsky Blvd., Kiev 252680 Ukraine; [2]Doctor of Technical Science, 3901 Conshohocken Ave., Apt. E-21, Philadelphia, PA 19131 USA

Hypereutectoid low-alloyed steels use frequently for fabrication of different details (for example, cold and hot rollers, hammer and press stamps at. al). These details undergo to high cycle stresses in the work processes. Therefore, the surface of the details must be possessed of quite enough contact fatigue dependents on the phase and structural state. Since alloying elements change the position of critical points Ac1 and Ac3 temperature, it was built the state diagram of binary system (Fe, Mn, Ni, Mo, V) -C. It is investigated the quantitative influence of the austenitization temperature on the structural components and the chemical composition of the solid solution, and also the quantitative influence of these factors on the contact fatigue. The obtained experimental data was generalized by means of the multiple regression analysis with step-by-step elimination of the insignificant regressors. It is built the mathematical interpolation models determine relation between the number of cycles to reach the spalling and the austenitization temperature, amount of the retained austenite, the formed martensite and the residual carbides, and also of amount of the alloying elements in the solid solution. The graphic representation of the obtained models allow to determining the quantitative dependence of the indicate factors easy and visually.

3:20 PM
Characterization of (TixCr0.6-x) N0.4 Coatings and their Tribological Behaviors Sliding Against Epoxy Molding Compound: *W. H. Zhang*[1]; J. H. Hsieh[2]; [1]Gintic Institute of Manufacturing Technology, Process. Tech. Div., 71 Nanyang Dr., Singapore 638075 Singapore; [2]I-Shou University, Dept. of Matls. Sci. and Eng., 1 Section 1 Hsueh Cheng Rd., Ta-Hsu, Hsiang 84008 Taiwan

In this paper, several graded Ti-Cr-N coatings were deposited by a reactive magnetron sputtering method. These ternary coatings were then characterized using XRD, SEM, TMA, indentation adhesion testing, and wear testing. The focus was on the compositional dependence of the coatings' microstructures, crystal structure, mechanical and thermal properties, and the wear resistance against filled epoxy molding compound. The results can therefore be used as a reference for selecting a proper Ti-Cr-N coating to protect molding tools.

3:40 PM Break

4:00 PM
Effect of Filler Addition on Mechanical Properties of Glass Fiber Reinforced Epoxies: *Nikhil Gupta*[1]; Balraj Singh Brar[2]; Eyassu Woldesenbet[1]; [1]Louisiana State University, Mech. Eng. Dept., 1209 CEBA Bldg., Baton Rouge, LA 70803 USA; [2]Baba Banda Singh Engineering College, Mech. Eng. Dept., Fatehgarh Saheb, Punjab, India

Incorporation of spherical fillers in polymers gives advantage of increased impact strength. A decrease in compressive strength is observed due to such addition but compressive modulus value increases. Present study deals with the theoretical considerations arising due to incorporation of hollow spherical fillers in epoxies. In an attempt to correlate the experimental observations with the theoretical prediction, validity of various commonly used theories is examined. Experimental observations show specimen behavior in close agreement to the slip line field theory. Applicability of this theory for such systems at microscopic and macroscopic level is discussed. The discussion is useful in preparing groundwork for better understanding of such systems and finding suitable and acceptable assumptions to develop a better approach for estimation of mechanical properties of such systems.

4:20 PM +
Synthesis of Magnesium Based Metal Matrix Composites Reinforced by In-Situ Reaction Process: *Md. Abdul Matin*[1]; Lu Li[1]; Gupta Manoj[1]; [1]National University of Singapore, Dept. of Mech. and Product. Eng., 10 Kent Ridge Crescent, Singapore 119260 Singapore

In this study, magnesium-based composites were fabricated using in-situ reaction process followed by hot extrusion. The composites thus synthesized were characterized for microstructural and mechanical properties. Microstructural characterization of the composite samples showed uniform distribution of the reinforcing particles. Tensile test conducted on the composite samples revealed that 0.2% yield strength, ultimate tensile strength and the average elastic modulus increased when compared to that of the monolithic magnesium. XRD analysis conducted on the composite samples showed the presence of magnesium borides and titanium borides phases. The amount of phases formed, their microstructures and the mechanical properties of the composites were rationalized in terms of the process variables.

4:40 PM
Multiple Layer Ceramic Materials from Rolled Sheet Preforms: *Yuri M. Lytvynenko*[1]; Victor P. Katashynsky[1]; [1]Institute for Problems of Materials Science, 3 Krzhizhanovsky St., Kyiv 03142 Ukraine

Production the layering ceramic materials on the base of silicon nitride with layer thickness of around 0,2 mm after a hot pressing is investigated. The sheet preforms for the hot pressing were made by method of rolling of ceramic powders which preliminary treated with a synthetic binder. A mixture formability and an influence of deformation runs on the thickness, porosity and structural strength of the sheet preforms on the rolling are researched. The first layer has been made from the silicon nitride powder but the same material with 20-40% titanium nitride additions was used for second layer. The same basis of the layers results their good sinterability on the hot pressing. The material with numbers of alternate layers after the hot pressing consists of a row of plane boundaries as well as the zones of tensile and pressing stresses between them. The materials were created for heaters.

Rate Processes in Plastic Deformation II: Towards an Unified Theory of Deformation - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA; Thomas R. Bieler, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; Bimal Kad, University of California, AMES Laboratory, La Jolla, CA 92093-0411 USA; Farghalli A. Mohamed, University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697 USA

Monday PM — Room: Lewis
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chairs: Prof. John J. Jonas, McGill University, Dept. of Metall. Eng., Montreal, Quebec H3A 2A7 Canada; Dr. Thomas R. Bieler, Michigan State University, Dept. of Matls. Sci. and Mech., East Lansing, MI 48824-1226 USA

Thermally Activated Deformation Processes - I

2:00 PM Keynote
Influence of Grain Size, Solute Atoms, and Second-Phase Particles on the Creep Behavior of Polycrystalline Solids: *Oleg D. Sherby*[1]; Eric M. Taleff[2]; [1]Stanford University, Depts. of Matls. Sci. and Eng., Stanford, CA 94305 USA; [2]The University of Texas at Austin, Texas Matls. Instit., C 2200, Austin, TX 78712-1063 USA

This paper is written in honor of the late Prof. John E. Dorn, whose life as a teacher and researcher was previously memorialized at an ASM Conference on Creep, held in Cleveland, Ohio. In the 28 years since that conference, understanding of creep behavior has progressed greatly. Three important gains addressed in this paper are the understanding of creep behavior in materials with fine and ultrafine grain sizes, in solid-solution alloys, and in materials containing second-phase particles. Many factors influencing creep of materials with fine and ultrafine grain sizes are now well understood based on diffusion-controlled grain-boundary sliding and dislocation creep processes. Grain-boundary sliding is shown to create very high tensile elongations, ie. superplasticity. High-strain-rate superplasticity (HSRS) is observed for ultrafine grain sizes, often aided by partial grain-boundary melting. HSRS is usually accompanied by a strong threshold-stress behavior, the origins of which are still a topic of debate. Methods for analyzing data have been developed to provide insight into deformation mechanisms even without knowledge of the physical basis for the threshold stress. The influence of solute atoms in binary solid-solution alloys is well understood through Weertman's solute-drag creep mechanism. The effects of solute atoms in more complex material systems, particularly those containing numerous solutes and second-phase particles, are still mysterious. The creep behavior of metal-matrix composites, for example, is seen to be a function of chemistry, particle size, particle volume fraction, and, most importantly, processing history. Experimental results and models proposed on these effects are reviewed. The influence of soaking time and temperature on subsequent creep behavior, which is seldom considered, is shown to be an important factor in all three of the categories addressed in this paper.

2:30 PM Keynote
Creep Resistance of Directionally Solidified Ceramic Eutectics: *Ali S. Argon*[1]; Elizabeth C. Dickey[2]; A. Sayir[3]; [1]Massachusetts Institute of Technology, Dept. of Mech. Eng., Rm. 1-306, 77 Massachusetts Ave., Cambridge, MA 02139 USA; [2]University of Kentucky, Chem. and Matls. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA; [3]NASA Glenn Research Center, Matls. Div., MS 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Creep in directionally solidified ceramic eutectics of Al203/Zr02(Y203) has many parallels to the kinematical features and the kinetics of the nickel based superalloy single crystals of the CMSX-III type where the morphological scale in the ceramic eutectics is similarly in the sub-micron range but components lack coherence requiring separate forms of crystal plasticity in them for compatible deformation. While details of this crystal plasticity are not yet complete, the existing TEM evidence suggests that the extraordinary creep resistance of these eutectics derives primarily from the similarly channeled dislocation flow in the Al203 component, constrained into the sub-micron wide passages between the aligned cubic Zr02 fibrils deforming by entirely different slip systems. The creep resistance is also in part due to the high lattice resistance to dislocation glide and in part due to the high recovery resistance of the Al203 component resulting from the very sluggish diffusional processes in it.

3:00 PM
Power-Law and Exponential Creep in Class M Materials: Discrepancies in Experimental Observations and Implications for Creep Modeling: *S. V. Raj*[1]; [1]NASA Glenn Research Center at Lewis Field, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Certain common characteristics have been observed to be the governing features in the power-law creep regime, which are now considered as fundamental axioms describing creep behavior of materials. However, the mechanisms dominant in the exponential creep region are poorly understood. This paper discusses our current understanding of the processes thought to be dominant in this regime as well as the implications for creep modeling relating to both power-law and exponential creep regions. It is pointed out that creep substructures, other than subgrains, have been reported in the literature, and a bifurcation diagram is presented to demonstrate how this evolution can occur from an initially homogeneous dislocation substructure. The values of $n > 3$ are rationalized using a constant structure plot of normalized creep rate vs. normalized stress. Finally, the use of nonlinear dislocation dynamics in creep modeling is advocated.

3:20 PM Break

3:35 PM Keynote
The Jogged Screw Unit Dislocation in Gamma Ti-Al: *Peter M. Hazzledine*[1]; Satish I. Rao[1]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Electron microscope observations have shown that samples of gamma Ti-Al deformed at moderate temperatures or in creep contain a large density of near-screw unit dislocations. They are bowed out on one or more {111} planes between discrete pinning points. The exact form of the pin is controversial but it results from cross- (or double cross-) slip which is facilitated by the very compact cores of unit screw dislocations revealed in EAM atomistic simulations. In order to move, the dislocation must either drag or circumvent the pins, processes which involve creation and anihilation of point defects. Such processes are rate- and temperature- sensitive. The paper reviews the observations and calculations on the cross-slip of unit screw dislocations and relates these to the macroscopic mechanical properties of creep resistance and yield stress anomaly.

4:05 PM
Evidence for Dynamic Recrystallization during Harper-Dorn Creep: *Farghalli A. Mohamed*[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci. Depts., Irvine, CA 92697-2575 USA

Harper-Dorn creep refers to the anomalous creep behavior first reported in 1957 by Harper and Dorn in their study on the creep behavior of large-grained aluminum. The creep rates associated with Harper-Dorn creep are two orders of magnitude faster than those predicted by Nabarro-Herring creep (diffusional creep). The present investigation provides new substructural evidence that dynamic recrystallization occurs during Harper-Dorn creep. Such evidence is manifested by the distribution of dislocations across grain boundaries and the formation new grains in the microstructure of crept specimens. In addition, it is shown that the shape of the creep curve is consistent with the characteristics of dynamic recrystallization.

4:25 PM
Mechanical Modeling and Microstructural Observation of Pure Aluminum Crept Under Constant Stress: *Keisuke Ishikawa*[1]; Masataka Maehara[1]; Yasuo Kabayashi[1]; [1]Toyo University, Dept. of Mech. Eng., 2100 Kujirai, Kawagoe, Saitama 350-8585 Japan

We carried out the precisely constant stress creep tests on 5 nine pure aluminum. The constant applied stress was kept within the 0.5% to the fracture of the specimen. The deformation behavior of the ductile aluminum was examined macroscopically and microscopically. The macroscopic response of the aluminum was scrutinized through the mechanical models, which was a combination of the Maxwell and the Voigt element. The plastic strain changes were fairly described by the parallel combination of the Maxwell and the Voigt element. We have experimentally obtained the material parameters, which were the viscosity coefficients of the both elements. The results suggest that the dual phase structure would be constructed in the deformed aluminum. The direct observation reveals the cell structure in the deformed aluminum. The viscosity coefficient is assigned to the mobility of dislocation in the both phases. The temperature dependence of the both coefficients is similar. The activation energy is similar to that of the self-diffusion of aluminum. Furthermore, the constant strain rate was not recognized clearly for the aluminum under a condition of constant stress. The process for the decreasing strain rate is dissipative and the cell structure maintains the high stability. After the minimum strain rate, the cell structure would begin to collapse and the damage process is going on. Then the reduction of the density suggests that the actual cross-section of the specimen decreases and the true applied stress increases. Hence the strain rate is accelerated to the rupture.

4:45 PM
Anelastic Deformation Behavior of the Superplastic Zn-22%Al Alloy: *Kyung T. Park*[1]; Hyuk J. Kwon[1]; W. J. Kim[2]; Dong H. Shin[3]; [1]National University of Technology, Div. of Adv. Matl. Sci. and Eng., Taejon 300-717 Korea; [2]Hongik University, Depts. of Metall. and Matls. Eng., Seoul 121-791 Korea; [3]Hanyang University, Depts. of Metall. and Matl. Eng, Ansan 425-791 Korea

Phenomenological description for the anelastic behavior of fine-grained superplastic Zn-22%Al was made in the present investigation. In addition to very large neck-free strain, fine-grained superplastic materials exhibit the extraordinary large relaxation strength that measures the ability of anelastic deformation. Although the abnormal anelastic behavior of these materials may play an important role on materials processing, its characteristics are known very little. This study was aimed at providing the systematic information regarding anelasticity of fine-grained superplastic materials. For this purpose, data for anelastic deformation of fine-grained superplastic Zn-22%Al reported previously were analyzed by expressing the anelastic strain rate as a function of remaining anelastic strain which was regarded as a true driving force for anelastic deformation. The results yielded that three distinct independent deformation mechanisms operated during anelastic deformation of the alloy; two thermally activated processes at long anelastic deformation region and one probably a thermal process at short anelastic deformation region. The characteristics of each deformation region were analyzed in terms of driving force, activation energy and grain size sensitivity. In addition, the present results were discussed in light of the mechanisms suggested for anelasticity of fine-grained superplastic materials.

5:05 PM Extended Discussion

State of the Art in Cast MMC's: Microstructure, Interface and Bonding between Reinforcements and Matrix - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing and Manufacturing Division, Jt. Composite Materials Committee, Solidification Committee
Program Organizer: Pradeep Rohatgi, University of Wisconsin, Materials Department, Milwaukee, WI 53211 USA

Monday PM — Room: Laclade
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chairs: D. Stefanescu, The University of Alabama, Dept. of Metall. and Matls. Eng., Tuscaloosa, AL 35487-0202 USA; N.K.BatraIndian, Institute of Technology, Depts. of Matls. and Metall. Eng., Kanpur 208016 India

2:00 PM
Development of Novel Tool-Less Net-Shape Pressure Infiltration Casting Technology for Manufacturing of Metal Matrix Composites: *M. L. Seleznev*[1]; J. A. Cornie[1]; S. Zhang[1]; A. O. Salvi[1]; [1]Metal Matrix Cast Composites, Inc., 101 Clematis Ave., Unit 1, Waltham, MA 02453-7030 USA

Novel tool-less technology for manufacturing of complex metal matrix composite parts was developed. Preforms for the composites are 3-D printed using CAD/CAM model of the part as an input. Ready preforms are then invested into refractory material and pressure infiltrated with liquid metal. After solidification, the investment refractory is washed away leaving behind a net-shaped composite part. The main benefit of the technology is that the part manufactured literally without tools and design changes are possible at a late stage in development process. Another benefit is that composite preform dramatically gains in quality and consistency. Examples of application of the new technology to manufacturing of prototype aerospace parts are described.

2:20 PM
Analysis of Liquid Convection Effects on the Pushing-Engulfment Transition: *S. Mukherjee*[1]; D. M. Stefanescu[1]; [1]The University of Alabama, Dept. of Metall. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

During the solidification of a liquid containing an insoluble particle, the particle can be instantaneously engulfed, or continuously pushed and then subsequently engulfed. A critical velocity for the pushing-engulfment transition (PET) is observed experimentally. Most models proposed to date ignore the complications arising from the liquid convection ahead of the solid-liquid interface (SLI). They simply solve the balance between the attractive drag force exercised by the liquid on the particle and the repulsive interfacial force. Recently, a dynamic model (non-steady-state) for the interaction between an insoluble particle and an advancing solid-liquid interface has been proposed. It takes into account the virtual mass force, due to the acceleration of the portion adheres to the particle besides the drag and the pushing forces. The Basset force, due to unsteady history effect, is however neglected because it does not have a significant contribution. Besides these forces, the particles are affected by three principle lifting mechanisms: 1) random velocity components greater than the terminal velocity of the particles; 2) Saffman force, due to velocity gradient in the liquid and relative translation velocity between the liquid and the particles; 3) Magnus force due to velocity gradient in the liquid and relative rotation velocity between the liquid and the particle. The present work is an effort to calculate analytically the Saffman and Magnus forces under certain assumptions regarding the nature of fluid flow ahead of the SLI. This allows for quantitative evaluation of the three possible regimes occurring during particle-interface interaction: 1) low convection–no effect on critical velocity for PET; 2) intermediate convection–increased critical velocity; 3) high convection–no particle-interface interaction.

2:40 PM
Particle-Interface Interaction: An Asymtotic Approach: *Layachi Hadji*[1]; [1]The University of Alabama, Math. Dept., P.O. Box 870350, 345 Gordon Palmer Hall, Tuscaloosa, AL 35487-0350 USA

Understanding the interaction between second-phase particles and a solidifying interface is of fundamental importance in the solidification processing of particulate-reinforced metal matrix composites. In this talk, we describe recent results obtained by means of singular perturbation techniques and asymptotics applied to a strongly coupled particle-interface

model. The mathematical model consists of the Navier Stokes equations for fluid flow in which an immobile solid spherical particle is positioned near the moving and deformable solid front. The effects of convection in the melt and heat conduction in the solid and particle are accounted for. The interface equilibrium temperature accounts for the Gibbs-Thomson effects and the disjoining pressure due to the long range intermolecular forces in the gap separating the particle from the front. Our investigation of the crystal-melt morphology near the particle shows that under very low growth rates, quantified here by a very small Stefan number, the solid-liquid interface profile exhibits a singular behavior, namely, the front profile has an infinitre curvature at the origin when the disjoining pressure is the main cause of interface deformation. The effect of the thermal conductance difference is also studied. We show that there exists a disparity in scales in the effects of the disjoining pressure and thermal conductiviy difference in determining the shape of the interface, and thus these two effects are mainly uncoupled and can be investigated separately. Expressions for the gap thickness separating the particle from the front are derived and used to obtain expressions for the critical growth velocity for engulfment. The question of the morphological instability of the interface and how it relates to the engulfment process is also addressed. The asymptotic results are contrasted with other analytical and experimental results.

3:00 PM
Optimized Wetting of Silicon Carbide by Aluminum Alloys: *M. I. Pech-Canul*[1]; M. M. Makhlouf[1]; R. N. Katz[1]; [1]Worchester Polytechnic Institute, Dept. of Mech. Eng., Worchester, MA USA

The quantitative effects of magnesium and silicon additions to aluminum, free silicon on the SiC substrate, nitrogen gas in the atmosphere, and the process temperature on the wetting characteristics of SiC by aluminum alloys was investigated using the sessile drop technique. The contribution of each of these parameters and their interactions, in terms of relative power, to the contact angle, surface tension, and driving force for wetting were determined. In addition, an optimized process for advanced wetting was suggested. Wetting experiments were performed using the optimum conditions and the results show excellent agreement with projected values. Results indicate that the presence of free silicon on the surface of SiC significantly reduces the contact angle between the molten alloy and the substrate. The positive effect of silicon on the contact angle is attributed to a chemical reaction in which both SiC and aluminum are active participants. The results also indicate that nitrogen gas as the process atmosphere positively influences the liquid/vapor surface tension and the presence of magnesium in the aluminum alloy favorably affects the overall driving force for wetting. The optimum conditions for wetting SiC by aluminum that were arrived at were used to infiltrate SiC_p preforms and the mechanical properties of the resulting metal matrix composites were measured.

3:20 PM
Stabilization In and Rejection of Ceramic Particles from Molten Al-Alloy–Modeling and Experimental Testing: *V. M. Kevorkijan*[1]; [1]Independent Researcher, Partizanska 38, Slovenska Blstrica 2310 Slovenia

A model was developed to explain the stabilization of ceramic particles dispersed in a molten metal by equilibrium interfacial coupling between the ceramic particles and the molten metal. Such equilibrium interfacial coupling is proposed to be an energy activated process determined by the equilibrium conversion and the overall kinetics of the applied interfacial chemical interaction. In addition, the model was experimentally verified by observing the rejection of BN-coated SiC particles from the molten aluminum alloy. The moment of rejection was experimentally determined by measuring of changes in electrical resistance of the slurry and the variations in electrical power required for its constant stirring. Experiments also showed that BN surface coated SiC powder used in this study can be successfully immersed in an aluminum melt. Furthermore, this cost effective technique can be applied in order to prepare a stable and highly concentrated (>30 vol. %) metallic suspension of ceramic particles different in their particle size and specific surface area, which is of the great practical interest in production of discontinuously reinforced metal matrix composites.

3:40 PM Break

4:00 PM
Influence of Wettability on the Mechanical Properties of the Interface in Metal-Alumina: *N. Sobczak*[1]; R. Asthana[2]; M. Ksiazek[1]; W. Radziwill[1]; B. Mikulowski[3]; I. Surowiak[3]; [1]Foundry Research Institute, Zakopianska Str 73, Krakow 30-418 Poland; [2]University of Wisconsin-Stout, Manu. Eng. Pgm., Menomonie, WI 54751 USA; [3]Academy of Mining and Metallurgy, Al Mickiewicza 30, Krakow 30-059 Poland

A fresh approach has been advanced to evaluate the relationship between the wettability and interface properties in Al-Al_20_3, Al-6Ti-Al_20_3, Al-llSiAl_20_3, and Al-(Ti-coated) Al_20_3 systems. The high-temperature wettability in the above systems was characterized in terms of contact angles measured using the sessile-drop tests. The solidified sessile-drops were then carefully bisected perpendicular to the substrate at the mid-plane of the contact circle to obtain samples for 1) measuring the bond shear strength using a 'pushoff' test on the flat end of one-half of the bisected droplet, and 2) evaluating the interface microstructure and chemistry on thin wafers obtained by serial sectioning of the other half of the bisected sessile-drop, The new approach overcomes some of the drawbacks of the earlier 'push-off' tests in which the applied stress was not truly perpendicular to the pushed surface (i.e. parallel to metal-alumina interface) due to curvature of the droplet at the joint. The new approach also facilitates assessment of the variation of the interface structure, chemistry, and bond shear strength within an environment identical to that during the wettability test, thus allowing a more reliable correlation to be established between the bond strength and the wettability parameters in metal-ceramic systems. The key test variables include the alloy composition, nature of the alumina substrate, substrate modification using coatings, coating thickness and application technique, and the stability of the coating at elevated temperatures.

4:20 PM
Thermal Mismatch Stresses in a Metal Matrix Composite–A Finite Element Analysis: *S. C. Sharma*[1]; M. Krishna[1]; [1]R.V. College of Engineering, Dept. of Mech. Eng., Bangalore, India

This investigation focused on the finite element analysis of distribution of residual thermal stress in the interfacial region of aluminium-albite metal matrix composites. The residual thermal stress was induced by coefficient of thermal expansion (CTE) mismatch between the matrix and reinforcement. The result indicates that the properties of the interfacial region affect the stress distribution, the interfacial crack initiation and propagation, and the mechanical response of the composites.

4:40 PM
Effect of Mg and Cu on Superplasticity of Discontinuous Fiber Reinforced Aluminum Composites: *T. Imai*[1]; I. Tochigi[1]; T. Kawasaki[1]; J. Mao[1]; M. Takagi[1]; [1]Kanagawa High-Technology Foundation, 7-1 Ohgigaoka Nonoichi, Kanagawa 921 Japan

Si_3N_4w/Al-Mg, Si_3N_4w/Al-Cu composites fabricated by squeeze casting were extruded and the superplastic characteristics were investigated. Si_3N_4w/Al-3wt%Mg composite indicates the m value of 0.4 and the total elongation of about 260% at the strain rate of $10^{-1}s^{-1}$ and 858 which is just above solid temperature of Al-3wt%Mg matrix. But Si_3N_4w/Al-2wt%Cu shows the total elongation of about 100%.

The Mechanisms of the Massive Transformation - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Phase Transformations Committee
Program Organizers: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; V. K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

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October 9, 2000 — Location: Regal Riverfront Hotel

Session Chair: Vijay K. Vasudevan, University of Cincinnati, Matls. Sci. and Eng. Depts., Cincinnati, OH 45221-0012 USA

2:00 PM
The α to γ_m Massive Transformation in Ti-Al Alloys: Ping Wang[1]; D. Veeraraghavan[2]; Mukul Kumar[3]; Zhihong Zhang[4]; *Vijay K. Vasudevan*[4]; [1]Brown University, Div. of Eng., P.O. Box D 182 Hope St., Providence, RI 02912 USA; [2]VLSI Technology, 9651 Westover Hills Blvd., San Antonio, TX 78251 USA; [3]Lawrence Livermore National Laboratory, 7000 East Ave., Livermore, CA 94550 USA; [4]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

The massive-matrix interphase interfaces associated with the α to γ_m massive transformation in Ti-(46-48)Al alloys were studied. Nucleation and growth kinetics were determined and the former compared with calculations for various nuclei shapes using classical nucleation theory. Special experiments were performed to arrest the transformation at an early stage. Orientation relations between the γ_m and parent α (retained α_2) phases were determined using EBSD in an SEM and by electron diffraction in a TEM, and the interphase interfaces characterized by two-beam bright-field/weak-beam dark-field TEM and HRTEM. The results reveal that the γ_m nucleates at grain boundaries with a low-index orientation relation and coherent interface with one parent grain, but grows into the adjacent grain with a

high-index/irrational orientation relation. The growth interfaces between the two phases are generally free of misfit dislocations and consist of curved parts as well as planar facets whose macroscopic habit varies from high -index/irrational to low-index orientation. On an atomic scale the interphase interfaces are often found to be faceted along {111} planes with steps, but are incoherent with respect to the parent grain into which growth occurs. The implications of these results on the nucleation and growth mechanisms associated with the α to γ_m massive transformation will be discussed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

2:40 PM
The Massive Transformation in Gamma-Based Titanium Aluminides: *Raju Vijayaraghavan Ramanujan*[1]; [1]Bhabha Atomic Research Center, Matls. Sci. Div., Trombay, Mumbai, Maharashtra 400085 India

Gamma-based titanium aluminides have attracted intense interest as high temperature structural materials. Solid state phase transformations play a dramatic role in determining the morphology of these intermetallics. A change in the cooling rate for the alpha to gamma phase transformation can produce a composition invariant massive transformation. The parent phase is disordered while the product phase is ordered, unlike conventional massive transformations. This ordering results in a complex defect structure which has been analysed via TEM. The interfacial structure of the alpha:massive gamma boundary is semi-coherent and ledges have been observed via TEM. The defect structures interact with the interphase interface. The thermodynamics and kinetics of the formation of the massive morphology is analysed in the light of this experimental evidence. The importance of Aaronson's predictions regarding the interfacial structure will be highlighted.

3:20 PM
HRTEM Investigation of Massive Transformation Interfaces in a TiAl Alloy: *James M. Howe*[1]; Vijay K. Vasudevan[2]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., 116 Engineer's Way, Charlottesville, VA 22904-4745 USA; [2]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

In this study, high-resolution transmission electron microscope (HRTEM) techniques were used to investigate the atomic structure of massive transformation interfaces in a Ti-46.5Al alloy, quenched from the high-temperature alpha phase field to form massive-gamma. Although grains of massive-gamma generally had an irrational orientation relationship with the alpha-2 phase (alpha at high temperature), in at least one case, it was possible to image the interface edge-on and closely model the atomic structure of the interface by matching simulated and experimental HRTEM images. These results reveal that alpha-2:massive-gamma interfaces can exhibit commensurability along at least one direction in the interface between relatively high-index planes in the two phases by suitable choice of interface plane. The interfaces may also be faceted on an atomic scale. The implications of such findings in terms of possible alpha-2:massive-gamma interface motion are discussed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9908855 (JMH) and DMR-9224473 and 9731349 (VKV), Dr. Bruce MacDonald, Program Monitor.

4:00 PM Break

4:05 PM
Interfacial Structure Associated with Massive Transformation Product γ_m in a Near TiAl Alloy: *Jian-Feng Nie*[1]; Barry Muddle[1]; [1]Monash University, Dept. of Matls. Eng., Clayton, Victoria 3800 Australia

Massive transformations have been observed in a range of ferrous and non-ferrous alloy systems. However, the interfacial structures of massive-matrix boundaries and their role in the growth of the massive product remain controversial. A feature characteristic of this class of phase transformations is the lack of a rational orientation relationship between the massive product and the matrix phase, and it is on this basis that the structure of the massive/matrix interface is often suggested to be incoherent. In the present study, the interface orientation and the interfacial structure of the massive phase γ_m, which forms in a Ti-46.5wt%Al alloy, have been characterised in detail using transmission electron microscopy. Preliminary observations indicate that there is no rational orientation relationship between the massive phase γ_m and the matrix phase α_2, and three types of interface have been observed between the two phases: (i) planar interfaces that are irrational with respect to both phases but which are parallel to a near coincidence site lattice plane, (ii) faceted interfaces that contain arrays of linear defects, and (iii) faceted interfaces that are apparently free of linear defects. The structure of the interface between the γ_m and α_2 phases will be discussed in the context of these experimental observations.

4:45 PM
The Alpha-Gamma Massive Phase Transformation in Titanium Aluminides: *James Edward Wittig*[1]; [1]Vanderbilt University, Matls. Sci. and Eng. Dept., P.O. Box 351683 STA B, Nashville, TN 37235-1683 USA

The massive phase transformation of hexagonal alpha titanium aluminide into the L1o TiAl gamma phase is well documented. However the nature of the nucleation and growth is still a topic of some controversy. This study uses an undercooling rapid solidification process that captures the earliest stages of nucleation as well as the later stages of growth. In addition to a binary Ti52Al48 (at%) alloy, this investigation has studied the effects of ternary additions (Cr and Nb) on the massive transformation. Characterization of the massive phase transformation utilized standard transmission electron microscopy (TEM) diffraction contrast experiments combined with high resolution TEM and atom location by channeling enhanced microanalysis (ALCHEMI) for ternary element sub-lattice site occupancy. The data show that the kinetics of the transformation can be strongly influenced by the ternary elements whereas Cr strongly retards the gamma growth. In this material system, the massive gamma heterogeneously nucleates at alpha grain boundaries and then grows into the adjacent alpha grain with preferred {111} facets and an incoherent strain free interface. This research was sponsored by the National Science Foundation Division of Materials Research. The author gratefully acknowledges the contributions of Ulrich Dahmen (National Center for Electron Microscopy, Lawrence Berkeley Laboratory) and Ian Anderson (SHARE Program, Oak Ridge National Laboratory).

The Science of Alloys for the 21st Century: A Hume-Rothery Symposium Celebration - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, and the University of California Lawrence Livermore National Laboratory

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552

Monday PM — Room: Missouri
October 9, 2000 — Location: Regal Riverfront Hotel

Session Chair: Robert J. Gottschall, U.S. Department of Energy, Office of Basic Energy Sciences (SC-13), Germantown, MD 20874-1290 USA

2:00 PM Invited
Vision for the Future of the Science of Alloys: *Robert J. Gottschall*[1]; Angus A. Rockett[2]; [1]U.S. Dept. of Energy, Matls. Sci. and Eng. Depts., SC-13, 19901 Germantown Rd., Germantown, MD 20874-1290 USA; [2]University of Illinois, Dept. of Matls. Sci. and Eng., 1-107 Eng. Sci. Bldg., 1101 W. Springfield Ave., Urbana, IL 61801 USA

The DOE/Basic Energy Sciences/Materials Sciences and Engineering program is driven by ideas from the research community. Broad areas of opportunity for the science of alloys exist and will be discussed including nanoscience and the understanding of how the deliberate tailoring of materials on the nanoscale can lead to novel and enhanced functionalities; complex and collective phenomena involving very large numbers of interacting atoms; computer simulation and modeling over a wide range of length scales that may lead to convergence between earlier models that were valid at limited and non-overlapping length scales, or unified models that are valid over all length scales, that can link chemical and physical principals over all size ranges from 0.1 nm to macroscopic to predict the properties and behavior of real materials; and the development of an improved or new suite of real-time and in-situ diagnostics with electron beam microcharacterization.

2:25 PM Invited
The Hume-Rothery "Parameters" and Bonding in the Hume-Rothery and Transition Metal Alloys: *Richard E. Watson*[1]; M. Weinert[1]; L. H. Bennett[2]; [1]Brookhaven National Laboratory, Phys. Dept., P.O. Box 5000, Upton, NY 11973-5000 USA; [2]George Washington University, Instit. for Magnet. Rsch., Washington, DC 20052 USA

It is timely to reconsider the parameters in terms of which Hume-Rothery and others considered alloying trends, namely size, electronegativity and valence electron count. Two of these have remained useful to this day while one requires some redefinition. These, and what must be

added to them, will be discussed in terms of experiment and calculation. Recent calculations of ordered compound phase stability will be inspected and their relevance to our understanding of the above parameters reviewed. Work supported by the Division of Materials Sciences, U.S. Dept. of Energy under Contract No. DE-AC02-98CH10886.

3:05 PM Break

3:30 PM Invited
From Hume-Rothery's 8-N Rule to Valence Electron Rules for Zintl Phases and Extensions to Rationalize Observed Structural Features of Iono-Covalent and Semimetallic Compounds: *Erwin Parthé*[1]; [1]University of Geneva, Dept. of Inorg. Chem., Science II, 30 Quai E.Ansermet, Geneva 4 CH-1211 Switzerland

The 8-N rule by Hume-Rothery (1931) for structures of main group elements relates the number of covalent atom bonds with the group number, i.e. the number of valence electrons. The atoms, by forming shared two-electron bonds, complete their octets. Hume-Rothery's idea was subsequently applied to rationalize anion-anion bonds in ionic compounds. A generalized 8-N rule which considers both anion-anion and cation-cation bonds was published in 1964/65. It can be used with iono-covalent and also strongly polar intermetallic compounds, the latter referred to as Zintl phases. Structural features are interpreted in terms of underlying ionic bonds where electropositive elements transfer valence electrons to electronegative elements which complete their octets, if necessary by forming homonuclear bonds. The generalized 8-N rule has recently been combined with other valence rules to derive for iono-covalent compounds possible base polyhedra, small building units, with which anionic polyhedron complexes can be constructed.

4:10 PM Invited
Value of Chemical Bonding Models for Prediction of Properties of New Classes of Materials: *Leo Brewer*[1]; [1]University of California at Berkeley, Dept. of Chem., M.C. 1460, Berkeley, CA 94720 USA

New processes to solve the many problems that threaten life on earth will require materials with novel properties. Many multi-component phases will be required. Just mixing elements together randomly is not practical. Chemical bonding models can be very powerful in predicting the crystal structures and properties that one can obtain for given mixtures, but they require promotion energies and bonding energies for the various combinations of s, p, d and f electrons. As an example, the three configurations that would predominate for bcc, hcp and ccp crystal structures are given with their promotion energies for ruthenium in kilokelvin: d^7s, 0; d^6sp, 36.3; d^5sp^2, 70. As the number of d electrons is replaced by p electrons, the bonding energies increase and the stability of the solids are 0.4, 0 and 11 kK. In calculating the solubilities in other elements, one would start with the Ru with the same crystal structures as the elements to which it is added. Ruthenium behaves as three different elements depending upon the environment.

4:50 PM Invited
Controversial Concepts in Alloy Theory Revisited: *David G. Pettifor*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

Hume-Rothery strongly believed that electron theory would help industrial metallurgists develop new and better alloys by providing concepts that were underpinned by quantum mechanics rather than empiricism. This talk will focus on three concepts that aroused confusion and controversy amongst academics in Hume-Rothery's day: firstly, the relevance of Jones' theory of Brillouin Zone touching to the experimental Hume-Rothery rules; secondly, the relevance of Pauling's theory of resonant bonds to transition metal cohesion; and thirdly, the relevance of the Engel-Brewer theory to the structural stability of transition metals and their alloys. I will end by asking whether the advent of quantitative electron theory will indeed help industrialists design new and better alloys in the 21st century.

NOTES

Tuesday, October 10, 2000

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
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Advances in Interconnect & Packaging Materials - III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Thin Films & Interfaces Committee
Program Organizers: Frank G. Shi, University of California-Irvine, Department of Chemical Engineering & Materials Science, Irvine, CA 92697 USA; Bin Zhao, Conexant System Inc., Advanced Process Technology, Newport Beach, CA 92660 USA

Tuesday AM Room: Field
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Changming Jin, International SEMATECH, Inc., Austin, TX 78741 USA; Paul Besser, AMD-Motorola Alliance Logic Technology, Austin, TX 78721 USA

8:30 AM Invited
Evaluation of Ultra-Low-k Dielectric Materials for Advanced Interconnects: *Changming Jin*[1]; H. S. Lin[1]; J. T. Wetzel[1]; [1]International SEMATECH, Inc., 2706 Montopolis Dr., Austin, TX 78741 USA

Interconnects become the bottleneck for continued IC performance improvement when the feature size scales down to deep-sub-micron region and below. The gain in device speed at the gate level is offset by propagation delays at metal interconnects due to the increased RC time constant. The RC time delay can be reduced by the incorporation of low dielectric constant (k) materials and/or high conductivity metals. The use of low-k dielectric materials also lowers power consumption and reduces crosstalk. Currently, low-k materials with dielectric constant k ~ 3.0 3.8 are being used in advanced semiconductor manufacturing. As predicted by the International Technology Roadmap for Semiconductors, continued scaling of devices will require ultra-low-k materials with k values less than 2.5 for the 100 nm 130 nm technology nodes. Incorporation of porosity into dense dielectrics is an attractive way to obtain ultra-low-k materials. The ultra low dielectric constant results from the incorporation of pores. Porous dielectric films are deposited through sol-gel or templated processes. Electrical and physical properties of ultra-low-k materials have been characterized. Integration evaluations showed both feasibility and challenges of porous low-k materials. This paper discusses issues and recent progresses made with ultra-low-k material properties, deposition processes, characterization metrologies, and process integration.

8:50 AM Invited
Advanced Metrology for Rapid Characterization of Low K and Copper Materials: *S. H. Lau*[1]; Eli Tollentino[1]; Yuen Lim[1]; Ann Koo[1]; [1]Frontier Semiconductor, 1631 N. First St., San Jose, CA 95112 USA

With the proliferation of several types and classes of low k materials, the screening, evaluation and integration of these new materials into copper interconnect structures require a new and more effective approach. Evaluation of thermal mechanical properties of new materials require multiple complementary metrology toolsets. For example, during initial material screening, information on stress hysteresis, thermal stability, outgassing, film shrinkage, thermal expansion coefficient, adhesion and other material and electrical property changes, after a heat cycle need to be determined. Following curing, cleaning, etching, stripping and CMP steps, outgassing, adhesion and electrical properties need to be remeasured. With new materials, samples are often limited in quantities and process conditions may not be repeatable. Besides, conditions for analysis from multiple metrology toolsets are not uniformed. Correlating results from these analysis can be very time consuming and conclusions ambiguous. To overcome these limitations, we report two advanced metrology tools that have a potential to rapidly screen and evaluate thermal mechanical properties through the integration process. The first is an insitu integrated metrology tool that simultaneously measures several physical, optical, chemical and electrical changes during a heat cycle, including Stress hysteresis, Outgassing, In situ film shrinkage, insitu reflectivity and sheet resistivity changes. Complementing this tool, we also describe a new image analysis based Quantitative Adhesion Tester based on the MELT (modified edge lift off test) principle, for fast screening and ranking of adhesive or cohesive qualities of new materials through their integration process.

9:10 AM Invited
Comparative Study on the Effect of Misalignment on Bordered and Borderless Contacts: *J. S. Huang*[1]; A. S. Oates[1]; S. H. Kang[1]; T. L. Shofner[1]; R. A. Ashton[1]; Y. S. Obeng[1]; [1]Lucent Technologies, Bell Labs., 9333 S. John Young Pkwy., Orlando, FL 32819 USA

Continued shrinking of feature sizes in IC circuits has raised increasing reliability concern. In order to achieve higher packing density, interconnects are migrating toward borderless contacts and vias. The penalty with the absence of metal extension, however, is the decrease in electromigration lifetimes. The issue arises particularly for the misalignment case, which may occur due to process variability. In this paper, we study the effect of misalignment on bordered and borderless contacts. We found that the misalignment effect becomes more evident in borderless contacts. Microstructure is examined by focus-ion-beam and scanning electron microscopy. The case of multiple via configuration is also studied. The results indicate that electromigration lifetimes increase with increasing number of vias in series. The experimental data is consistent with finite element model results.

9:35 AM
Modeling of Joule Heating and its Implications in Multilevel Interconnects: *Yu-Lin Shen*[1]; [1]The University of New Mexico, Dept. of Mech. Eng., Albuquerque, NM 87131 USA

Thermal management is becoming an increasingly important issue in advanced interconnects. A main challenge is Joule heating resulting from more metallization levels, higher current densities, and the incorporation of low-k dielectric materials. In this work interconnect Joule heating is studied numerically. Representative models featuring multilevel structures (from single level to eight level) are used for the finite element analysis. Particular attention is devoted to the effects of current density, the number of conductor levels, and the materials used as the metallization and interlevel dielectrics. Transient heat conduction analyses are carried out to quantify the temperature rise. It is found that increasing the total number of metal levels and/or switching the dielectric from silicon oxide to polymer-based low-k dielectric can cause substantial temperature increases. The maximum temperature increases with the current density and the metal wiring density in an exponential manner. Incorporating low-k dielectrics based on an embedded insertion scheme can greatly relieve the heating problem. An example of reducing the temperature rise by adapting a cooling mechanism at the package level is also illustrated numerically. The effects of constant versus dc currents are discussed. The effects of Joule heating in metal lines containing voids are also explored.

9:55 AM Invited
Microstructural Characterization of In-Laid Copper Interconnect Lines: *Paul R. Besser*[1]; Stewart Rose[2]; Matt Herrick[3]; Brett Baker[3]; Martin Gall[3]; Mike Tiner[2]; Cristiano Cappasso[3]; Ehrenfried Zschech[4]; Werner Blum[4]; Greg Braeckelmann[3]; Cindy Simpson[3]; Hisao Kawasaki[3]; Larry Zhao[1]; Stacye Thrasher[3]; Yusheng Feng[3]; Greg Hamilton[3]; [1]AMD-Motorola Alliance Logic Technology, Mail Stop MD K-10, 3501 E. Bluestein Blvd., Austin, TX 78721 USA; [2]Motorola, Phys. Analytical Lab., Austin, TX USA; [3]Motorola, Adv. Prod. Rsch. and Dev. Lab., Austin, TX USA; [4]AMD Saxony Manufacturing GmbH, Matls. Analysis Dept., Wilschdorfer LandstraBe 101, M/S E32-MA, Dresden D-01109 Germany

The 0.18μm technology node marks the point at which many IC manufacturers are replacing Al interconnects with Cu. Copper interconnect lines present a host of reliability and manufacturing challenges since they are fabricated using in-laid methods. Many researchers are investigating the room temperature grain growth phenomena in blanket films. Since Cu will be implemented on the final product in the form of lines, the current research has focussed on microstructure of in-laid Cu lines as a function of annealing conditions, post-plating and post-CMP. Samples in the current work were produced using conventional fabrication techniques for in-laid Cu lines. Lines with 0.25, 0.6 and 1.0 μm widths were patterned into an oxide dielectric. Following deposition of a barrier and Cu seed, the 4.5 kA deep trenches were completely filled by electrochemical deposition of Cu prior to CMP. The grain size distribution was measured from FIB images using the mean intercept method, and crystallographic texture was characterized by full pole figures. Grain size and texture were measured as a function of anneal temperature (pre and post-CMP) and after deposition of the capping dielectric. It will be shown that the median grain size is a function of temperature and time of the anneal. It will also be shown that, for these samples, the grain growth in the field is independent of the grain growth in the trenches.

10:15 AM Break

10:25 AM
Spontaneous, Non-Aqueous Electrochemical Deposition of Copper and Palladium: R. Fang[1]; H. Gu[1]; W. -S. Shih[2]; *M. J. O'Keefe*[1]; J. Snook[2]; K. D. Leedy[3]; R. Cortez[3]; Y. -K. Lee[1]; T. J. O'Keefe[1]; [1]University of Missouri-Rolla, Dept. of Metall. Eng., 1870 Miner Circle, 292 McNutt Hall, Rolla, MO 65409 USA; [2]Brewer Science, Inc., 2401 Brewer Dr., Rolla, MO 65401 USA; [3]Air Force Research Laboratory, AFRL/SNDI, 2241 Avionics Circle, Bldg. 620, WPAFB, OH 45433 USA

Traditional methods for depositing thin films onto semiconductor wafers, chemical vapor deposition (CVD) and physical vapor deposition (PVD), must now compete, and be compatible, with electrochemical deposition techniques such as electroless and electrolytic plating operations. A novel,

non-aqueous process of spontaneously depositing copper and palladium from organic solutions onto patterned and unpatterned PVD metallic thin films commonly used in the microelectronic industry, such as aluminum, titanium, and titanium nitride, has been developed. The organic solution based process can be used to selectively activate only the exposed surface of PVD metallization, making subsequent deposition of layered films by electroless and electrolytic plating processes feasible. A description of the technology will be presented along with examples of technology integration for underbump metallization on aluminum bond pads, metallization build up for multi-chip module fabrication, and copper seed layer deposition on IC interconnect diffusion barriers.

10:45 AM Invited
New Developments in Diffusion Barriers for Copper Interconnect Technology: *Christoph Steinbruchel*[1]; Bin Zhao[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. and Eng. Depts., 110 8th St., Troy, NY 12180 USA; [2]Conexant Systems, Newport Beach, CA 92660 USA

The present status of work on diffusion barriers for copper in multilevel interconnects is reviewed, with particular emphasis on TiN and TaN, and silicon dioxide as the interlayer dielectric. New results are presented on these materials for 5, 10, and 20 nm thick barrier layers, combining thermal annealing and bias temperature stress testing. With both stress methods, various testing conditions are compared using capacitance-vs-voltage (C-V) and leakage current-vs-voltage (I-V) measurements to characterize the stressed samples. From an evaluation of these results and a comparison with results from other testing approaches, conditions for a consistent testing methodology of barrier reliability are outlined. Additional results are reviewed regarding: 1) the properties of new barrier materials (primarily other metal nitrides and ternary compounds) on silicon dioxide; 2) the effect of different deposition methods for producing a barrier material on its effectiveness as a diffusion barrier; 3) emerging work on diffusion barriers for copper on new interlayer materials with a very low dielectric constant.

11:00 AM Invited
Effect of Polydispersity on the Electrical Conduction in Electrically Conductive Adhesives with a Bimodal Size Distributed Conducting and Inert Particles: *Mikrajuddin*[1]; F. G. Shi[2]; K. Okuyama[1]; [1]Hiroshima University, Fac. of Eng., Kagamiyama, Higashi-Hiroshima 739-8527 Japan; [2]University of California, Chem. and Biochem. Eng. and Matls. Sci. Dept., Irvine, CA 92697-2575 USA

We have investigated theoretically the electrical conduction development in electrically conductive adhesives (ECA) composite of bimodal size distribution of conductive fillers and inert particles. We showed that the percolation threshold for the presence of large conductivity depends not only on the ratio of conducting filler particles and inert particles sizes but also on the geometrical standard deviation of size distributions of two kinds of particles. The percolation threshold can be reduced by increasing the ratio of geometrical standard deviation of inert particles relative to that of the conducting fillers. The previous models have been proved to be special cases of our model for monosized distribution of both fillers and inert particles. Here we report the experimental observation of the electrical conduction development of ECA to verify the predictions of our model. A number of samples of conducting filler and inert particles with definitive size distribution were used. The electrical conductivity was measured by firstly mixing various fractions of fillers and inert particles and then compressed in the pellet form to remove all pores between particles. The measurement was performed by a simple method by placing electrodes at two pellet sides and reading the I-V characteristic of DC current.

11:15 AM
Effect of Particle Size Distribution on the Percolation Threshold of Electrically Conductive Adhesives: *Hong Zhou*[1]; H. K. Kim[1]; Hongki Lee[1]; Frank Shi[1]; [1]University of California, Irvine, Chem. and Biochem. Eng. and Matls. Sci. Dept., Irvine, CA 92697-2757 USA

Electrically conductive adhesives have been found more and more microelectronic applications. The purpose of this work is to investigate the effect of filler particle size distribution on the electrical percolation threshold of electrically conductive adhesives. Experimental results on the effect of mean fille particle size, the geometric standard deviations have been obtained. The experimental observations are explained in terms of the theoretical models.

11:30 AM Invited
Synthesis and Properties of Porous Polymer Thin Films having a Low Dielectric Constant: *Yuhuan Xu*[1]; Yi-Pin Tsai[1]; Dawei Zheng[1]; K. N. Tu[1]; Bin Zhao[2]; Q.-Z. Liu[2]; Maureen Brongo[2]; [1]University of California-Los Angeles, Dept. of Matls. Sci. and Eng., 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA; [2]Conexant Systems, 4311 Jamboree Rd., Newport Beach, CA 92660-3095 USA

We have succeeded in synthesizing a porous poly (arylethers) thin films by using the method of organic phase separation and evaporation. A solid organic material (Abetic acid) is mixed to the polymer solution, and in curing on a substrate the solute phase will evaporate from the solution and left behind numerous pores inside the rigid polymer film. A dielectric constant of 1.8 was achieved for a porous film with an estimated porosity of about 40%. The elastic modulus and thermal expansion coefficient of the porous films were measured by a combination of bending wafer and membrane budge test methods. A comparison between the dense and porous polymerfilms will be reported.

11:45 AM Invited
Experimental and Tribological Investigation of Cu Wear in CMP: *Hong Liang*[1]; [1]University of Alaska Fairbanks, Dept. of Mech. Eng., Fairbanks, AK 99775-5905 USA

In this work, we used surface analysis techniques, such as a field-emission high-resolution analytical TEM, X-ray spectroscopy, and XPS to analyze abrasive particles after polishing. Results showed evidence of copper oxide (Cu2O) in the polished slurry. However, there was no metallic crystalline copper detected. This finding was further investigated by estimating frictional force effects on copper passivation and polishing. A chemical wear mechanism was proposed.

Electron Backscatter Diffraction: Hardware and Software for EBSD - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Thin Films & Interfaces Committee
Program Organizers: Adam J. Schwartz, Lawrence Livermore National Laboratory, L-355, Livermore, CA 94550 USA; Mukul Kumar, Lawrence Livermore National Laboratory, L-356, CA, 94550 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Tuesday AM Room: Meramac
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Joseph R. Michael, Sandia National Laboratories, Albuquerque, NM 87185-1405 USA; Valerie Randle, University of Wales Swansea, Dept. of Matls. Eng., Swansea, UK

8:30 AM Invited
Automated Electron Backscatter Diffraction: *Robert A. Schwarzer*[1]; [1]Physics Institute, Physikalisches Institut der TU, AG Textur, Grosser Bruch, Clausthal-Z 38678 Germany

A noticeable advancement in spatially resolved diffraction analysis has been made by the development of automated EBSD with backscatter Kikuchi Patterns in the SEM. The principal objectives are a quantitative representation of the microstructure on a grain-specific level by the determination of crystal orientations, misorientations, the character of grain boundaries, or derived entities and stereological data. They are commonly depicted in pseudo-colors on the scanning grid to form crystal orientation maps, COM, or “orientation images” of the microstructure. For digital beam scan, dynamic focusing of the SEM as well as dynamic pattern calibration are indispensable. If intragranular structure is not considered, mesh refinement may be used to speed up measurement. At present 25,000 grain orientations or more are acquired per hour with a modern system. The limitations of video-based acquisition of diffraction patterns and upcoming developments are discussed.

9:00 AM Invited
EBSD: Buying a System: *Alwyn Eades*[1]; [1]Lehigh University, Matls. Sci. and Eng. Depts., 5 E. Packer Ave., Bethlehem, PA 18015-3195 USA

There is no EBSD system suited to all applications. In choosing a system, comprise is necessary. EBSD is now an established technique. There are at least four manufacturers who offer commercial systems to fit almost any SEM. Since it will soon be the case that an SEM used for physical sciences and engineering applications will be considered deficient if it lacks an EBSD system, many microscopists (and managers) should now be considering which system to buy. This paper will discuss some of the criteria which should be taken into account when selecting a system. No one system will serve all users well. A potential buyer must decide which things are of most importance (and where, therefore, they will be most demanding), and which things are less critical where compromise is possible.

9:20 AM Invited
Hardware and Software Optimization for Orientation Mapping and Phase Identification: *Patrick P. Camus*[1]; [1]NORAN Instruments, Inc., 2551 W. Beltline Hwy., Middleton, WI 53562 USA

The advent of high-resolution, high-sensitivity CCD cameras supplied with fast computers and software algorithms has permitted the routine collection and measurement of EBSD patterns for microcharacterization. Modern camera technology is required for easy acquisition of low intensity patterns and fast pattern analysis makes real-time acquisitions possible. Once the pattern is collected and the Kikuchi bands are identified, there are at least two analyses possible: orientation mapping of known crystal(s) and phase identification of unknown crystals. Although both analyses use EBSD patterns, it has been found that both the hardware and software should be optimized for these different tasks. This chapter will discuss the optimizations deemed necessary for optimal EBSD performance.

9:40 AM Invited
Advanced Software Capabilities for EBSD: *Stuart I. Wright*[1]; David P. Field[1]; David J. Dingley[1]; [1]TSL/EDAX, 392 E. 12300 South, Ste. H, Draper, UT 84020 USA

In conventional metallographic imaging, contrast is often produced by differences in crystallographic orientation. For instance, topological contrast can be formed by applying an etchant that preferentially attacks grain boundaries or attacks grains of different orientation at different rates. The contrast in images formed by polarized light in an optical microscope or channeling contrast in a scanning electron microscope is due to crystallographic orientation. However, quantitative orientation information is difficult to extract from these types of images and is thus usually excluded in standard metallographic analyses. However, with the advent of automated EBSD, quantitative information on crystallographic orientation can now be obtained at the microstructural scale. In automated EBSD, images can be formed by mapping of orientation data. While these images provide a useful visualization of the orientation aspects of a microstructure, the orientation data can also be used to form statistical and quantitative descriptions of the orientation aspects of the microstructure as well. This presentation will highlight a few of the current capabilities of automated EBSD software. These features are described in the context of application to real world materials problems.

10:00 AM Invited
Automated EBSD Acquistion and Processing System: *Pierre Rolland*[1]; [1]Oxford Instruments Analytical, Halifax Rd., High Wycombe-Bucks HP12 3SE UK

Abstract text unavailable.

10:20 AM Break

10:35 AM Invited
Strategies for Analysis of EBSD Datasets: *Wayne E. King*[1]; James S. Stölken[1]; Mukul Kumar[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, Chem. and Matls. Sci. Direct., L-356, 7000 E. Ave., Livermore, CA 94550 USA

Abstract text unavailable.

11:05 AM Invited
Fractography and Calibrated Serial Sectioning Linked to Electron Backscatter Diffraction: *Valerie Randle*[1]; [1]University of Wales Swansea, Matls. Eng. Dept., Singleton Park, Swansea SA2 8PP UK

Both metallographic serial sectioning and use of fractography in the scanning electron microscope (SEM) are established techniques for analysis of microstructure, providing more information than from a single polished section. When coupled to electron backscatter diffraction (EBSD) these techniques become especially powerful because crystallographic analysis of flat surfaces and facets, such as grain boundaries, microcracks and cleavage fractures, becomes available. In this paper the various investigation methodologies for obtaining combined EBSD/surface information will be described, followed by two examples from ongoing experimental programmes. The first of these examples relates to the analysis of grain boundary planes in brass and a nickel-based superalloy, and how the parameters thus derived relate to material properties. The second example illustrates crystallogaphic analysis of fracture surfaces, comprising regions of cleavage fracture and intergranular accommodation surfaces.

11:35 AM Invited
Three Dimensional Orientation Imaging: *Dorte Juul Jensen*[1]; [1]Riso National Laboratory, Matls. Dept., Roskilde DK4000 Denmark

A limitation of the EBSD technique is that it is limited to two dimensions. 3D information is typically achieved by time consumining, labor intensive and often problematic serial sectioning. In the present paper a new method is presented which allows non-destructive characterization of crystallographic orientations in 3 dimensions. The method is based on high energy synchrotron radiation, and a newly developed so-called 3D X-ray diffraction microscope is described. The potentials of the microscope are illustrated by examples on i) in-situ investigations of recrystallization and ii) non-destructive characterization of 3D grain structures.

General Abstracts: Chemistry & Physics of Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Tuesday AM Room: Jefferson A
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Katherine Chen,California Polytechnic State University, San Luis Obispo, CA 93407 USA

8:30 AM
Extracting Grain Boundary Properties from Microstructural Information: *Anthony D. Rollett*[1]; Chih-Chao Yang[1]; William W. Mullins[1]; [1]Carnegie Mellon University, Depts. of Matls. Sci. and Eng., Wean Hall 3327, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The Mesoscale Interface Mapping Project at CMU is engaged in determining maps of grain boundary properties based on their crystallographic type. Maps of the variation of grain boundary energy and mobility will be invaluable in understanding microstructural evolution in processes such as grain growth and recrystallization. By developing novel approaches to imaging, EBSD indexing, image analysis and image registration, it is now possible to obtain large data sets for statistical analysis. The focus is on measuring boundary character, dihedral angles and curvature at triple junctions with serial sectioning to determine the inclinations of the triple junctions. Current work is focused on obtaining improving the resolution of the energy maps by increasing the size of the data set. In addition, the first results have been obtained for curvature at triple junctions in a thin aluminum foil. The columnar structure observed obviates the need for serial sectioning thereby reducing errors in curvature measurement. Image analysis is performed on digitized micrographs to obtain "skeletonized" images with only pixels adjacent to boundaries highlighted. Conic sections are fitted to each boundary from which tangents and curvatures at the triple junction are extracted. The strong cube texture restricts the range of boundary character that needs to be taken into account in the analysis. Based on the relationships between curvature driven grain boundary migration, and the normal velocities of boundaries at a triple junction, maps of (relative) mobility are constructed. Examples will also be shown of incorporating anisotropic boundary properties into the Potts model for grain growth.

8:50 AM +
Directional Recrystallization of Cold-Rolled Copper Single Crystal: *Jiying Li*[1]; Ian Baker[1]; Harold J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA

High-purity (99.999%) copper single crystals with different orientations have been cold-rolled to reductions in the range from 20% to 100%. In each case, calorimetry (DSC) was used to determine the recrystallization temperature and stored energy which characterized the initial deformed microstructure. A modified optical-image furnace was used to directionally-recrystallize the cold-rolled Cu at different temperatures with hot zone movement rates varied from 1mm/h to 200mm/h. For polycrystals, the resulting grain size was determined; for single crystals, the resulting orientation was determined by X-ray diffraction; and for columnar-grained structures, electron back scatter patterns (EBSP) were used to determine grain orientation. The dependence of the resulting microstructure on the processing conditions was simulated by a front -tracking algorithm technique. This research is funded by the National Science Foundation grant DMI-9976509.

9:10 AM
Molecular Polarizability of Semiconductor Clusters and Nanostructures: *Francisco Torrens*[1]; [1]Universitat de Valencia, Dept. de Quimica Fisica, Dr. Moliner-50, Burjassot, Valencia E-46100 Spain

The interacting induced dipoles polarization model implemented in program PAPID is used for the calculation of the molecular dipole-dipole polarizability ALPHA. The method is tested with (Si)n, (Ge)n and (Ga)n(As)m small clusters. On varying the number of atoms, the clusters show numbers indicative of particularly polarizable structures. The results

for the polarizability are in agreement with reference calculations carried out within the density functional theory. The bulk limit for the polarizability is estimated from the Clausius-Mossotti relationship. The clusters are all more polarizable than what one might have inferred from the bulk polarizability. Previous experimental work have yielded the opposite trend for somewhat larger clusters. At present, the origin of this difference is problematic. One might argue that smaller clusters need not behave like those of intermediate size. The high polarizability of small clusters is attributed to arise from dangling bonds at the surface of the cluster.

9:30 AM Break

9:50 AM
Shape Bifurcations in Equilibrium Shapes of Coherent Precipitates in Systems with Cubic Elastic Constants: *Sankarasubramanian Ramachandran*[1]; Chandrashekhar S. Jog[2]; Abinandanan A. Thennathur[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore, Karnataka 560 012 India; [2]Indian Institute of Science, Dept. of Mech. Eng., Bangalore, Karnataka 560 012 India

Motivated by the shape bifurcation study of Johnson and Cahn, on the shapes of coherent precipitates with isotropic elastic moduli and dilatational misfit, we have explored systems with non-dilatational misfit and cubic elastic moduli. We define the equilibrium shape as that shape which minimizes the sum of the elastic and interfacial energies. We have used a combination of a finite element method and an optimization program, to find out the equilibrium shape. We have found symmetry-breaking shape transitions with break-down of rotational symmetry and loss of mirror symmetries depending on the kind of misfit and elastic anisotropy. We have also found a criterion for continuous and discontinuous transitions.

10:10 AM
Physical Processes in Laser Assisted Al-Foaming: *Yash P. Kathuria*[1]; [1]Laser X Company Limited, 7 Kofukada, Shinbayashi-cho, Chiryu-shi, Aichi-ken 472-0017 Japan

Aluminium is one of the most commonly used metal in various domain of applications. In the automobile industries, it has gained a solid ground due to its recyclability, good corrosion resistance, low weight and the energy saving benefits. Recently aluminium foams have evoked a special interest as a new alternative material due to its wide range of applications ranging from microelectronics and automobiles to aerospace industries. But the manufacturing techniques and characterization methods needs more attention for their effective and economical use in the industries. Various methods including the casting and powder metallurgy techniques have been used, which employ the conventional molding and thermal heating to produce the Al-foam. But here an alternative process of using laser as a heat source shall be described. It has an inherent advantage of unidirectional and localized foaming. Besides that the process is much faster, due to the rapid solidification process, where the stabilization of pore formation could partly be controlled by co-axial gas flow and the laser processing parameters. It is produced by mixing powdered material (AlSi7) and a foaming agent (TiH2) and subsequently cold isostatic pressing the mixture to a foamable sandwich precursor material. The material is foamed by heating it up to its melting point by a high power laser beam irradiation. The unidirectional expansion of the foamable precursor material can be observed during the entire foaming process in the irradiation direction with different scale of porosity. It is believed that the stabilization of pores formation in foaming process occurs during the solidification process. The possible reasons are: 1. Dissociation of the hydrogen gas from the foaming agent TiH2 which bubbles out leading to the formation of foam as a macroscale porosity but in the microscale domain it get trapped in the solidified foamed metal due to its 20-times solubility difference in liquid and solid aluminium. 2. Possibility of the blowing or shielding argon gas that helps in foaming process may also gets trapped in the foamed metal. The objective is to gain insight into the alternative laser process in the expanding melt and to optimise the process parameters with respect to foam quality and process stability.

10:30 AM +
Solidification of Galvanized Layer by Electrostatically Charged Aerosol Particles: *Kim Sangheon*[1]; [1]Pohang Iron and Steel Company Limited, Tech. Rsch. Labs., 699, Kumho-Dong, Kwangyang-Shi, Cheonnam 545-090 ROK

A novel electrostatic spraying method for solidifying galvanized coating layers was studied. The sprayed droplets also served as nucleation sites but might leave the pitting mark by impinging on the melted coating layer in the conventional methods. Our computer simulations showed that the electric field could change the sprayed particle trajectories especially in the space near the steel surface and assist the fines to attach on the surface. And owing to the phenomena so called electro-hydrodynamic atomization, large particles breaks themself down into small ones with the applied high voltage such as 40 KV. Thus, by spraying the solution in low pressure as 1.2~1.5 kg/cm2 within the electric field, we could produce the spangle free coating layer without pitting mark even with antimony added zinc bath.

10:50 AM
Finite Difference Model to Predict Carbon Diffusion and Precipitation in Nickel-Based Super Alloys: *T. Madhavi Latha*[1]; S. N. Malhotra[2]; N. K. Khosla[2]; [1]Indian Institute of Technology/Regional Engineering College, Dept. of Metall. Eng. and Matls. Sci., Bombay 400 076 India; [2]Indian Institute of Technology, Bombay 400 076 India

Carburization of nickel based and iron-nickel based super alloys is often observed in carbon containing industrial atmospheres. Over-all phenomena of carburization is a complex gas-metal interaction process involving diffusion and precipitation of carbon from the surrounding atmosphere to the alloy matrix. A reliable estimation of carbon diffusion and precipitation can be made theoretically by solving Fick's second law of diffusion using finite difference method. Precipitation of two carbides, namely $Cr_{23}C_6$ and Cr_7C_3, is taken into account in solving the diffusion equation in the present work. An implicit method was used for better convergence of the solution. The model developed calculates carbon concentration vs distance profiles in the alloy matrix as a function of carburization time at constant temperature and carbon activity of the gaseous atmosphere. Carbon diffusion into the alloy matrix was based on thermodynamic and kinetic parameters such as diffusion coefficient of carbon, solubility products of carbides, activity coefficients of carbon etc. These are composition dependent parameters and were evaluated using appropriate experimental and theoretical methods. The model is applied to Nimonic-80A, Inconel 600 and Incoloy 800H alloys at a temperature of 1173 K and carbon activity value of 0.7. A good agreement is observed between the theoretically calculated and experimentally obtained carbon concentration profiles

Materials Issues in Nuclear Waste Management: Performance Testing of Waste Form and Waste Package Materials - II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Nuclear Materials Committee
Program Organizers: Thad M. Adams, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Robert Sindelar, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Patrick R. Taylor, University of Idaho, Department of Metals & Mining Engineering, Moscow, ID 83843-3024

Tuesday AM Room: Clark
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Thad M. Adams, Westinghouse Savannah River Company, Savannah River Technology Center, Aiken, SC 29808 USA

8:30 AM Opening Remarks: Dr. Thad M. Adams

8:35 AM Invited
Corrosion Testing of Spent Nuclear Fuel Performed at Argonne National Laboratory for Repository Acceptance: *Margaret M. Goldberg*[1]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

Corrosion tests of spent nuclear fuel are performed at Argonne National Laboratory to support the license application for the Yucca Mountain Repository. The tests are designed to determine corrosion rates and degradation products formed when fuel is reacted at elevated temperature in different aqueous environments, including vapor, dripping water, submersion, and liquid film contact. Corrosion rates are determined from the quantity of radionuclides released from wetted fuel and from the weight loss of the test fuel specimen as a function of time. Degradation products include secondary mineral phases and dissolved, adsorbed, and colloidal species. Solid phase examinations determine fuel/mineral interface relationships, characterize radionuclide incorporation into secondary phases, and determine corrosion mechanisms at grain interfaces within the fuel. Leachate solution analyses quantify released radionuclides and determine the size and charge distribution of colloids. This paper presents selected results from corrosion tests on ceramic oxide and metallic fuels with emphasis on the metallic uranium fuel.

9:10 AM
Baseline Characterization of Neutron Absorber Alloying Behavior in the Melt-Dilute Al-SNF Form: *D. W. Vinson*[1]; T. M. Adams[1]; R. L. Sindelar[1]; [1]Westinghouse Savannah River Company, Savannah River Tech. Ctr., Aiken, SC 29808 USA

Abstract text unavailable.

9:40 AM

Dissolution-Rate Testing of Unirradiated N-Reactor Fuel: *Walter J. Gray*[1]; Dennis W. Wester[1]; Colleen Shelton-Davis[2]; [1]Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352 USA; [2]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID 83415 USA

Initial results from dissolution-rate testing of Hanford N-Reactor spent nuclear fuel (HSNF) over a range of potential geologic repository aqueous conditions revealed that HSNF can corrode at a very rapid rate, called Stage 2 corrosion, producing a sooty black solid on the specimen surface. In order to examine possible causes of this phenomenon, the dissolution rate of unirradiated N-Reactor fuel was measured under similar conditions. Thus, dissolution rates were measured for fuel specimens in $NaHCO_3$ (0.02M, pH 8), HNO_3 (10^{-3}M), de-ionized water, and simulated J-13 water (a well near Yucca Mountain). Specimens were placed in flow-through columns with water flowing up. Effluents from the columns were analyzed for U content. The dissolution rates and the condition of the specimens in the above solutions indicated that Stage 2 corrosion occurred in some instances. The results will be discussed with respect to H_2 evolution and oxic and anoxic modes of U dissolution.

10:00 AM

Colloid Generation from Metallic Uranium Fuel: *Carol J. Mertz*[1]; Jeffrey A. Fortner[1]; Margaret M. Goldberg[1]; Colleen V. Shelton-Davis[2]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Idaho National Engineering and Environmental Laboratory, Nat. Spent Nuc. Fuel Pgm., 1955 North Fremont Ave., Idaho Falls, ID 83415 USA

Potential generation of colloids by corrosion of spent nuclear fuel in an unsaturated environment has significant implications for storage of these fuels in the proposed repository at Yucca Mountain. Because colloids may be a transport medium for sparingly-soluble radionuclides, it might be possible for colloid-associated radionuclides to migrate large distances underground and present a human health concern. This study examines colloidal materials produced during corrosion of unirradiated metallic uranium fuel in simulated groundwater at elevated temperature in an unsaturated environment. X-Ray powder diffraction of corrosion products spalled from the fuel indicates the presence of a series of uranium oxides, hydroxides, and silicates. The predominant reaction product is UO_2 with a diffracting unit dimension of approximately 10 nm. Scanning electron microscopy reveals that the colloidal-sized UO_2 particles aggregate into larger particles with dimensions of <1 μm to >10 μm. The distribution of colloidal particle sizes by dynamic light scattering will be presented.

10:20 AM Break

10:30 AM

Influence of Thermal Aging on the Mechanical and Corrosion Properties of C-22 Alloy Welds: *Tammy S. Edgecumbe-Summers*[1]; Raul B. Rebak[2]; [1]Lawrence Livermore National Laboratory, Mail Stop L-638 7000 E. Ave., P.O. Box 808, Livermore, CA 94551 USA; [2]Haynes International, Inc., 1020 W. Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

A nickel-base alloy containing high concentrations of chromium (Cr) and molybdenum (Mo), C-22 alloy (UNS #N06022), is a candidate material for high-level radioactive waste disposal containers in the potential repository at Yucca Mountain. Heat generated by radioactive decay of this waste is estimated to raise the temperature of the waste packages to as high as 200-400°C depending on design of the repository. Although these temperatures are fairly low, the desired container lifetime is at least 10,000 years. Because of the long time required for container integrity, phase stability of N06022 base metal has recently received a lot of attention. The welds required in fabrication of these disposal containers, especially the closure weld which can receive no post weld heat treatments that may adversely affect the waste contained within, are also of interest. N06022 alloy welds have a segregated microstructure typical of welds. Also, Tetrahedrally Close Packed (TCP) phases such as P, μ, σ, which are known to degrade the corrosion resistance of the base metal, form in these welds during solidification. Relatively little is known about the mechanical and corrosion properties of N06022 alloy welds and the influence of aging on them. In order to estimate the stability of the microstructure of N06022 alloy welds, Gas Tungsten Arc Welded (GTAW) samples made with a Single V notch geometry and matching filler metal were aged at 430, 590, 650, 700 and 760°C for several times up to 40,000 hours. The uniaxial tension properties, Charpy impact toughness, and the corrosion properties in aggressive reducing and oxidizing acids of both aged and unaged weld samples were measured and correlated to microstructural observations made with Scanning Electron Microscopy (SEM). These data were used for a preliminary estimation of the stability of the segregated microstructure and TCP phases of N06022 alloy welds. Parts of this work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48. The work done at LLNL is supported by Yucca Mountain Site Characterization Project, LLNL.

10:50 AM

Non-Equilibrium Thermodynamic Dissolution Theory for Multi-Component Solid/Liquid Surfaces Involving Surface Adsorption and Radiolysis Kinetics: *Ray B. Stout*[1]; [1]University of California/ LLNL, P.O. Box 808, L-631, Livermore, CA 94550 USA

Multi-component spent nuclear fuels (SNFs) are being evaluated for geological disposition with compositions containing a wide range of fission product and actinide concentrations which decay by alpha, beta, and gamma radiation. The subset of SNFs emplaced in a geologic repository will potentially be wetted by multi-component aqueous solutions, which are potentially altered by radiolytic aqueous species due to radiation. The steady-state concentrations of radiolytic chemical species are analyzed only approximately. The solid states of the SNFs are not thermodynamically stable and will dissolve when wetted, with or without radiolysis. For this reason, a theoretical expression is developed for the dissolution rate response of a multi-component solid that has surface adsorption kinetics and radiolysis kinetics when wetted by a multi-component aqueous solution. The theory is a non-equilibrium thermodynamic analysis across a solid/liquid phase change discontinuity that propagates at a quasi-steady velocity. The energy equation contains the internal energy functionals of classical thermodynamics for both the SNFs' solid state, the adjacent liquid state, and the radiolytic chemical species. The irreversible production of configurational entropy across the solid-liquid phase change discontinuity is assumed proportional to the propagation velocity of the solid-liquid interface. For purposes of illustration a modified Temkin adsorption isotherm was assumed for the surface adsorption kinetics. For idealized dissolution processes that depend on surface adsorption and radiolysis kinetics, approximations are made to obtain a theoretical rate expression for empirical and/or regression analysis of data. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48. This work is supported by Yucca Mountain Site Characterization Project, LLNL.

11:10 AM

Volatilization of Fission Products from UAl_x Melt-Dilute Tests: *T. S. Bray*[1]; A. B. Cohen[1]; R. V. Strain[1]; H. Tsai[1]; T. M. Adams[2]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Westinghouse Savannah River Company, Aiken, SC 29808 USA

Savannah River (SRL) is the interim storage site for aluminum clad fuel elements and is determining whether to store the fuel elements as-received or to treat them before storage. Treatment involves a "melt-dilution" process where the alloy is melted and the enriched uranium diluted. The majority of the elements have U-Al fuel or UAl_x dispersion fuels. Argonne National Laboratory (ANL) conducted three irradiated UAl_x material melt dilution tests; the purpose of the tests is to determine whether irradiated fuel presents issues not evident in tests using surrogate fission products. The specific issues to be addressed are 1) the amount of cesium release at a melt-dilution temperature of 850°C, 2) the ability to trap the cesium on a filter medium such as activated alumina or Zeolite, and 3) the release and entrapment of other fission products. Results show released cesium became trapped on filters; however, the tests also showed incomplete melting of the specimens.

11:30 AM

Materials Compatibility Testing for a Pilot-Scale Oxide Reduction System: *Terry C. Totemeier*[1]; Steven D. Herrmann[1]; [1]Argonne National Laboratory-West, P.O. Box 2528, Idaho Falls, ID 83403 USA

Materials compatibility tests were performed on five candidate steels for the pilot-scale oxide reduction system currently under development at Argonne National Laboratory West. Three stainless steels (types 316L, 304, 347) and two alloy steels (2.25Cr-1Mo and 9Cr-1Mo-V) were evaluated. Small, swagelok-sealed capsules constructed from each steel were loaded with a LiCl salt solution and exposed at 725°C for 30, 60, and 90 days. The LiCl salt solution contained 3.5 wt% Li2O, 1 wt% Li3N, and was saturated with Li metal. The extent of corrosion was assessed following exposure by destructive metallographic examination of each capsule. The extent of corrosion observed varied widely, from essentially none to full penetration of the capsule wall. The primary form of corrosion for all steels was grain boundary porosity and depletion of Cr from the exposed surface.

11:50 AM

Development of Gadolinium Containing Stainless Steels for Nuclear Criticality Control: *J. N. Dupont*[1]; C. V. Robino[1]; R. E. Mizia[1]; [1]Lehigh University, Dept. of Matls. Sci. and Eng., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Abstract text unavailable.

Rate Processes in Plastic Deformation II: Towards an Unified Theory of Deformation - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA; Thomas R. Bieler, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; Bimal Kad, University of California, AMES Laboratory, La Jolla, CA 92093-0411 USA; Farghalli A. Mohamed, University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697 USA

Tuesday AM Room: Lewis
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Dr. Peter M. Hazzeldine, UES, Dayton, OH 45432 USA; Prof. Erik Nes, Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Trondheim, Norway

Thermally Activated Deformation Processes - II

8:30 AM Keynote
Thermally Activated Plastic Flow of Metals and Ceramics with an Electric Field or Current: *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. and Eng. Depts., P.O. Box 7909, Raleigh, NC 27695-7907 USA

The affects of an external (electrostatic) electric field and of high density electric current pulses on the plastic deformation kinetics of metals is presented. An external electric field applied during the superplastic deformation of Al alloys reduced the flow stress through a reduction in activation energy, but more importantly it retarded cavitation and grain growth. The influence of the field appears to be on the migration of charged vacancies or solute atom-vacancy complexes along grain boundaries to the surface. The major effect of a high density current pulse at low and intermediate temperatures was to reduce the thermal component of the flow stress. This resulted from the combined action of: (a) an electron wind force, (b) a decrease in activation enthalpy and an increase in the pre-exponential, the last making the largest contribution. In the case of ceramics, an internal electric field in excess of 10 kV/cm enhanced dislocation mobility in halide single crystals at room temperature. However, a field of only 1 kV/cm reduced the flow stress of NaCl polycrystals at low and intermediate temperatures, which is considered to result from an enhancement of cross slip. At high temperatures a field E ¾0.3 kV/cm reduced the flow stress and generally increased elongation. This resulted mainly from an increase in the controlling diffusion rate.

9:00 AM Invited
Dislocation Movements Controlled by Friction Forces and Local Pinning in Metals and Alloys: *Daniel Caillard*[1]; A. Couret[1]; [1]CNRS, CEMES, 29 Rue J. Marvig, BP 4347, Toulouse, Cedex 31055 France

The plasticity of materials is usually controlled by a small number of elementary dislocation mechanisms, that may however play different roles in different materials. These mechanisms will be described and illustrated by dynamic recordings of in situ experiments in a TEM. Peierls friction forces will be described in pure metals (Mg, Be, Ti) and in intermetallic alloys Ni3Al, Ti3Al). Different mechanisms can operate to overcome the Peierls potential due to the non-planar spreading of dislocation cores: the kink-pair mechanism, and the cross-slip (or locking-unlocking) mechanism. A transition between these two mechanisms can account for the discontinuities in the activation volumes versus stress and temperature curves measured in Ti, and for the formation of either complete or incomplete Kear-Wilsdorf locks in Ni3Al. Interactions between dislocations and fixed atomic-size obstacles will be described in NiAl and Ti3Al (deformed in pyramidal slip). Interactions between dislocations and mobile solute atoms accounts for dynamic strain ageing and plastic instabilities in several intermetallics (e.g. Fe3Al). Lastly, pure climb can also play an important role at high temperatures, e.g. in AlPdMn quasicrystals.

9:20 AM Invited
Characterization of Thermally Activated Deformation Mechanisms Using Transient Tests: *J. L. Martin*[1]; B. Lo Piccolo[1]; T. Kruml[1]; J. Bonneville[1]; [1]Ecole Polytechnique Federale, Phys. Dept., Lausanne 1015 Switzerland

The identification of dislocation mechanisms as the crystal deforms requires determining separately the dislocation velocity v and the mobile dislocation density. Both parameters contribute to the strain-rate according to the Orowan equation. Conventional mechanical tests provide access to the latter parameter, but do not allow the separation of v and the mobile density, consequently the mobile density is a poorly documented parameter(1). We have developed through the years two types of transient tests, namely repeated relaxation and repeated creep experiments, which have the following advantages. They provide information about the actual dependence of the dislocation velocity on stress through the activation volume, while single transients yield apparent volumes only. It will be shown that by comparing both volumes, a structural parameter can be defined which quantifies the substructure evolution during straining. From this parameter useful information can be obtained about the work-hardening coefficient and the mobile dislocation exhaustion rate. The equations which describe the transients are presented together with the experimental assessments of the assumptions used, for a selection of materials. These include Ni_3Al and TiAl (2) compounds (both exhibit a strength anomaly and high work-hardening coefficients) and Cu (thermally activated deformation and moderate work-hardening coefficient). It will be shown, by comparing both types of materials, that the higher the work-hardening coefficient, the larger the dislocation exhaustion rate (3). These results will be discussed in terms of the exhaustion mechanisms typical of each of these materials. (1) Martin, J.L and Caillard, D.:"Thermally activated mechanisms in crystal plasticity," book in preparation for Elsevier-Pergamon Materials Series.(2000). (2)Viguier, B.,Bonneville, J. and Martin, J.L., Acta Mater. 44(1996)4403. (3)Kruml,T., Martin,J.L. and Bonneville,J Phil.Mag., accepted for publication(1999).

9:40 AM Invited
Toward Physically-Based Rate Laws for Dislocations: *John J. Gilman*[1]; [1]University of California at Los Angeles, Matls. Sci. and Eng. Depts., 6532 Boelter Hall, Los Angeles, CA 90095 USA

Rate laws of the Becker type are inappropriate for dislocations. They were developed for independent particles, not for lines where the motions are highly correlated. Also, since one end of a line cannot communicate with the other end faster than sound speed will allow, there are serious correlation problems when the activation area is considered to be larger than a few atomic squares; for example, with kink nucleation theory. Without arbitrary modifications this theory does not describe the observed temperature dependence of flow stresses satisfactorily. It will be shown how the intrinsically one-dimensional Becker-type theory can be combined with two-dimensional theory to yield a physically consistent model that describes the temperature dependence of plastic flow stresses satisfactorily without being arbitrary.

10:00 AM Break

Dislocation Substructure Modeling of Materials - I

10:15 AM Keynote
Rate Equation Approach to Dislocation Dynamics and Plastic Deformation: *Daniel J. Walgraef*[1]; [1]Free University of Brussels, CENOLI, Campus Plaine-CP 231, Bd. du Triomphe, Brussels B-1050 Belgium

Strain localization and dislocation microstructure formation are typical features of plastic deformation in metals and alloys. Plastic deformation occurs by the glide of dislocations, and, although dislocation distributions are rather uniform at its onset, they usually become unstable when deformation proceeds and undergo successive transitions towards various types of microstructures such as cells, deformation bands, persistent slip bands, labyrinth structures, etc. This phenomenon is experimentally well documented, but, despite a huge number of theoretical investigations, its modeling is still in its infancy. In the spirit of multiscale modeling, an intermediate step between micro- and macroscopic descriptions, consists in the derivation of mesoscopic rate equations describing the dynamical evolution of dislocation densities. It will be shown on some classical examples why this approach is interesting for the description of mechanical systems undergoing plastic instabilities, and how it may be used to determine the key physical processes for dislocation patterning. It will also be shown that the localization of plastic deformation is a natural consequence of collective behaviors induced by reaction and transport in dislocation populations.

10:45 AM Keynote
Revisiting the Internal Variable Theory: *Elias C. Aifantis*[1]; [1]Aristotle University of Thessaloniki, Polytech. Sch., Lab. of Mech. and Matls., Thessaloniki GR-54006 Greece

Internal variable theory has been used extensively to model rate processes in plastic deformation. A set of internal variables is introduced to model the evolution of underlying microstructure under applied loads. The evolution of the internal variables is commonly modeled by deterministic ordinary differential equations which are coupled with the evolution equation for the plastic strain. A variety of inelasticity phenomena are de-

scribed by this way. Two new elements are proposed now to introduce in this approach: (i) Stochastic considerations for the evolution of the internal variables. (ii) Gradient terms of diffusion type in the evolution equations for the internal variables. The advantages of each approach are discussed. Their implications to interpreting patterning phenomena are considered.

11:15 AM Invited
Constitutive Description of Dynamic Deformation: Physically-Based Mechanisms: *Marc Andre Meyers*[1]; David J. Benson[1]; Vitali F. Nesterenko[1]; Qing Xue[1]; Hsueh-Hung Fu[1]; [1]University of California, Mail Code 0411, Dept. of MAE, La Jolla, CA 92093 USA

The response of metals to high-strain-rate deformation is successfully described by physicallly-based mechanisms which incorporate dislocation dynamics, twinning, displacive (martensitic) phase transformations, grain-size, stacking-fault, and solution hardening effects. Several constitutive equations for slip have emerged, the most notable being the Zerilli-Armstrong and MTS. They are based on Becker's and Seeger's concepts of overcoming obstacles through thermal activation. The temperature and strain-rate sensitivity for twinning are lower than for slip; on the other hand, its Hall-Petch slope is higher. Thus, the strain rate affects the dominating deformation mechanisms in a significant manner, which can be quantitatively described. These equations are incorporated into computational simulations of plastic deformation at high strain rates. Other effects of dynamic deformation are shear localization and wave propagation. They can also be quantitatively described. In particular, shear-band spacing has been experimentally obtained and modelled. In wave propagation, the time available for plastic deformation can be reduced to the nanosecond domain. Research funded by the U.S. Army Research Office and Office of Naval Research.

11:35 AM
The Possible Role of Point Defects during Plastic Deformation of IF Steel: *Shig Saimoto*[1]; Bradley J. Diak[1]; [1]Queen's University, Matls. and Metall. Eng. Depts., Nicol Hall, Kingston, Ontario K7L3N6 Canada

The generation of point defects during plastic deformation of crystalline solids has been one of the early discoveries in the studies of plastic flow. However, determining its precise effect on the dynamic microstructural evolution under the available energy conditions has been elusive. The reasons for this difficulty are that quasi-static examinations using resistivity, stored work and transmission electron metallography can not capture the dynamic reactions of thermally activated flow. Moreover, the deformation products are not only point defects but also a wide variety of debris, the simplest of which are dipole loops. Our past studies have shown that the retention of the debris is highly dependent on the solute content in the matrix of specific pinning solutes, for example Fe in Al or Cu. The effect of these solute atoms above a few ppm can be readily manifested during precision strain rate sensitivity (PSRS) tests. Our new discovery shows that the level of these pinning solutes can be lowered below 1.0 ppm by selective gettering at low temperatures where its solubility becomes very small. In this situation the debris grows dynamically such that its thermal barrier becomes very similar to that of monopole dislocations and the deviation from the Cottrell-Stokes relation disappears. In such high purity matrix systems, dynamic and/or continuous recrystallization can be induced to result in grain sizes below 2 micrometers. In the deformation of such systems, the up change of the strain rate is not identical to the down change. Moreover, the mean slip distance can be quantitatively assessed from the bulk tensile parameters and correlated to the measured activation volume. How such studies elucidate the role of dynamic dislocation annihilation in the composite work hardening theory of Mughrabi will be described.

State of the Art in Cast MMC's: Structure Property Relationships - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing and Manufacturing Division, Jt. Composite Materials Committee, Solidification Committee
Program Organizer: Pradeep Rohatgi, University of Wisconsin, Materials Department, Milwaukee, WI 53211 USA

Tuesday AM Room: Laclade
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: TBA

8:30 AM
Mechanical Properties and Microstructure of Aluminum Matrix Composites Produced Using 3-Dimensional Printing of Ceramic Preforms and Pressure Infiltration Casting: *M. L. Selemev*[1]; J. A. Comic[1]; S. Zhang[1]; A-O. Salvi[1]; [1]Metal Matrix Cast Composites, Inc., Waltham, MA USA

Three-dimensional printing (3DP) is a novel technology that is quickly establishing itself as not just a rapid prototyping tool but also as an extremely flexible manufacturing process. Novel tool-less pressure infiltration casting technology was developed which allows fabrication of a net-shape metal matrix composites using 3-dimensional printing for ceramic preform manufacturing. Technological process which involves 3D-printing of the preforms, sintering of the preforms, pressure infiltration of sintered preforms with liquid metal and heat treatment of the resulting composite was optimized to yield highest mechanical properties in the resulting material. Study describes the influence of the major technological factors as well as matrix alloy composition on mechanical properties and microstructure of the new 3DP metal matrix composites reinforced with alumina and silicon carbide particles.

8:50 AM
Optimized Processing of Al-SiCp Metal Matrix Composites by Pressureless Infiltration of SiCp Preforms: *M. I. Pech-Canul*[1]; M. M. Makhlouf[1]; R. N. Katz[1]; [1]Worcester Polytechnic Institute, Dept. of Mech. Eng., 100 Institute Rd., Box 2933, Worcester, MA 01609 USA

An optimum method for producing Al-SiCp metal matrix composites was developed by determining the optimum parameters for pressureless infiltration of SiCp preforms by aluminum. The effect of SiC particle size, infiltration time, preform height, vol. % SiC in preform, and Si coating on the SiC particles on the pressureless infiltration of SiCp compacts with aluminum was investigated and quantified. The contribution of each of these parameters and their interactions to the retained porosity in the composite, the modulus of elasticity, and the modulus of rupture were determined. The parameter that affects retained porosity in the composites most significantly is the surface condition of the SiC particles. The presence of a thin coating of silicon on the silicon carbide particles enhances the spreading of the alloy over the SiC particle surface and consequently, helps minimize porosity in the composite. The parameters that affect the modulus of elasticity of the composites most significantly are the infiltration time and the preform porosity. The interaction between infiltration time and preform height affects the modulus of rupture of the composites most significantly. Processes for obtaining optimum density, modulus of elasticity and modulus of rupture were projected. Al/SiC_p composites were produced using the conditions that were projected to produce optimum modulus of elasticity and their modulus of elasticity was evaluated. The results show excellent agreement between the projected and measured values. Under optimum infiltration conditions, MMCs with less than 3% porosity, over 200GPa modulus of elasticity, and about 300 Mpa modulus of rupture were routinely produced.

9:10 AM
The Thermal Shock Resistance of Particulate- and Short-Fiber Reinforced Cast Aluminum-Matrix Composites: *Jerzy Sobczak*[1]; Zenon Slawinski[2]; Natalia Sobczak[1]; Rajiv Asthana[3]; [1]Foundry Research Institute, Krakow, Poland; [2]Technical University of Lublin, 36 Nadbystrzycka St., Lublin 20-618 Poland; [3]University of Wisconsin-Stout, WI USA

The thermal shock resistance of several monolithic Al alloys and Al-matrix composites containing particulates of graphite, silicon carbide, and flyash, and short fibers of alumina was characterized by measuring the total length of all microcracks as a function of number of thermal cycles in a specified temperature range. During each temperature cycle, the test

specimens were heated and stabilized in air at 375°C, water quenched to room temperature, and air stabilized. A total of 1000 to 5000 thermal cycles were employed. The test specimens were fabricated using gravity casting in permanent molds or squeeze casting, followed by heat treatment (T6) of selected materials prior to thermal cycling. In all the tested materials, the total crack length increased with increasing number of thermal cycles. Squeeze cast and heat treated Al-matrix composites containing alumina short fibers exhibited the best thermal shock resistance. Among monolithic alloys, squeeze cast and heat treated Al-12SiCuNiMg alloy exhibited higher resistance to cracking during thermal cycling than Al-25Si and Al-2OSiNi alloys. On the basis of crack length measurements and microstructural observations of the tested materials, conceptual schemes were proposed for crack propagation behaviors in monolithic alloys and composites under thermal cycling conditions. A first approximation to predicting the thermal shock resistance of composites in terms of selected physical properties was also presented.

9:30 AM

Using Fly Ash Waste Material for the Synthesis of Light Weight Low Cost Aluminum Matrix Composites: *Natalia Sobczak*[1]; Jerzy Sobczak[1]; [1]Foundry Research Institute, 73 Zakopianska St., Krakow 30-418, Poland

Scientific, technological and economic problems related with the utilization of fly ash waste materials for the synthesis of composites have been discussed. Fly ash materials as the waste by-products, the very large amounts of which are produced mainly during combustion of coal by thermal power plants, present an important ecological problem associated with their storage and disposal. Therefore, the development of suitable scientific, technical and economic solutions of fly ash utilization is very pressing and important. Because of the complex of both physical and mechanical properties with respect to low price and low density, the fly ash can be an extremely attractive material applicable for the synthesis of composites. Generally, the technology of producing composites of the Al-fly ash system is difficult because of light weight of fly ash and its poor low temperature wettability in as-received state that leads to such unfavorable phenomena as flotation, agglomeration and segregation of fly ash particles and, finally, to their non-uniform distribution in a casting. The favorable structural changes driving in the direction of desirable structural homogeneity of composite castings and, therefore, remarkable improvement in their physical and mechanical properties have been stated in squeeze castings due to an application of external pressure during their solidification. Several composite materials of Al-fly ash system with wide density range of 1.00-2.68 g/cm_3 have been developed using liquid phase processing by means of different casting techniques. Introducing fly ash into aluminum alloys reduces their cost and improves their physical, mechanical and utility properties. The new aluminum matrix composite, characterized by mechanical properties and coefficient of thermal expansion similar to thos of the well-known F3N.20S DURALCAN composite, has been synthesized. The advantages of the new material are lower cost and density, and much better machinability. The composites of Al-fly ash system are potential materials in various light weight structural applications.

9:50 AM

Deformation Behavior of Aluminium Composite: Upsetting and Cold Indirect Extrusion: *S. Das*[1]; K. Hatsukano[2]; D. P. Mondal[1]; A. H. Yegneswaran[1]; K. Aoi[2]; [1]Regional Research Laboratory CSIR Bhopal 462 026 India; [2]Mechanical Engineering Laboratory, Tsukuba, Japan

An attempt has been made to examine the effect of particle reinforcement on the upsetting (compressive) behaviour and cold indirect extrusion of 2014 Al alloy at a strain rate of ~$2.85x10^{-2}$s. The Al composite was synthesized using two types of reinforcing agents i.e., SiC and Al_2O_3 in particulate forms having a size range of 50-80 μm and amount 10wt%, through solidification processing route (vortex method). It has been noted that the alloy and composites show more ductile behaviour during upsetting as compared to that under tensile loading condition. It is further noted that even composites are deformed up to 80% reduction in height without any cracking under compressive loading. The flow stress of the materials is found to follow the relation as given below: $\sigma_f = \sigma_e + K\varepsilon$ Where σ_f=flow stress; σ_e=stress at elastic limit; K=a constant which represent the resistance against the plastic deformation; ε=plastic strain and n=strain hardening coefficient. The value of σ_f at a selected strain value is noted to be significantly higher in composite as compared to that of the alloy. It was further noted that the value of σ_e and K are also higher in the composites. On the other hand, the measured value of n is noted to be same in all the materials. Among the composites, SiC reinforced one exhibits higher values of σ_f, σ_e, and K as compared to that obtained in Al_2O_3 reinforced one. After examining these facts, the composites and the alloy are successfully cold extruded (indirectly) without any surface defects to form a cylindrical cup having an internal diameter of 14 mm, wall thickness of 3 mm and height of 32mm. It has been noted from microstructural examination after the cold indirect extrusion that material flows upward at an angle of 45° with respect to the axis of loading and few of the reinforcing particles in the composite samples are fractured and reoriented themselves during extrusion.

10:10 AM Break

10:30 AM

Abrasive Wear Behaviour of Al-Alloy Hard Particle Composites: Effect of Matrix Alloy, Type of Particle, Abrasive Size and Load: *D. P. Mondal*[1]; S. Das[1]; S. Gupta[1]; [1]Regional Research Laboratory, Hoshangabad Rd., Bhopal 462026 India

Two body abrasive wear behaviour of cast Al alloys with different chemical composition has been investigated under varying loads and abrasive sizes. The alloys have been reinforced with SiC particle in order to examine the effect of SiC reinforcement on the abrasive wear of the alloys. One of the alloys has also been reinforced with Al2O3 particle in order to examine the effect of different particles on the abrasive wear response of the alloy. In most of the cases, either alloy or composites (particle reinforced alloy), wear rate is found to be invariant with sliding distance but it increases with increasing load and abrasive size. It is noted that among the alloys, the wear rate is found to be minimum for LM13 alloy and in case of composite, the minimum wear rate is noted for SiC reinforced composite. The relative contribution of each of the factors and their interaction towards the wear rate has been studied through factorial design of the experimental data. It is examined that the effect of load on the wear rate is more severe as compared to the effect of abrasive size and these effects vary from material to material. It is further noted that the interaction effect of load and abrasive size in all the alloys are same but is smaller than that in composites. It is further noted that the interaction of load and abrasive size towards the wear of all the SiC reinforced composites is same and significantly smaller than that in Al2O3 reinforced composite.

10:50 AM

Study on the Mechanical Properties of ZA-27 Alloy Reinforced with Quartz Particles: *P. V. Kruparkara*[1]; S. C. Sharma[2]; S. Manjunath[2]; M. Krishna[2]; J. Uchil[3]; [1]R.V. College of Engineering, Dept. of Chem., Bangalore, Karnataka 560059 India; [2]R.V. College of Engineering, Dept. of Mech. Eng., Bangalore India; [3]Mangalore University, Dept. of Matl. Sci., Mangala Gangotri, Dakshina Karnataka, Mangalore, Karnataka 574 199 India

This paper aims to study the effect of Quartz reinforcement on the mechanical properties of cast ZA-27 alloy composite containing various % of Quartz particulates. Results show that the composites exhibit higher Young's modulus and ultimate strength in comparison with the un-reinforced material with a decrease in ductility. The investigation also shows that compression strength appears to increase as the percentage of particulate content of the MMC increases. Hardness also appears to improve with % particulate addition.

11:10 AM

Damping Properties and Tribological Behaviour of Al-fly Ash Particle Composites: *Y. B. Liu*[1]; Z. Y. Cao[1]; Y. Y. Li[1]; [1]Jilin University of Technology, Changchun, Jilin 130025 PRC

Al/fly ash composites containing 5-20(V/V)% of fly ash particles are fabricated using stir-cast techniques. The internal friction values of these composites are measured with a multifunction internal friction apparatus to evaluate the damping properties of Al/fly ash composites. Under these experimental conditions, Al-fly ash composites possess higher internal friction values than that of Al matrix. The internal friction values of composites are quite strongly depended on the vibration frequency and temperature, and also on the particle size or volume percentage of fly ash. The damping mechanisms are also discussed.

The Mechanisms of the Massive Transformation - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Phase Transformations Committee
Program Organizers: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; V. K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Tuesday AM Room: Mississippi
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Masato Enomoto, Ibaraki University, Dept. of Matls. Sci., Hitachi 316-8511 Japan

8:30 AM
Massive Transformation and the Formation of the Ferromagnetic τ-MnAl Phase in Mn-Al Base: Cagatay Yanar[1]; Jorg M.K. Wiezorek[1]; Velimir Radmilovic[1]; *William A. Soffa*[1]; [1]University of Pittsburgh, Matls. Sci. and Eng. Depts., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Recent work on the evolution of microstructure and the defect structure in the MnAl-base permanent magnet materials has revealed that the formation of the uniaxial, high anisotropy ferromagnetic τ-MnAl ($L1_0$) phase involves a compositionally invariant, diffusional transformation or massive ordering. The metastable τ-MnAl phase nucleates virtually exclusively at the grain boundaries of the parent ε (hcp)-phase and grows behind advancing incoherent interfaces. The isothermal nucleation and growth kinetics of the ε (hcp) to τ ($L1_0$) are analyzed. The characteristic polytwinned structure and high density of lattice defects are shown to derive from atomic attachment processes at the interphase interfaces. High-resolution electron microscopy (HREM) studies of the interphase interfaces are reported.

9:10 AM
Diffusion Along Interphase Boundaries: Eugen Rabkin[1]; *Wolfgang Gust*[2]; [1]TECHNION, Dept. of Matls. Eng., Haifa 32000 Israel; [2]University of Stuttgart, Instit. of Phys. Metall., Seestr. 92, Stuttgart D-70174 Germany

It will be tried to compare interphase boundary diffusivities deduced from measurements of massive growth kinetics with independently determined diffusivities in the same or similar system. Finally, we would like to consider the question of the comparability of measured diffusivities along interphase boundaries with the diffusivities which apply across interphase boundaries, the operative situation during massive transformations.

9:50 AM
Radiotracer Studies of Interphase Boundary Diffusion: Chr. Herzig[1]; *Y. Mishin*[2]; [1]Universitaet Muenster, Institut fuer Metallforschung, Wilhelm-Klemm-Strasse 10, Muenster D-48149 Germany; [2]Virginia Polytechnic Institute and State University, Dept. of Matls. Sci. and Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

Radiotracer diffusion measurements offer the most direct access to diffusion characteristics of interphase boundaries. The modern radiotracer techniques employed in such measurements are presented and illustrated by applications to grain boundary diffusion. Applications to interphase boundary diffusion are relatively scares. Some experimental problems in this area, such as preparation of well characterized oriented interphase boundaries, are pointed out. Earlier work on interphase boundary diffusion is briefly reviewed, and the recent radiotracer measurements on oriented (110) and (110) Ag/Cu and (110) Ag/Ni interphase boundaries are presented in most detail. The obtained diffusion characteristics together with their temperature dependencies and anisotropy are interpreted in terms of the interfacial structure-diffusion correlations. The relationship between interphase boundary diffusion and grain boundary diffusion in the adjacent phases is also discussed. Preliminary results for interfaces in the Ti-Al system are presented. Atomistic modeling and simulation results for interphase boundary structure and diffusion are briefly reviewed.

10:30 AM Break

10:35 AM
Interphase Boundary Diffusion in Ti-Al: *David E. Luzzi*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci., 3231 Walnut St., Philadelphia, PA 19104-6272 USA

The strongly crystallographic twins, pseudo-twins and interphase boundaries between the lamellae of polysynthetically-twinned (PST) Ti-Al crystals provide a system with which to study interface diffusion. Interface diffusion in PST Ti-Al has been studied experimentally and theoretically. PST crystals of lamellar TiAl containing dispersed lamellae of Ti3Al were grown in an optical floating zone furnace by zone melting and directional solidification. Candidate diffusants were placed on oriented, cut and polished surfaces of PST crystals by sputter deposition or diffusion bonding. Penetration of the diffusants along the interfaces and within the bulk as a function of time and temperature was determined using Auger electron spectroscopy and energy dispersive spectroscopy within scanning or transmission electron microscopes (TEM). The structure and microstructure of the diffusion couples were characterized using TEM. Diffusion in lamellar TiAl was modeled by atomistic simulation employing many-body central-force potentials. Both stoichiometric interfaces and interfaces with segregated Ti were studied.

11:15 AM
Atomic-Scale Studies of Segregation at and Diffusion Across Heterophase Interfaces: *David N. Seidman*[1]; Dieter Isheim[1]; [1]Northwestern University, Matls. Sci. and Eng. Dept., Matls. and Life Sci. Bldg., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Solute-atom segregation at heterophase interfaces in metallic alloys is studied on an atomic scale employing one-dimensional (1DAP) or three-dimensional atom-probe (3DAP) microscopy, which gives a real-space image of the position of atoms and their chemical identities. Experimental evidence is presented for segregation of vanadium at iron/molybdenum heterophase interfaces, in terms of the Gibbsian interfacial excess of vanadium; the interfaces are created by solid-state decomposition of an Fe(Mo,V) ternary alloy. Also experimental evidence is presented for segregation of antimony or tin at iron/molybdenum nitride heterophase interfaces, where the Gibbsian interfacial excesses are measured as a function of the state of coherency of this heterophase interface; the interfaces are created by the internal nitridation of Fe(Mo,Sb or Sn) ternary alloys. The results are mechanistically interpreted in terms of segregation models. The effects of segregation on diffusion across heterophase interfaces are also discussed.

The Science of Alloys for the 21st Century: A Hume-Rothery Symposium Celebration - III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, and the University of California Lawrence Livermore National Laboratory
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

Tuesday AM Room: Missouri
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Mikael Ciftan, Dept. of the Army, Army Research Office, Research Triangle Park, NC 27709-2211 USA; Michael V. Nathal, NASA Glenn Research Center, Matls. Div. (Mail Stop 49-3), Cleveland, OH 44135 USA

8:30 AM Invited
Evolution of Materials Science: A Personal View: *Mikael Ciftan*[1]; [1]Army Research Office, Dept. of the Army, P.O. Box 12211, Research Triangle Park, NC 27709-2211 USA

I present today the thrust that Dr. John Hurt and I started at the Army Research Office in the area of alloy science in the early seventies when ARO was on the Duke University campus and how far we have come. Our vision then was centered on national needs, including mimicking the properties of strategic metals by producing alloys that would mimic their desirable properties such as those derived from Chromium. There was no "Materials Division" or "Materials Department" around at the time, rather, Metallurgy and Ceramics Departments. We had a difficult time to convince these specialists of the need and potentiality of a microscopic physics/chemistry approach to alloy problems. We wanted to and did succeed in initiating a microscopic first-principles approach in alloy theory, including a quantum mechanical one as appropriate, starting from realistic potentials. We were keen on trying to understand the role of defects and defect structures in the same vein. I will sketch our efforts in this program, starting with our support of Professor Ryoichi Kikuchi's research on Phase Diagrams in the early seventies and ending with the calculations that Professor Nicholas Kioussis has performed recently, on a quantum mechanical level, on the role of defect structures in intermetallic alloys.

8:55 AM Invited
Quantitative Principles of Network Glass Chemistry: *James C. Phillips*[1]; [1]Lucent Bell Laboratories, 1E-245, 700 Mountain Ave., Murray Hill, NJ 07974-0636 USA

The optimized compositions of network glasses are successfully predicted by a remarkably simple model, which is called constraint theory. The model avoids explicit constructions of the ball-and-stick type, and even utilizes the methods of molecular dynamics only very sparingly. Instead it counts the number of bond-stretching and -bending interactions and compares these with the number of degrees of freedom of the system. In general optimized compositions are found at or near those for which these two quantities are equal. At such compositions the network under goes a stiffness transition which changes qualitatively the nature of internal stresses. In such regions there is also a reversibility (or non-entanglement) window in the enthalpy of the glass transition, as measured by the new method of modulated differential scanning calorimetry. The theory determines the composition of window glass, one of nature's most remarkable materials, without adjustable parameters.

9:35 AM Invited
Liquids, Crystals, and Glassy Metals-Science and Technology of Bulk Metallic Glasses: *William L. Johnson*[1]; [1]California Institute of Technology, W.M. Keck Lab. of Eng. Matls. 138-78, Pasadena, CA 91125 USA

During the past decade, the development of complex metallic alloys with exceptional glass forming ability has opened new opportunities for the study of deeply undercooled alloy melts. This has led to new understanding of atomic transport, rheological properties, liquid phase separation, and crystallization kinetics as the glass transition is approached. The talk will discuss progress in understanding the fundamental science of liquid alloys and glass formation, as well the development of metallic glass as an engineering material.

10:15 AM Break

10:55 AM Invited
Trends in Metallic Materials Research for Aerospace Propulsion Programs: *Michael V. Nathal*[1]; [1]NASA Glenn Research Center at Lewis Field, Matls. Div., Mail Stop 49-3, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Materials development programs at NASA have traditionally reflected a tension between the desire to produce revolutionary advances through strong basic research programs and the need to meet near term vehicle-based goals through focussed development programs. This tension tends to produce "pendulum swings" in funding between short range and long range research. In many cases, metallic materials development efforts must counter the perception that many metallic systems are approaching the asymptotic limits of their performance, as for example, the temperature capability of nickel base superalloys. As such, metals are facing competition from polymer matrix composites at low temperatures, and ceramics at higher temperatures. One aspect of future development efforts that is growing in emphasis is the use of physics-based modeling to both enhance materials development and to improve reliability of materials in harsh engine environments. Examples of some recent modeling success stories will be highlighted.

11:20 AM Invited
The Use of Alloy Theory for the Development of Active and Passive Magnetic Refrigerant Materials: *Karl A. Gschneidner*[1]; Vitalij K. Pecharsky[1]; Alexandra O. Pecharsky[2]; [1]Iowa State University, Ames Lab., Dept. of Matls. Sci., 255 Spedding, Ames, IA 50010 USA; [2]Iowa State University, Ames Lab., 255 Spedding, Ames, IA 50010 USA

In designing active magnetic refrigerant materials the most critical parameter is the behavior of the entropy as a function of temperature and magnetic field in the vicinity of the anticipated operating temperatures. The most important property for passive magnetic refrigerants is a large low temperature ($T < 60$ K) volumetric heat capacity over a reasonable temperature range (± 10 to ± 20 K) around the magnetic ordering temperature. Although the theory of alloy formation takes a back seat to these two parameters, it still plays an important part in developing new and/or improved magnetic materials for refrigeration technologies. Its use allows one to determine which alloying agent(s) can be most effectively substituted in a magnetic intermetallic compound, or a pure magnetic element to shift its magnetic ordering temperature to a more desirable temperature, or to modify the magnetic properties to change the shape (width and height) of the magnetic heat capacity, or to form a two phase alloy in which each compound orders magnetically at appropriate temperatures to give the desired entropy and heat capacity behavior for a particular application.

Electron Backscatter Diffraction: Applications of EBSD - III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Thin Films & Interfaces Committee
Program Organizers: Adam J. Schwartz, Lawrence Livermore National Laboratory, L-355, Livermore, CA 94550 USA; Mukul Kumar, Lawrence Livermore National Laboratory, L-356, CA 94550 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Tuesday PM Room: Meramac
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Dorte Juul Jensen, Risø National Laboratory, Matls. Rsch. Dept., Roskilde Denmark; Terry R. McNelley, Naval Postgraduate School, Dept. of Mech. Eng., Monterey, CA USA

2:00 PM Invited
Material Sensitive Design in Fourier Coefficient Space: *Brent Larsen Adams*[1]; Benjamin Lyman Henrie[1]; Surya Kalidindi[2]; [1]Brigham Young University, Dept. of Mech. Eng., 435 CTB, Provo, UT 84602 USA; [2]Drexel University, Dept. of Matls. Eng., 3141 Chestnut St., Philadelphia, PA 19104 USA

Automated EBSD provides essential data that can be used to form estimates of various mechanical and physical properties. These data include the orientation and misorientation distributions, and higher-order information such as the 2-point orientation correlation functions. The known structure-properties relations are briefly reviewed, and limitations of the theory are noted. As an example of applications we consider the design of a compliant fixed-guided beam. Performance of the beam for various microstructures is examined in a low-order fourier coefficient space for the orientation distribution. It is illustrated that typical properties objectives are represented by hyperplanes in the fourier space, and that properties are limited by the intersection of these hyperplanes with the convex hull of fourier coefficient space describing the range of possible microstructures.

2:30 PM Invited
Use of EBSD Data in Mesoscale Numerical Analyses: *Richard Becker*[1]; Hasso Weiland[2]; [1]Lawrence Livermore National Laboratory, Mech. Eng./NTED, P.O. Box 808, L-228, Livermore, CA 94551 USA; [2]Alcoa Technical Center, Matls. Mech. Dept. ATC-C, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

The spatial maps of lattice orientation made available by automated EBSD provide an excellent means for obtaining input for deformation models based on crystal plasticity and for evaluating the accuracy of the models. Results from model validation studies are presented where deformation was simulated using finite elements with a crystal plasticity constitutive description. These results demonstrate the necessity for three dimensional models which accurately represent the grain structure and boundary conditions, but the agreement between the predicted and the EBSD-measured spatial distribution of lattice orientation is still far from satisfactory. Although the source of modeling errors is not resolved, potential influences of the details of the spatial discretization and neglecting coarse slip bands are discussed.

3:00 PM Invited
Characterization of Deformed Microstructures: *David P. Field*[1]; Hasso Weiland[2]; [1]TexSEM Laboratories, 392E 12300S Ste. H, Draper, UT 84020 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA

Electron back scatter diffraction offers insight into the structure of heavily deformed materials that is difficult or impossible to obtain using any other characterization technique. This paper discusses the results of orientation imaging analysis used in three independent investigations of deformed structures. The first study examines rolled Aluminum and compares the EBSD results with those obtained in the TEM. The second study is of equal channel angular extrusion and cold rolling of electronic Cu. The final study investigates the structures formed during friction stir welding of Aluminum. The results are discussed in the context of characterization strategies for deformed materials using orientation imaging techniques.

3:30 PM Break

3:40 PM Invited
Anisotropic Plasticity Modeling Incorporating EBSD Characterization of Tantalum and Zirconium: *John F. Bingert*[1]; Thomas A. Mason[2]; George C. Kaschner[2]; Paul J. Maudlin[3]; George T. Gray[2]; [1]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, T-3, MS B216, Los Alamos, NM 87545 USA

Automated EBSD techniques have been applied to characterize tantalum and zirconium as part of a predictive materials modeling effort incorporating anisotropic properties. The application of EBSD toward improved representations of anisotropic behavior will be illustrated through experimentation and modeling of high-rate Taylor cylinder impact tests for tantalum plate, tantalum rolled rod, and zirconium plate. Anisotropic plasticity was incorporated into the constitutive models through a 5-dimensional yield surface calculated from the discretized pre-impact texture. Experimental results from Taylor tests, in the form of cylinder profiles and footprints, are compared to those predicted from continuum simulations of the impact event. EBSD results from post-impact cylinders and bend-beam samples revealed the dependence of deformation microstructure evolution on initial orientation and deformation twin activity. Relevant issues regarding yield surface calculations, deformed microstructure length scales, twinned microstructures, the complementary nature of x-ray and EBSD data, and the role of anisotropy in damage evolution will also be addressed.

4:10 PM Invited
Measuring Strains using Electron Backscatter Diffraction: *Angus J. Wilkinson*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxon OX1 3PH UK

The electron backscatter diffraction (EBSD) technique has undergone significant development in recent years. The technique is most widely used for crystal orientation measurement. However, EBSD also provides a powerful tool for the analysis of local strains, which this paper reviews. Plastic strains cause (i) a reduction in the contrast and sharpness of Kikuchi bands in EBSD patterns, and (ii) small local misorientations due to dislocations stored in the deformed crystals. Methods for measuring both pattern quality and small local lattice rotations are described and their application to the characterisation of plastic deformation is reviewed. Elastic strains can also be determined from EBSD patterns by measuring the small changes in the positions of zone axes that are induced by the distortion of the diffracting lattice. Image processing methods to determine the small zone axis shifts will be described and the analysis that relates them to elements of the strain tensor presented.

4:40 PM Invited
On Mapping Residual Plastic Strain in Materials Using Electron Backscatter Diffraction: *Edward Mark Lehockey*[1]; [1]Ontario Power Technologies, Matls. Integrity, 800 Kipling Ave., Toronto, Ontario M8Z 5S4 Canada

A new approach for imaging and quantifying residual plastic strains in materials is introduced based on the area density of low-angle misorientations measured by changes in Electron Backscatter Diffraction (EBSD) patterns. The area density of low-angle misorientations in the substructure of Alloy 600 and 1020 carbon steel exhibit a reliable correlation with applied strain offering a convenient means of calibrating deformation in field samples of these materials. Numerous examples are presented demonstrating the ability to image the spatial distribution of deformation with a combination of superior resolution and wider dynamic range in scan area than is possible with alternative techniques. Contributions of highly localized strain fields associated with microstructural features (e.g., inclusions, precipitates, etc.) to the overall "global" strain distribution can be readily appreciated. Significant potential exists for this approach in failure analysis and quality assurance applications for evaluating strains that evolve in component from manufacturing, installation, or service.

5:10 PM
Electron Backscatter Diffraction Study of Intergranular Stress Corrosion Cracking in FCC Alloys: *Val Y. Gertsman*[1]; Stephen M. Bruemmer[1]; [1]Pacific Northwest National Laboratory, P.O. Box 999, P8-16, Richland, WA 99352 USA

Paths of intergranular stress corrosion cracks were examined in several iron- and nickel-base stainless alloys tested in high-temperature water environments. Particular attention was paid to the crack arrest points. In general, only twin $\Sigma 3$ boundaries exhibited improved resistance to crack propagation. If the $\Sigma 3$ were factored out, the distribution of grain boundary types among cracked boundaries corresponds to the grain boundary character distribution for the bulk alloy. Other CSL boundaries, including $\Sigma 3^n$ (i.e. $\Sigma 9$ and $\Sigma 27$), were observed to crack when appropriately oriented in the crack path. The cracks were often (but not always) arrested at grain boundary junctions containing $\Sigma 3$ boundaries even if such boundaries did not lie along the most favorable crack path. Possible benefits of multiple twinning for material's resistance to intergranular degradation are discussed. On the other hand, sometimes cracks were observed to stop at or bypass general grain boundaries even though they were favorably orientated with respect to the applied stress. The results obtained indicate that grain boundary crystallography alone does not fully determine its susceptibility to stress corrosion cracking, and other factors need to be taken into account when assessing material's propensity to intergranular failure.

General Abstracts: Corrosion & Environmental Effects

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Tuesday PM — Room: Jefferson A
October 10, 2000 — Location: Regal Riverfront Hotel

Session Chair: Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

2:00 PM
Corrosion Behavior of Al2O3-C-TiC (TiO2) Refractory in Molten Bath of Smelting Reduction with Iron Bath: *Qingcai Liu*[1]; Jing Lin[2]; Dengfu Chen[3]; Yuan Xu[3]; [1]University of California at San Diego, Depts. of MAE and USCD, 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]Chongqing Jianzhu University, Dept. of Appl. Sci., Chongqing 400045 PRC; [3]Chongqing University, Sch. of Matls. Eng., Chongqing 400044 PRC

It is one of the key problems to solve the corrosion of lining refractory materials by molten bath in the smelting reduction technology with iron bath. The most important qualities of melts for smelting reduction with iron bath are high concentration of ferrous oxides, complex structure of molten slag and the high reducing rate of ferrous oxides. The composition and behavior of melts were also changed dynamically and constantly in melting process. Research results shown that corrosion rate and behavior of lining refractory in melts of smelting reduction with iron bath were much higher and complex than that of blast furnace technology. By means of pre-reduction with fixed bed and arc furnace smelting process, the corrosion rate and corrosion mechanism for Al2O3-C-TiC and Al2O3-C-TiO2 refractories in molten bath of smelting reduction with iron bath were investigated. The relationship between the composition, metallurgical qualities of melts, melting conditions and technology parameters were studied in detail. Titanium carbide, existing in the interface between refractory and melts, will deter the moving ability of slag film and decrease the diffusion speed of refractory components, the corrosion rate of Al2O3-C-TiC(TiO2) refractory was dropped much more than that of Al2O3-C refractory. Meanwhile, the corrosion mechanism and corrosion rate of Al2O3-C-TiC refractory was much difference from that of Al2O3-C-TiO2 refractory in the same melting condition.

2:20 PM
Oxidation Behavior of Cu-8Cr-4Nb: *Linus U.J.T. Ogbuji*[1]; Donald L. Humphrey[1]; Dennis J. Keller[2]; David L. Ellis[3]; [1]Dynacs Engineering Company, M.S. 106-1, NASA-Glenn Rsch. Ctr., Cleveland, OH 44135 USA; [2]RealWorld Quality Systems, Inc., 20388 Bonnie Bank Blvd., Rocky River, OH 44116 USA; [3]Case Western Reserve University, Mail Stop 49-1, NASA-Glenn Rsch. Ctr., Cleveland, OH 44135 USA

GRCop-84 (Cu-8 at.% Cr-4 at.% Nb), developed at NASA-GRC, is a strong candidate for use as a thrust cell liner and nozzle ramps for advanced reusable launch vehicles. Its excellent elevated temperature strength, thermal stability, and high thermal conductivity have been documented. However, little is known about its oxidative durability, which will be an issue in a high-temperature engine flame. The oxidation characteristics and kinetics of this material have been studied by TGA in air and oxygen at close temperature intervals ranging from 500 to 900°C. At ~800°C and above two regimes were observed in the GRCop-84 oxidation kinetics. The parabolic rate constant for the first ~10 hours is over twice that of the final 30 hours in a 50-hour TGA run. The transition to a lower kp was found to correlate with the emergence of oxides other than Cu2O and CuO. These and other results will be presented and compared with the oxidation behavior of two related baseline materials: copper and NARLOY-Z (Cu-3 wt.% Ag-0.5 wt.% Zr).

2:40 PM
Gallium Suboxide Attack of Selected Pure Metals and Alloys at 1200°C: *David G. Kolman*[1]; Thomas N. Taylor[1]; Marius Stan[1]; Carl Maggiore[1]; Joseph Tesmer[1]; George Havrilla[1]; YoungSoo Park[1]; Darryl P. Butt[1]; [1]Los Alamos National Laboratory, Mail Stop G755, Los Alamos, NM 87545 USA

A process has been established for separating Ga_2O_3 from PuO_2-1 wt % Ga_2O_3 powder by exposing the powder to 1200°C Ar-6% H_2 gas. Second-phase Ga_2O_3 precipitates react with H_2 to produce H_2O and $Ga_2O_{(g)}$. Unfortunately, Ga_2O gas is a known corrodant. Therefore, the susceptibility of materials of construction for the reactor to Ga_2O attack are a concern. The objective of this work is to find a fabricable metal which can withstand 1200°C H_2, H_2O, and Ga_2O gas. While information is available regarding the high temperature H_2 and oxidation resistance of metals, there is no information available regarding Ga_2O resistance. This work will examine the attack of candidate materials (Cr, Co, Co-Cr-W-Ni, W, W-Cu, Mo, and Mo-Re) by Ga_2O and compare their resistance to materials (SS and Ni-Cr-Mo) which have failed in service. Materials attack will be examined using thermodynamics calculations and a suite of surface science techniques including X-ray photoelectron spectroscopy, proton-induced X-ray emission, Rutherford backscattering spectroscopy, micro X-ray fluorescence, and scanning electron microscopy/electron microprobe techniques.

3:00 PM Break

3:20 PM
Investigation on the Role of Microstructure on Initiation of SCC Cracks of Pipeline Steel in Bicarbonate Solutions: Vladimir Sizov[1]; Biao Gu[2]; Lin Yang[2]; *Scott Mao*[1]; [1]University of Pittsburgh, Dept. of Mech. Eng., 3700 O'Hara St., 648 Benedum Hall, Pittsburgh, PA 15261 USA; [2]University of Calgary, 2500 University Dr., Calgary T2N 1N4 Canada

The initiation and propagation process of stress corrosion cracking (SCC) for X-52 pipeline steel in near-neutral pH bicarbonate solution has been studied using slow strain rate tests (SSRT) and SEM fractographic examination. The results showed that multiple SCC colonies can be generated during SSRT in a certain range of cathodic potential. The critical stress necessary for SCC initiation is around the yield stress of the pipeline steel. The most susceptible potential range to SCC initiation is between -800 to -900mVSCE. As the potential decreased to more negative cathodic potential, the SCC initiation stress increased. No multiple crack colonies were found at -1200mVSCE. The measured average SCC growth rate is in an order of 10-8mm/sec and increased with decrease in applied potential. It was found that the preferential dissolution and hydrogen evolution is important in SCC initiation process. Lower pH value of solution or precharged hydrogen will favor SCC initiation. There is a critical strain rate, 2.5x10-7/sec, most susceptible to SCC. Above and below this strain rate, the SCC initiation crack density decrease. That suggested some kinetics of chemical reaction are involved in SCC initiation process.

3:40 PM
Surface Roughness for Cr.13 Pipes (OCTG): *Fred Farshad*[1]; [1]University of Louisiana, Dept. of Chem. Eng., P.O. Box 44130, Lafayette, LA 70504 USA

Engineers and scientists use transmission factors in flow equations to determine pressure losses in pipes. In turbulent flow, transmission factors are a function of surface roughness in "rough" pipes. A large number of experimental and theoretical studies have been performed on friction pressure losses of fluids in smooth and rough pipes. However, relatively little work has been published on the standard surface roughness values in internally coated pipes and on its effect on the production rate and hydrodynamics in pipes. In particular, reports on research of physical measurements, mathematical modeling studies and statistical analysis of surface roughness are still scarce. The primary use of coatings has been to extend the life of the pipe by mitigating any interaction between corrosive fluids and the pipe. To a much lesser extent, these same coatings have also been used to decrease or even eliminate problems associated with paraffin and scale depositions in pipe. Until recently, most investigators and operators have overlooked the hydraulic benefits that can be gained by using internally coated pipe. This paper deals with experimental analysis of pipe roughness for coated pipes. The objective of this research work is to measure and analyze surface roughness for coated tubing and bare tubing using Surface Profiler, Hommel Tester T1000, and Atomic Force Microscope (AFM). In addition to this, statistical analysis of Root Mean Square (RMS) roughness, Rq and Mean peak to valley height, Rzd also included.

Metastability in Bulk and Thin Film Materials: Interfacial and Stress Effects on Phase Stability - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Metallurgical & Materials Engineering, Houghton, MI 49931 USA; Frank G. Shi, University of California-Irvine, Department of Chemical Engineering & Materials Science, Irvine, CA 92697 USA

Tuesday PM Room: Field
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Douglas J. Swenson, Michigan Technological University, Dept. of Metall. & Matls. Eng., Houghton, MI 49931 USA

2:00 PM Invited
Stress Induced Metastability in Thin Films: *William C. Johnson*[1]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., 116 Engineer's Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA

The sequence of phase formation in thin films often differs from that observed in bulk alloys owing to various elastic effects. Interfacial stresses, compositional strains, changes in crystal structure owing to the formation of one or more new phases, the presence of a compliant substrate, and externally applied stresses all affect the elastic state of the film and, hence, its microstructural evolution. Results of numerical calculations will be presented which illustrate how these stresses introduce additional equilibrium states not observed in stress-free systems, alter the sequence of phase formation from that observed in bulk alloys, and stabilize phases that are non-equilibrium phases in the absence of stress. This work is supported by NSF and DOE.

2:30 PM Invited
Phase Hierarchy and Metastability in Inorganic Systems: *Carlos G. Levi*[1]; Vikram Jayaram[2]; [1]University of California-Santa Barbara, Matls. Dept., Engineering II, Rm. 1361D, Santa Barbara, CA 93106-5050 USA; [2]Indian Institute of Science, Metall. Dept., C.V. Raman Ave., Bangalore 560 012 India

Materials processing often yields metastable structures that can be important on their own merit or because of the role they play in the evolution of the final microstructure and properties. A notable example is the pyrolytic decomposition of precursors for oxides, which can lead to structures with varying degrees of metastability. Pyrolysis occurs at low homologous temperatures, where diffusion is constrained, and hence the transformations tend to be partitionless. The phenomena share a common conceptual base with metastability produced by rapid solidification and vapor deposition. The concept of phase hierarchy maps will be presented and used to discuss the role of kinetics in phase selection. These maps are derived from phase diagrams and used to represent the menu of possible phases as a function of temperature and composition. Kinetic constraints that bias the phase selection will also be discussed, illustrating the effects of ordering, structural complexity, and the need for partitioning during crystallization.

3:00 PM Invited
Linear Perturbation Analysis of the Stability of Stressed Thin Films: *Jeffrey J. Hoyt*[1]; M. Asta[2]; V. Ozolins[1]; [1]Sandia National Laboratories, Livermore, CA USA; [2]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL USA

The energetics of a stressed, binary thin film can be conveniently described by the Fourier transform of the effective pair interactions, V(k), a function which describes the effects of both chemical interactions and atomic displacements (ie. elastic strains). The V(k) formalism has been incorporated into a set of diffusion equations and the stability with respect to small perturbations in concentration has been investigated. It is found that below a critical temperature the film is unstable over a band of wavenumbers, but remains stable in the limit of both high and low k. Stability at k®0 is a direct consequence of the atomic size mismatch and insures that a compositionally-modulated film (a striped or droplet phase) is energetically favored. The kinetic model assumes that transport of atoms within the film takes place via exchange with the adatom gas, a mechanism identified in recent surface diffusion experiments. As a result, the wavenumber of the fastest growing unstable perturbation depends on the ratio of the solute diffusion coefficient in the adatom gas to the film-adatom exchange rate.

3:30 PM Invited
Metal-C60 Thin Film Nanosystems: *Rodica Manaila*[1]; [1]National Institute of Physics, P.O. Box MG-7, 105bis Atomistilor St., Bucharest, Magurele RO-76 900 Romania

Complex interaction effects take place at the interface between a metal and C60 fullerite. They include charge transfer, but also formation of hybridized metal-carbon bonds. These effects are amplified in thin film nanosystems, consisting in metal nano-clusters, dispersed within a fullerite matrix, with a high interface area. Interaction effects also determine the (meta)stability of these metal clusters, which can decompose or grow, depending on their radius and nature. Evidence (from XRD, TEM, EXAFS, SERS) will be brought forward on the structure, microstructure and vibrational properties of the nanosystems (Au, Cu)-C60, as influenced by interface interaction effects.

4:00 PM Invited
Metastable Phase Formation by Reactive Diffusion in Multilayers: *Patrick Gas*[1]; Claire Bergman[1]; [1]Université Aix-Marseille, L2MP, Fac. St Jerome-Case 511, Marseille 13397 France

Reactive diffusion in thin films and multilayers appear as a smart method to produce stable and metastable materials with specific properties. The main advantage of solid state reaction in thin films (vs. bulk couples) is the existence of a strong phase selection which may in certain conditions lead to the growth of single phased metastable films. The basic mechanisms involved in such reactions are discussed. Attention is focussed on the physical factors promoting phase selection and metastable phase formation. Some typical examples of formation of crystalline, amorphous or quasicrystalline metastable phases in Al/transition metal (TM) and Si/TM systems are presented. Emphasize is put on reaction in Al/Co multilayers and on the formation of a metastable decagonal quasicrystal as a second reaction product.

4:30 PM Invited
Modeling Structural Metastability of Irradiated Thin Films: *Paolo M. Ossi*[1]; [1]INFM, Dipartimento di Ingegneria Nucleare, Politecnico di Milano, via Ponzio 34-3, Milano 20133 Italy

It is experimentally observed that when a compound film, both metallic and non-metallic is bombarded with massive, energetic particles, such as ions, the crystal structure can be damaged up to a point where amorphization eventually occurs. Understanding and possibly predicting the structural stability of irradiated materials is a long standing, basic problem, with considerable technological implications, due to the changes of material properties at the transition between the crystalline and the vitreous state. In this work a microscopic model is presented, which is based on the evolution in the bombarded target of collision cascades, which are formed as a consequence of projectile stopping. Basically, a cascade can be considered to be an exiguous volume of excited matter which relaxes towards equilibrium via different mechanisms, both non-equilibrium and thermodynamic, depending on the relevant timescale. In the Segregation Charge Transfer (SCT) model, so called from its main ingredients, attention is focused on the evolution of the interface between a cascade and the surrounding crystalline matrix, considered to be prototypical of the behaviour of all such interfaces in the target. On the basis of experimental evidence, the development at the interface of a non-equilibrium compositional profile is assumed; this is the consequence of the interface segregation of one of compound constituents and it is associated to the concurrent development of an electronic density profile, which can be non-equilibrium over the typical cascade quenching timescale. Relaxation to (meta)stable equilibrium is simulated by a Charge Transfer Reaction (CTR), involving a pair of dissimilar atoms of the initial compound (IC), from which a dimer of an effective compound (EC) is formed. A given EC is compared to the corresponding IC, considering the energy expense required for the CTR to occur and the local volume variation, related to strain effects. The model has been applied to a number of binary compounds, including intermetallics, silicides, nitrides, carbides and oxides, which are known from the literature to be amorphised, or to remain crystalline under ion bombardment. Threshold values of the above structural stability parameters have been found, which allow to definitely separate the two families of materials. Thus the SCT model constitutes a single conceptual framework within which it is possible to interpret phase evolution in irradiated materials.

5:00 PM
Thin Film of Electrodeposited Metal as Product of Formation and Solidification of a Metastable Supercooled Liquid Phase: *Oleg B. Girin*[1]; Il'ya M. Kovenskii[2]; [1]Ukrainian State University of Chemical Engineering, Dept. of Matls. Sci., Prospekt Gagarina 8, Dnipropetrovsk 49005 Ukraine; [2]Tyumen State Oil and Gas University, Ul. Volodarskogo 38, Tyumen 625000 Russia

Experimental data on crystal defects are discussed for electrodeposited films of copper, nickel, iron and chromium. The point defects were studied by positron annihilation, the linear by X ray diffraction, the surface ones by transmission electron microscopy, and the volume defects by hydrostatic weighing techniques. It was found that increased supercooling in electrodeposition of the metals leads to higher densities of vacancies, dislocations and pores while the grain boundary images change from a line type to a point type contrast. The fact that the crystal defects are dependent on supercooling in the electrodeposition of a metal film supports an earlier discovery of a phenomenon consisting in that the precipitation of the metal being electrodeposited occurs via a metastable supercooled liquid phase.

Powder Metallurgy Alloys and Particulate Materials for Industrial Applications: Ferrous and Wear Resistant Alloys - I

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: David E. Alman, US Department of Energy, Albany, OR 97321 USA; Joseph W. Newkirk, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday PM Room: Clark
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Joseph W. Newkirk, University of Missouri-Rolla, Metall. Eng. Dept., Rolla, MO 65409 USA; Shankar M.L. Sastry, Washington University, St. Louis, MO 63130 USA

2:00 PM
Properties of High Nitrogen Steels Produced by High Pressure Gas Atomization: *John S. Dunning*[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321-2198 USA

The production of metal powder by gas atomization typically combines melting at ambient pressure and atomization through high pressure gas jet nozzles into an atomization chamber, also at ambient pressure. To achieve high nitrogen contents, a series of Fe-Cr-Mn-Ni steels were melted under nitrogen pressures of 150 psi and atomized with nitrogen gas into an atomizing chamber held at 100 psi. A small scale atomizing unit (charge size 300 lbs) was modified to achieve the desired production characteristics. High nitrogen steels produced by high pressure gas atomization exceeded the atmospheric solubility limit of nitrogen by as much as 500% and nitrogen contents between 0.5 to 1.3 wt. pct. was achieved. Three Fe-Cr-Mn-Ni alloys each different nitrogen contents were atomized and consolidated by hot-extrusion. The microstructure and tensile properties were evaluate and correlated to conditions during atomization, extrusion and final nitrogen content.

2:20 PM Invited
Fully Dense Particle Metallurgy Tool Steels: Microstructure and Properties Characteristics: *Andrzej Wojcieszynski*[1]; Robert Dixon[1]; Edward Tarney[1]; [1]Crucible Research, 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA

The Crucible Particle Metallurgy (CPM) process, commercialized in the early 1970's, has permitted the production of many highly alloyed high speed steels, corrosion and abrasion resistant steels, wear resistant tool and die steels, and superalloys. The CPM process involves atomization of fully alloyed melts, HIP consolidation of the resulting powder, and forging or rolling to typical bar sizes, (or use in the as-HIP'd condition for near net shape components). The rapid solidification inherent in gas atomizing results in uniform dispersions of fine carbides in the wear-resistant grades. The resulting alloys have improved mechanical properties, particularly elevated resistance to abrasion, without the usual sacrifice of toughness or other properties. This paper reviews the current state of the PM tooling materials market, examining the development of microstructures of CPM alloys, and focuses on the development of new grades and a comparison of properties and performance among conventional and HIP-consolidated PM tooling materials.

2:45 PM Invited
P/M Materials for Wear Applications: *Jeffrey A. Hawk*[1]; [1]U.S. Department of Energy, Matls. Conserv. Div., Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321-2198 USA

Wear resistant materials usually consist of either very hard homogeneous single phase materials (e.g., ceramics like Al2O3, SiC, etc.) or heterogenous materials (e.g., white cast irons, composites or cermets, or composite-type materials), typically with a hard reinforcing phase dispersed in

a softer matrix. In both instances, the result is the same, less penetration of the abrasive into the surface of the material being worn. Composite-type materials can be produced using either a melting/solidification scheme or through powder metallurgy (P/M) techniques. In either case the result is the same, a microstructure that consists of a high volume fraction of hard, usually brittle, second phase particles in a softer matrix. However, P/M can be used to create a wider range of these materials than can melting/solidification, because in P/M processing, the desired phase does not have to be precipitated during solidification. Thus, more materials can be produced with higher volume fractions of reinforcing phases. Obviously, other factors like reinforcement size, matrix-particle interfacial strength, plastic accommodation of the matrix, etc. become important in the wear behavior of these materials. Various categories of P/M wear resistant materials will be discussed, and their wear behavior will be compared against traditional wear resistant cast materials like white cast iron and tool steels.

3:10 PM Invited
Applications of P/M in Heavy Equipment Industry: *Keith H. Boswell*[1]; Gerald A. Gegel[1]; [1]Caterpillar, Precision Comp., 315 Cardiff Valley Rd., Rockwood, TN 37854 USA

As the world's largest manufacturer of construction and mining equipment, diesel and natural gas engines and industrial gas turbines, Caterpillar has, for 30 years, manufactured some of the largest P/M parts made by the industry. These parts weigh up to 4.5 kilograms. Caterpillar has used the near-net-shape capabilities of the P/M process to reduce manufacturing costs for over 700 components that were previously either castings or stampings. This paper will review current opportunities for using large P/M parts. Ongoing development projects will expand the application of P/M technology by providing lower cost MIM technology for manufacture of controlled porosity parts and near full density turbine engine components, and process technologies that will enable the fabrication of large, high density alloy steel P/M parts. There will be a review of the manufacturing methods and facilities used to produce these low volume, large complex components.

3:35 PM Break

3:50 PM
The Functional Properties of the Laser Consolidated Wear Resistant Stellite 6 Alloy: L. Xue[1]; *J. -Y. Chen*[1]; M. U. Islam[1]; [1]National Research Council Canada, Integ. Manufact. Tech. Instit., 800 Collip Circle, London, Ontario N6G 4X8 Canada

Laser consolidation (LC) is a novel process that produces a net shape functional part layer by layer directly from a CAD model by using a laser beam to melt the injected powder and re-solidifying it on the previous pass. As an alternative to the conventional machining process, this computer-aided manufacturing (CAM) process can build complete parts or features on an existing part by adding instead of removing material. The Stellite 6 samples built up by the process are metallurgically sound, free of cracks and porosity. The laser-consolidated (LC) Stellite 6 is harder, stronger and even more ductile than the same material produced by either casting or the powder metallurgy method. The tensile strength of the LC Stellite 6 is about 50% higher than that of the cast or powder metallurgy material. The hardness of the LC Stellite 6 is about Rc 58, which is much harder than the same material produced through the casting or powder metallurgy method. The LC Stellite 6 also shows substantially higher yield strength and slightly higher elongation than the cast or powder metallurgy Stellite 6. The excellent mechanical properties of the LC Stellite 6 can be attributed to its exceptionally fine columnar dendrite microstructure produced by the rapid solidification associated with the process. The material was examined in detail by optical microscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD) and Auger electron microscopy (AES). The wear and corrosion resistance of the LC Stellite 6 were also measured and compared with other materials.

4:10 PM
Devitrified Nanocomposite Steel Powder: *D. J. Branagan*[1]; [1]Bechtel BWXT Idaho LLC, Idaho Nat. Eng. and Environ. Lab., 2351 N. Blvd., Idaho Falls, ID 83415-2218 USA

Using basic principles of metallic glass formation, multicomponent steel alloy compositions were developed with low critical cooling rates (»104 K/s) for metallic glass formation. This enabled the production of spherical micron sized metallic glass powder by inert gas atomization. The metallic glass precursors were found to typically devitrify from 775 to 925 K with enthalpies of transformation from -75 to -150 J/g. Due to the characteristic uniform nucleation and high nucleation frequency found in a glass, multiphase nanocomposite microstructures developed with phases sizes from 2 to 75 nm. The extremely large fraction of two dimensional defect interfaces/boundaries which exist in the nanoscale structure interact with and prevent dislocation motion resulting in extremely high strength and hardness. Measurements using nanoindentor and Vickers hardness tests show that the hardness of the devitrified nanocomposite steel is in excess of 15.5 GPa. The low-cost and high hardness of the devitrified steel alloys makes them excellent candidates for a wide variety of wear resistant applications.

4:30 PM
Wear Study of Carbide Reinforced P/M Ferrous Alloys: *Jeffrey A. Hawk*[1]; Omer N. Dogan[1]; Rick D. Wilson[1]; [1]U.S. Department of Energy, Matls. Conserv. Div., Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321-2198 USA

Ferrous alloys have been used for decades as wear resistant materials for applications where severe wear (primarily abrasion) has been a problem. Irons and steels have an advantage in these applications because they can be alloyed with various elements to create structures which are quite harder than the iron matrix. For example, martensitic matrices can be developed as well as carbide structures (consisting usually of Cr-, Nb- or V-based carbides or some other complex carbide structure). However, these materials are usually made through melt/solidification techniques which can sometimes limit the type, volume fraction, and morphology of the carbide. In P/M processing these factors can be ignored in most instances. This paper will discuss the abrasive and impact-abrasive wear behavior of one class of wear resistant materials based on TiC. The materials removal mechanisms will be discussed with emphasis on microstructure-wear interactions. The results will be compared to traditional wear resistant ferrous-based alloys.

4:50 PM
Powder Processing and Abrasion Resistance of Insitu Iron Matrix-TiC Reinforced Composites: *Omer N. Dogan*[1]; *David E. Alman*[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321-2198 USA

Reactive powder processing techniques were used to produce TiC reinforced ferrous matrix composites. Mixture of elemental Fe, Cr, Ti and graphite powders were hot-pressed to full density. During heating a Self-propagating High-temperature Synthesis (SHS)-type reaction initiated at about 1150°C and transformed the Ti, Cr and graphite powders to TiC and (Fe,Cr)x,Cy in an steel (Fe-Cr-C) matrix. This reaction occurred only in those mixtures containing Ti powder (i.e., no such reaction was detected in mixtures of Fe, Cr and graphite powders). The resultant hot-pressed microstructures consisted of uniformly dispersed and large carbide particles in a steel matrix, and ideal microstructure for abrasion resistance. The abrasion resistance of the composites were evaluated by pin-abrasion testing and the results compared favorable to traditional wear resistant materials, such as cast iron.

5:10 PM Invited
Recent Advances in Mechanical Alloying: *C. Suryanarayana*[1]; [1]Colorado School of Mines, Depts. of Metall. and Matls. Eng., 1500 Illinois St., Golden, CO 80401-1887 USA

Mechanical Alloying is an advanced powder metallurgy technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill. Originally developed in the late 1960's to produce oxide dispersion strengthened nickel- and iron-base superalloys, it has been recently recognized that mechanical alloying is capable of producing both stable and metastable phases, at room temperature, starting from blended elemental powders. The metastable phases synthesized include supersaturated solid solutions, metastable intermediate phases, amorphous alloys, and nanocrystalline materials. Mechanically alloyed materials exhibit properties that are often superior to those produced by conventional processing techniques. The technique of mechanical alloying is now applied to produce materials of importance to a variety of industries including high-temperature intermetallics, ceramic materials, electronic materials, and composites. It has also been reported recently that pure metals can be produced from their oxides or chlorides at room temperature and thus mechanical alloying has now become established as an advanced processing technology. The present talk would review the recent developments in this exciting field.

Rate Processes in Plastic Deformation II: Towards an Unified Theory of Deformation - IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA; Thomas R. Bieler, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; Bimal Kad, University of California, AMES Laboratory, La Jolla, CA 92093-0411 USA; Farghalli A. Mohamed, University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697 USA

Tuesday PM Room: Lewis
October 10, 2000 Location: Regal Riverfront Hotel

Session Chairs: Dr. S. V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA; Dr. George T. Gray, Los Alamos National Laboratory, Los Alamos, NM USA

Dislocation Substructure Modeling of Materials - II

2:00 PM Keynote
Modelling the Evolution in Microstructure and Properties during Plastic Deformation of FCC-Metals and Alloys; An Approach Towards a Unified Model: *Erik Nes*[1]; K. Marthinsen[1]; [1]Norwegian University of Science and Technology, Depts. of Matls. Tech. and Electrochem., Alfred Getz Vei 2B, Trondheim N-7034 Norway

A new approach to the modeling of work hardening during plastic deformation of fcc metals and alloys has recently been proposed by the present authors. By combining a solution for the dislocation storage problem with models for dynamic recovery of network dislocations and sub-boundary structures, a general internal state variable description is obtained. The model includes effects of: (i) stacking fault energy, (ii) grain size, (iii) solid solution content, and (iv) particle size and volume fraction. The result is a work hardening model which in principle is capable of providing the stress strain behavior for a given metal or solid solution alloy under any combination of strain rate and temperature. It will be demonstrated that the model predictions, in terms of microstructure evolution and associated properties, are in good agreement with experimental observations.

2:30 PM Invited
Cyclic Plasticity and Substructure of Metals: *Petr Lukas*[1]; [1]Academy of Sciences of the Czech Republic, Instit. of Phys. of Matls., Zizkova 22, Brno CZ-61662 Czech Republic

Fatigue of metals is consequence of cyclic plastic deformation. Without cyclic plasticity there would be no fatigue of metals at all. Fatigue properties as well the mechanical properties in general are strongly dependent on the starting substructure. Moreover, the microstructure undergoes changes during cyclic loading. This is valid both for the quasi-homogeneous cyclic plastic deformation in the whole cyclically loaded metal and for the highly localized cyclic plastic deformation at the crack initiation sites and later at the crack tip. The paper will deal with the delimitation of the cyclic plasticity and its specific features which distinguish it from the monotonic plasticity. At the beginning of the cycling a more or less expressive cyclic hardening and/or softening takes place. This is due to changes of the dislocation and/or precipitation structure. The cyclic hardening/softening often has a saturation character. The steady state is characterized by the cyclic stress-strain curve. It will be shown how far this cyclic stress-strain curve represents a mechanical equation of state independent of the strain-path and how the steady state dislocation structures depend on the stress-strain history. The effect of temperature-path will be treated along the same lines. The most prominent feature of the cyclic plasticity is formation of persistent slip bands (PSB's) in some metals under suitable conditions. The conditions for the formation of the PSB's (type of metal, stress and strain requirements, temperature etc.) will be shown and the role of the PSB's in the overall cyclic plasticity, especially their effect on the cyclic stress-strain curve will be examined. The relation between the cyclic plastic deformation and the initiation of fatigue microcracks will be discussed for metals both with and without formation of the PSB's.

2:50 PM Invited
A Review of Deformation Mechanisms and Their Mapping: *H. J. Frost*[1]; [1]Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755-8000 USA

It has now been more than 30 years since Professor John Dorn and colleagues formulated the description of high temperature creep in metals and alloys into the power-law creep equation now known as the Dorn equation. It is almost 30 years since Professor M. F. Ashby suggested that this power-law creep, together with other deformation mechanisms, could be conveniently graphically displayed in the form of maps showing the complete relationship among the variables stress, strain-rate and temperature, and various parameters of describing the microstructure. In this paper we will review our efforts to update the deformation-mechanism map description of steady-state and transient deformation in polycrystalline materials, so as to include the continuing developments over the last quarter-century in constitutive equations for thermally-activated deformation mechanisms.

3:10 PM Break

The Role of Rate Processes in Materials Design and Forming Technologies

3:25 PM Keynote
Rate Processing in High Temperature Deformation: *John J. Jonas*[1]; [1]McGill University, Dept. of Metall. Eng., 3450 University St., Montreal, Quebec H3A 2A7 Canada

The rate processes involved in high temperature deformation are reviewed. These include static, dynamic and metadynamic (postdynamic) recrystallization. In commercial rolling processes, the effect of each of these softening mechanisms depends, not only on the length of the interpass interval, but also on whether the strain-induced precipitation of carbonitrides has taken place. The kinetics of each mechanism are also affected by the composition of the alloy under consideration. Because of the complexities of the interactions between these phenomena, models of wide or general applicability have not yet been developed. Progress in the simulation of metal processing operations is thus being made by comparing the predictions made by various models with mill measurements. Removal of the discrepancies observed then leads to improvements in the algebraic forms of the relevant equations as well as to more accurate values of the associated coefficients and parameters.

3:55 PM Invited
Kinetics of Cavity Growth in Superplastic Materials: *Amit K. Ghosh*[1]; [1]University of Michigan, Matls. Sci. and Eng. Depts., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

In this paper, the growth rates of superplastic cavities has been reexamined. Models based on grain boundary diffusional cavity growth, used previously for explaining the growth rates under slow creep conditions provide an underestimate of the rate of growth of cavities initiated at grain boundary particles during superplastic flow. In these materials, voids are "nucleated" due to incompatible deformation between phases, although a few cavities might preexist in certain alloys. Increased levels of local hydrostatic stress resulting from plastic constraints have shown to activate growth of interfacial defects leading to rapid dilatation, followed by a slower growth rate due to reduced constraints as voids expand. The role of diffusion is to provide accommodation for void growth in the submicron range and decrease the rate of early growth, not enhance it. The constrained plasticity growth model helps explain why the rate of void growth is enhanced by increased strain rate, larger particle size and decreasing forming temperature in metals, which are not all explainable by the diffusional cavity growth mechanism. Detailed experimental studies, utilizing image analysis of incrementally tested specimens as a function of test temperature and strain rate, have revealed how the population of cavity nuclei increase with increasing strain rate and decreasing temperature, and how this, in turn, modifies the growth rate of individual cavities. This work provides a basis for predicting the evolution of void size and overall void volume under a variety of forming conditions.

4:15 PM Invited
Orientation Effects on Shear Localization in Ti-6AL-4V: *Scott E. Schoenfeld*[1]; Bimal Kad[2]; [1]U.S. Army Research Laboratory, AMSRL-WM-TD, Aberdeen Proving Grounds, MD 21005-5066 USA; [2]University of California- San Diego, Dept. of AMES, La Jolla, CA 92093-0085 USA

The goal of the current work is to examine the influence of crystallographic texture on plastic flow during high-strain-rate shearing of Ti-6Al-4V. A continuum model for the Ti-6Al-4V aggregate will begin by implementing a two-dimensional kinematic approximation to the hexagonal close-packed (hcp) crystal structure for TiAl single crystals. The resistance to slip motion on this structure will follow a description used to model isotropic polycrystalline Ti-6Al-4V during impact events and will be implemented using an integration scheme specifically developed for high-rate applications. The resulting constitutive theory has been implemented into an explicit Eulerian numerical scheme in order to solve the boundary-value problem of an aggregate of single crystals subject to simple shearing at high strain-rates. The effects of various thermo-mechanical processing will be assessed via the incorporation of different orientation

distributions of the single crystals, and observations of shear band phenomenology (shear band path and directions) will be correlated with the ability of the aggregate to absorb energy within the high-rate environment. To the extent that such simple shear can be used to approximate the predominant failure mode (shear plugging) during ballistic limit testing, our model will guide the development of subsequent thermo-mechanical processing of ballistic resistant Ti.

4:35 PM
Microstructural and Substructural Changes during High Temperature Severe Plastic Deformation: *Shankar M.L. Sastry*[1]; Rabindra Nath Mahapatra[2]; Shailendra K. Varma[3]; [1]Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]Naval Air Systems Command, Matls. Lab., Code 4.3.4.2, Patuxent River, MD 20670 USA; [3]University of Texas, Metall. Eng. Dept., El Paso, TX 79968 USA

Severe Plastic Deformation (SPD) processing of materials by such methods as equal channel angular extrusion (ECAE) results in intense simple shear deformation and promotes dynamic or continuous recrystallization by subgrain rotation. High temperature SPD processing has been found to produce microstructural modifications not achievable by conventional thermomechanical processing. For example, hot working of gamma titanium aluminides in the single phase field, usually above 1100°C and at high strain results in dynamic recrystallization. At slow strain rates, single phase alloys exhibit dynamic recovery and two phase alloys dynamically recrystallize. Rapid grain growth and solid state transformations upon cooling through the gamma/gamma+alpha2 boundary and eutectoid isotherm generally results in coarse grained microstructures consisting of equiaxed gamma and lamellar gamma+alpha2 microstructures. Severe hot working at temperatures below the eutectoid by isotherm is required to produce submicron sized equiaxed microstructures and prevent solid state phase transformations. However, deformation of gamma titanium aluminides to large strains using conventional thermomechanical processing routes at temperatures < 1000°C has not been possible because of the onset of fracture at small strains. Using constrained deformation such as ECAE below the eutectoid isotherm, conversion of lamellar g+a2 morphology to equiaxed morphology has been accomplished. Such a method provides flexibility in producing greater microstructural control than is possible by conventional thermomechanical processing.

4:55 PM Extended Discussion

5:25 PM Closing Remarks

The Mechanisms of the Massive Transformation - IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Phase Transformations Committee
Program Organizers: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; V. K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Tuesday PM Room: Mississippi
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Jian-Feng Nie, Monash University, Dept. of Matls. Eng., Victoria 3800 Australia

2:00 PM
The C40 to C11b Transformation in MoSi2 Thin Films: A Polymorphic Massive Transformation: Xinyi Wang[2]; Isaac Chang[2]; *Mark Aindow*[1]; [1]University of Connecticut, Dept. of Metall. and Matls. Eng., 97 N. Eagleville Rd., U-3136, Storrs, CT 06269-3136 USA; [2]University of Birmingham, Sch. of Metall. and Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK

The compound MoSi2 exhibits two polymorphic structures, namely tetragonal C11b and hexagonal C40. There is some dispute about whether the C40 phase can exist at equilibrium but it is certainly metastable at temperatures below about 1900°C. The C11b and C40 structures are closed related and share as a common structural unit a stoichiometric closely-packed layer which corresponds to the {110} or (0003) planes, respectively. The main difference between these structures is the stacking sequence of these layers and so it has been proposed that the C40 to C11b transformation may proceed via a diffusionless shear process. It has been argued that the stacking faults and coherent twins on {110}C11b which arise in the microstructures of non-equilibrium-processed MoSi2 are consistent with such a displacive mechanism. In this paper, detailed TEM studies of the transformation microstructures which occur in stoichiometric magnetron-sputtered MoSi2 thin films will be described. The as-deposited films were amorphous and crystallized to give fine-grained C40 phase on annealing at 400-700°C. An irreversible transformation to the C11b phase occurred upon annealing at higher temperatures. It was found that the C40/C11b interfaces were incoherent, and that the coherent {110} twins in the C11b phase were growth twins rather than transformation twins as proposed previously. These observations are consistent with what one would expect for a massive transformation mechanism involving non-cooperative diffusional transfer of atoms across high-energy incoherent interfaces rather than any displacive process. The origins and significance of massive mechanisms for polymorphic transformations will be discussed.

2:40 PM
Characteristics of the Transformation to AuCuII in Equiatomic Au-Cu Alloy: Jian-Feng Nie[1]; *Barry Muddle*[1]; [1]Monash University, Dept. of Matls. Eng., Clayton, Victoria 3800 Australia

The formation of assemblies of ordered AuCuII plates from disordered (f.c.c.) equiatomic Au-Cu solid solution during quenching or isothermal ageing is commonly regarded as an example of a diffusional transformation in which the transformation product has many of the characteristics of a martensitic product and for which the transformation crystallography may be accurately predicted using the phenomenological theory of martensite crystallography. However, if one accepts a general definition of the massive transformation as a composition-invariant structural change involving diffusional nucleation and growth, then it could also be argued that the ordering reaction involved in the formation of AuCuII constitutes a massive transformation. Aside from satisfying this general requirement, the transformation product certainly shares some of the features considered characteristic of a massive product, including a defect substructure and the absence of a rational orientation relationship between parent and product phases. If one accepts the controversial view that the massive interface is at least partially coherent and migrates via the defect structure within that interface, then there are also aspects of interface structure in common. The present paper will provide a detailed characterisation of the substructure and interface structure of AuCuII plates and evaluate the transformation mechanism in the context of current classifications of phase transformations.

3:20 PM
Composition Invariant Solidification and Solid State Transformation: *Wilfried Kurz*[1]; Mehrdad Vandyoussefi[1]; Milton S.F. Lima[1]; [1]Swiss Federal Institute of Technology Lausanne EPFL, Dept. of Matls., Lausanne 1015 Switzerland

Solidification microstructures as well as solid state transformation structures have been produced in isovelocity experiments with a positive temperature gradient in Fe-3w/oNi and Fe-8w/oCr alloys. The chosen growth velocities are close to the lower limit of morphological stability of a plane front for solidification (constitutional undercooling, Vc), and at the same time close to the upper limit of plane front stability (absolute stability, Va) for the delta/gamma solid state transformation. In these transformations the parent phase has a larger diffusion coefficient than the product phase and therefore volume diffusion theory may be applied to both cases. This contribution shows that the interface response function for growth morphologies (plane front, cells, and dendrites), developed for directional solidification can be useful for the interpretation of solid state transformation phenomena. Above the critical velocity, Va, where the diffusion distance, D/V, becomes small with respect to the capillary length, the interface becomes planar. Due to the small diffusion coefficient this happens for the delta/gamma transformation of the alloys studied in the velocity range of some 10-30 microns/s. In this case, and for steady state conditions, the product and parent phases have the same composition and a very thin boundary layer (approx. 1 micron) exists at the interface. This compositional spike decreases in amplitude at higher velocity when solute trapping sets in. In our isovelocity experiments composition invariant or massive transformation is due to plane front stabilization above absolute stability. The corresponding plane front growth temperature is at solidus and rises with increasing velocity to To when the distribution coefficient approaches unity.

4:00 PM Break

4:05 PM
The Amorphous-Gamma-Alpha Phase Transformation In Alumina: A Massive Transformation?: *David R. Clarke*[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

In order to separate the nucleation and growth processes, we have been studying the effect of crystallographic orientation and selective dopants on the velocity of the transformation interface using an in-situ optical reflectivity method. In this method, an amorphous alumina film is first deposited on a sapphire substrate and then the motion of the transformation interface at temperature is monitored by optical interferometery. From the temporal variation in the optical reflectivity, the instantaneous velocity of the interface is determined. The reflectivity data and subsequent SIMS analysis of the dopant distributions are then used to determine the transformation kinetics. As will be discussed, the data provides new insights in the phase transformations in aluminum oxide and possibly also massive transformations.

4:45 PM
Massive Transformation in Bismuth Oxide-Based Ceramics: *Anil V. Virkar*[1]; [1]University of Utah, Matls. Sci. and Eng. Dept., 122 S. Central Campus Dr., Salt Lake City, UT 84112 USA

Bismuth oxide, Bi2O3, is known to exist in the cubic CaF2 structure (with 25% of the oxygen sites vacant) above 730°C, and exhibits excellent oxygen ion conductivity in this form. At lower temperatures, the cubic phase is unstable and displacively transforms into a monoclinic phase. Additions of rare earth oxide, RE2O3, can stabilize the cubic phase over a wide temperature range. In many cases, the stability is kinetic. In several RE2O3-Bi2O3 systems, the cubic phase undergoes a transformation into a rhombohedral phase at lower temperatures. This transformation is composition-invariant, is thermally activated, and satisfies the accepted criteria for a massive transformation. The kinetics of massive transformation under isothermal conditions were studied in Y2O3-Bi2O3 and Gd2O3-Bi2O3 systems. In the Y2O3-Bi2O3 system, the effect of interdiffusion coefficient, as modified through defect chemistry, on the kinetics of transformation was investigated. It was demonstrated that an increase/decrease in the interdiffusion coefficient also increases/decreases the kinetics of massive transformation. In the Gd2O3-Bi2O3 system, the kinetics were investigated as a function of temperature with an emphasis on the measurement of the interphase boundary velocity. The nature of the interphase boundary (coherent or incoherent) was addressed through an indirect measurement of the surface texture developed in transformed samples. The principal characterization tools used included X-ray diffraction, optical microscopy, electron microscopy, and electron probe microanalysis.

The Science of Alloys for the 21st Century: A Hume-Rothery Symposium Celebration - IV

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, and the University of California Lawrence Livermore National Laboratory

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552

Tuesday PM Room: Missouri
October 10, 2000 Location: Regal Riverfront Hotel

Session Chair: Bruce A. MacDonald, National Science Foundation, Div. of Matls. Rsch., Arlington, VA 22230 USA

2:00 PM Invited
How Does the Future Look for Fundamental Materials Research at the National Science Foundation?: *Bruce A. MacDonald*[1]; [1]National Science Foundation, Div. of Matls. Rsch., 4201 Wilson Blvd., Rm. 1065, Arlington, VA 22230 USA

At the time of the preparation of this abstract NSF is looking at a large increase in its requested budget (17.3% or $675M) for Fiscal Year 2001. In the spirit of Hume-Rothery's outstanding contributions to metal alloy science, it is timely to consider how this significant increase in NSF funding could result in new opportunities for fundamental materials research. Major NSF initiatives are discussed with this thought in mind and some challenges offered to the materials community.

2:25 PM Invited
Thermodynamics of Point Defects in Ordered Intermetallic Phases and the Effect of These Defects on Materials Properties: *Y. Austin Chang*[1]; [1]University of Wisconsin, Matls. Sci. and Eng. Depts., 1509 University Ave., Madison, WI 53706 USA

In addition to vacancies, other types of point defects exist in ordered intermetallic phases. Moreover, defect concentrations in these materials are often orders of magnitude higher those in pure metals. Existence of these defects may have a strong influence on the materials properties. In this presentation, I will focus primarily on Hume-Rothery phases typified by beta-brass such as CuZn, AgMg, AuZn, FeCo etc. The predominant point defects in these phases are the anti-site defects. On the other hand, there are phases such as NiAl, CoAl, PdAl, and PdIn. They also have the beta-brass structure but their predominant point defects consist of one anti-site defect with two vacancies, often referred to in the literature as triple-defects. For a perfect ordered structure at stoichiometric composition, which may be achieved only at absolute zero, the first atoms occupy the alpha-sublattice sites, such as the body-centered positions and the other atoms occupy the beta-sublattice sites, i.e. the corner positions. At high temperatures, thermally generated defects are formed. In addition, constitutional defects are generated at non-stoichiometric compositions. For the beta-brass type phases, two types of defects (the majority defects) have been observed as noted above. In this presentation, I will focus primarily on the thermodynamics of point defects for this class of ordered phases and the influence of these defects on their mechanical properties, first on binaries and then selected ternaries. The approach used can be readily extended to other types of ordered phases provided their defect structures are known.

3:05 PM Break

3:30 PM Invited
Thermodynamic Modelling of Solution Phases with Sublattices: *Mats Hillert*[1]; [1]KTH Royal Institute of Technology, Dept. of Matls. Sci. and Eng., Stockholm Se-10044 Sweden

Materials science and engineering is concerned with more complicated systems than usually studied by physicists and chemists. Another difference is that in the materials field one is less interested in detailed information on a particular phase than in the compositions of various phases and their relative amounts, as functions of temperature, pressure and average composition of a complex material. In order to rationalize such information and to make such predictions, one has developed a technique called CALPHAD which is based on thermodynamic descriptions of all the individual phases. For substitutional phases CALPHAD is based on the concept of Lattice Stability. In solution phases with sublattices the concept of Compound Energy of end-members plays the same role. It is used in the Compound Energy Formalism (CEF) which will be described. It is argued that the Kroger-Vink symbols are not adequate unless one is prepared to limit the treatment to dilute solutions. Thus, it should not be used in the CEF. Charged end-members may appear in the CEF description of ionic solutions and a reference for charges must be chosen, similar to the situation well-known from aqueous ionic solutions. Within CEF one can define a variety of models for individual phases by choosing various sets of constituents in the sublattices, but they can all be handled with a general type of computer program. For an equilibrium calculation in a multicomponent, polyphase material it is thus sufficient to define the formula unit for each phase. There is no need to derive any thermodynamic expressions or relations from the molar Gibbs energies. Finally, it is shown how a quasi-chemical treatment of short-range order can be included in the CEF. The CEF description of ionic melts is described and two methods of including a quasi-chemical treatment for short-range order are discussed.

4:10 PM Invited
CVM and PPM in the 21st Century: *Ryoichi Kikuchi*[1]; [1]University of California at Berkeley, Matl. Sci. and Min. Eng., 1702 Comstock Dr., Walnut Creek, CA 94595-2469 USA

After the CVM was introduced around 1950, it stayed dormant until 1970's when the tetrahedron basic cluster was used to compute binary FCC phase diagrams. As this history shows, applications of CVM followed the development of computers. In order to obtain numerical accuracy, large clusters and sometimes many-body interactions are needed. A particular emphasis is to be placed on the 4-point clusters, including the tetrahedron

(TTR) treatment. PPM-TTR will make us understand phase transition kinetics of FCC and BCC alloys, and will make us visualize intermediate stages of the transitions using the "Crystal Growth" Simulation Method. The 4-point cluster treatment in the CVM-Liquid theory will lead to the stable liquid state and further to formulations of melting. Other applications will be discussed.

4:50 PM Invited

Prospectives of Realistic Modeling of Mesoscopic Microstructures: J. Yongmei[1]; Yu Wang[2]; A. Artemev[3]; *A. G. Khachaturyan*[1]; [1]Rutgers University, Dept. of Cer. and Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]Rutgers University, Dept. of Mech. and Airspace Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA; [3]Carleton University, Dept. of Mech. and Airspace Eng., Ottawa, Ontario KIS 5B6 Canada

Recent advances in Phase Field theory and Phase Field Micromechanics of phase transformation combined with advanced supercomputing makes possible a realistic 3-dimensional simulations of mesoscopic microstructure development in coherent multiphase systems during decomposition, ordering and martensitic transformation. It will be shown that this approach is naturally extended to describe domain structures in ferromagnetics and ferroelectrics in terms of the mesoscopic model. It can be employed for realistic simulation of a 3-dimensional domain structure and its response to the applied magnetic/electric fields and thus for a prediction of the magnetic and electric hysteresis. It has been recently shown that the Phase Field methods and Phase Field Micromechanics can be also successfully extended to the new area-to the plastic deformation of crystalline materials- which opens a window to a vast area of application of computational methods for a realistic 3-dimensional simulation of behavior of arbitrary systems of interacting individual dislocations under applied stress. Recent works demonstrate that the developed Phase Field approach can be successfully extended to simulate the microstructure evolution not only in single-crystals but in polycrystals as well. These methods have a potentiality to be an effective tool for a future computational prototyping the nanoscale microstructures and their effect on the materials properties in structural, magnetic, and ferroelectric materials.

NOTES

Wednesday, October 11, 2000

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
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Electron Backscatter Diffraction: Applications of EBSD - IV

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Thin Films & Interfaces Committee

Program Organizers: Adam J. Schwartz, Lawrence Livermore National Laboratory, L-355, Livermore, CA 94550 USA; Mukul Kumar, Lawrence Livermore National Laboratory, L-356, CA, 94550 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Wednesday AM Room: Meramac
October 11, 2000 Location: Regal Riverfront Hotel

Session Chairs: Richard Becker, Lawrence Livermore National Laboratory, New Techn. Eng. Div., Livermore, CA 94550 USA; John F. Bingert, Los Alamos National Laboratory, MST:6 Matls. Techn.: Metall., Los Alamos, NM USA

8:30 AM Invited
Continuous Recrystallization and Grain Boundaries in a Superplastic Aluminum Alloy: *Terry R. McNelley*[1]; [1]Naval Postgraduate School, Mech. Eng. Dept., 700 Dyer Rd., Monterey, CA 93943-5146 USA

Computer-aided electron backscatter diffraction (EBSD) analysis methods have been employed in the analysis of microstructures in superplastic aluminum alloys. These EBSD methods have revealed the occurrence of deformation banding during thermomechanical processing of Supral 2004, a widely used superplastic Al-6%Cu-0.4%Zr alloy. Also, they have shown that the grain boundary character that enables superplastic response in this material becomes established in a refined, deformation- induced cellular dislocation structure within the bands in the as-processed condition. Continuous recrystallization in such a microstructure consists of the gradual, recovery-controlled sharpening of the cell walls within the bands; retention of the band structure in the absence of long-range high-angle boundary migration accounts for the retention of texture during annealing. Improved microstructural control may result from application of automated EBSD leading to improved superplastic aluminum alloys for industrial applications.

9:00 AM
The Application of Orientation Imaging Microscopy to Phase Transformations in Steels: *Allen Wayne Wilson*[1]; George Spanos[1]; [1]Naval Research Laboratory, Phys. Metall. Brnch., Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375-5000 USA

Orientation Imaging Microscopy (OIM) has been used in the materials community to characterize grain boundaries, measure texture and orientation relationships, and map local misorientations in many bulk materials. In this study, the technique has been applied to quantitatively examine solid-state phase transformations in steels. Microstructures examined include ferrite precipitates in a martensite matrix, and cementite precipitates in an austenite matrix. By utilizing misorientation maps to determine the orientation differences between individual crystals, the internal morphology of the solid state precipitates has been determined, i.e., what appear by other types of imaging to be monolithic single crystals are shown to be aggregates of finer crystals. The relative quality of the EBSD patterns have been correlated to specific phases using Image Quality (IQ) maps. These techniques have been adapted for identification of different microconstituents, and quantitative determination of their volume fractions. These observations provide insight into the nucleation and growth mechanisms of ferrite and cementite.

9:20 AM
Determination of Eutectic Solidification Mode in Sr-Modified Hypoeutectic Al-Si Alloys by EBSD: *Kazuhiro Nogita*[1]; Arne K. Dahle[1]; [1]The University of Queensland, Dept. of Min. Mins. and Matls. Eng., St. Lucia, Brisbane, QLD 4072 Australia

The effect of eutectic modification by Sr on nucleation and growth of the eutectic in hypoeutectic Al-Si foundry alloys has been investigated by electron back-scattering diffraction (EBSD) mapping. By comparing the orientation of the aluminium in the eutectic to that of the surrounding primary aluminium dendrites, the growth mechanism could be determined. If the eutectic nucleates on the primary aluminium phase, it would be expected that the crystallographic orientation of the aluminium in the eutectic would be related to that of the dendrites. On the other hand, if the eutectic is nucleated separately in the interdindritic melt, the orientation of aluminium in eutectic would be independent from surrounding dendrites. The mapping results indicate that the eutectic grew from the primary phase in unmodified alloys. When the eutectic was modified by Sr, eutectic grains nucleated separately from the primary dendrites. However, with overmodification, the eutectic again grew from the primary phase.

9:40 AM Invited
EBSD Contra TEM Characterization of a Deformed Aluminum Single Crystal: *Xiaoxu Huang*[1]; Dorte Juul Jensen[1]; [1]Risø National Laboratory, Matls. Rsch. Dept., Roskilde DK-4000 Denmark

EBSD technique is very popular in characterizing deformation microstructure, often replacing TEM techniques. In this work, a one to one comparison of the deformation structures revealed by EBSD and TEM techniques was made for a moderately deformed aluminum single crystal, to investigate whether the two techniques will lead to the same result. It was found that the crystal breaks up on a coarse scale into alternating bands with large (>1°) and small misorientations, respectively. The misorientations between the bands are as high as 10°. The coarse scale subdivision into band structures was identified by both techniques. However, the arrangement of the dislocation boundaries within the bands was observed only by TEM. This was attributed to the limit of the angular resolution in EBSD.

10:10 AM Break

10:25 AM Invited
Migration of Interfaces: *J. K. Farrer*[1]; N. Ravishankar[1]; J. R. Michael[2]; C. B. Carter[1]; [1]University of Minnesota, Matls. Sci. and Eng. Depts., Rm. 151 Amundson Hall, 421 Washington Ave. SE, Minneapolis, MN 55455 USA; [2]Sandia National Laboratory, Albuquerque, NM 87185-1405 USA

Sintering of ceramic materials generally involves boundary migration that is accompanied by mass transport across the interface. In the case of liquid-phase sintering, a liquid film may be present at the grain boundaries, which results in enhanced mass transport between grains. The use of bicrystals with glass layers in the boundary can provide a controlled geometry to study boundary migration. EBSD has been used to obtain orientation information from an Al_2O_3 bicrystal, sintered with a glassy phase of $CaAl_2Si_2O_8$. EBSD patterns were taken near the migrated grain boundary and compared to the patterns from the original bulk crystal to obtain the orientation relationship of the portion of crystal through which the boundary had migrated. The patterns indicate that there are small-angle misorientations in the migrated region. Obtaining this information by EBSD provides useful insight on the mechanism of boundary migration.

10:55 AM Invited
Grain Boundary Character Based Design of Polycrystalline, High Temperature Superconducting Wires: *Amit Goyal*[1]; [1]Oak Ridge National Laboratory, Mets. and Cers. Depts., P.O. Box 2008, MS 6116, Oak Ridge, TN 37923 USA

The use of electron backscatter diffraction (EBSD) to guide the design of high temperature superconducting (HTS) wires is discussed. Complicated microstructures typically present in HTS wires were understood using EBSD by discerning the grain boundary networks present in hundreds of connected grains. Microstructures and hence grain boundary assemblages were highly dependent on the processing technique employed to fabricate the wire. HTS wires with only uniaxial textures, with local biaxial textures and with macroscopic biaxial textures were fabricated to modify the grain boundary networks. EBSD was used at each stage to guide the design and fabrication of such wires as well as in correlating the final microstructure to the physical properties of the HTS wire. Fabrication of practical HTS wires with a controlled networks of grain boundaries and having near "ideal" physical properties is a fine example of how EBSD can be used in an effective and powerful manner to affect grain boundary control in real materials.

General Abstracts: High Temperature Alloys

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Wednesday AM Room: Jefferson A
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Mark L. Weaver, University of Alabama, Dept. of Metall. and Matls. Eng., Tuscaloosa, AL 35487-0202 USA

8:30 AM
Microstructure Development and Oxidation Resistance of a Mo-2.5Si-1.1B Alloy: *Keith J. Leonard*[1]; Brian Kowalski[1]; Madan G. Mendiratta[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. and Eng. Depts., 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA; [2]UES, Inc., AFRL/MLLM, Bldg. 655 WPAFB, Dayton, OH 45433 USA

Multi-phase alloys based on the Mo-Si-B system containing a dispersion of intermetallic particles in an Mo matrix have shown promise for very high-temperature structural applications that require a balance of mechanical and environmental properties. A Mo-2.5Si-1.1B (wt.%) alloy was produced through casting followed by extrusion to remove solidification inhomogeneities. The ability to control the microstructure for balanced properties was examined. Samples of the alloy were subjected to a matrix of heat treatments and the microstructures examined by XRD, OM, SEM and TEM. In addition, the cyclic oxidation behavior of this alloy between 800 and 1100°C was examined, with comparisons made to similarly tested alloys near the T2-Mo_5SiB_2 stoichiometry. Results for these studies are reported.

8:50 AM
Directional Recrystallization Processing: *Adebayo Yekeen Badmos*[1]; Ian Baker[1]; Harold Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA

How interactions between microstructural parameters and processing conditions control microstructural evolution in directional recrystallization are being investigated. An optical-image furnace is being used to directionally-recrystallize rolled single crystals of nickel with and without fine alumina particles, produced by internal oxidation. Particle sizes and spacings are varied using different aluminum concentrations and internal oxidation temperatures. Hot zone temperatures and rates of movement that produce single crystals, columnar grain structures or equi-axed grain structures are being determined for given dispersion parameters. Simulation via a front-tracking algorithm is being used to model the microstructural evolution. The ultimate goal is to use both the fundamental understanding developed and the modeling to process advanced aerospace materials efficiently. This research is supported by the Air Force Office of Scientific Research (AFOSR) grant F49620-00-1-0076.

9:10 AM +
The Influence of Age Hardening on the Primary Creep Resistance in a Near-Gamma 47XD Titanium Aluminide with 0.078 wt% Carbon: *Raymond J. Simpkins*[1]; *Thomas R. Bieler*[1]; Paul McQuay[2]; [1]Michigan State University, Matls. Sci. and Mech., 3536 Engineering Bldg., East Lansing, MI 48824 USA; [2]Howmet Research Corporation, 1500 S. Warner St, Whitehall, MI 49461 USA

The influence of carbon additions on microstructure and creep strength has shown an increase in primary creep resistance without significantly reducing the room temperature ductility in both duplex and fully lamellar microstructures. The effect of heat treatment on carbide precipitation and resulting creep properties was examined in a Ti-47Al-2Nb-2Mn-0.8vol%TiB2 +0.078wt%C alloy. After solutionizing at 1300°C (2372°F) for 8hrs, various age hardening heat treatments ranging from 950°C (1742°F) to 1100°C (2200°F) from 2 to 40 hours were carried out to precipitate the carbon out of solution. Primary creep tests conducted at 815°C (1500°F) and 138MPa (20ksi) on the aged specimens revealed a increase in primary creep resistance when aged at 1000°C (1832°F) for 40 hours. Scanning Electron Microscopy and Transmission Electron Microscopy have revealed the size and distribution of carbides in gamma grains, at gamma grain boundaries, around dislocations, and at $\alpha 2/\gamma$ interfaces. The impact of the various heat treatments on the two mechanistic processes of primary creep are discussed in terms of dislocation interactions with precipitates.

9:30 AM +
The Effect of HIPing on Removal of Centerline Porosity and Formation of Equiaxed Gamma Grains on Primary Creep Resistance in Near-Gamma XD Titanium Aluminides: *Micheal Patrick Rourke*[1]; Raymond J. Simpkins[1]; Thomas R. Bieler[1]; Paul McQuay[2]; [1]Michigan State University, Matls. Sci. and Mech. Dept., 3536 Engineering Bldg., East Lansing, MI 48824 USA; [2]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49461 USA

Extensive research efforts on the elevated temperature strength of near-gamma titanium aluminides have shown that the creep resistance of these alloys is extremely sensitive to microstructural features. All castings of these alloys require hot isostatic pressing (HIPing) to close porosity that is retained near the center line from the casting process. The effect of HIP closure is that the equiaxed gamma grain size is smaller in the center. This process produces a microstructure that favors formation of equiaxed gamma grains particularly in pore closure areas. The effect of fine equiaxed gamma grains on primary creep resistance is examined. Creep experiments were conducted at 815°C (1500°F) and 138MPa (20ksi) and at 700°C (1290°F), 200 MPa (29ksi), and 800°C (1470°F), 100 MPa (15ksi). The microstructure was characterized in the center and the edge, and a correlation between poorer creep resistance and a finer gamma grain size in the center was identified. Furthermore, the grain size was smaller (and the creep resistance was poorer) in the bottom of the bar where more porosity was trapped due to insufficient feed of the shrinking volume during solidification.

9:50 AM +
Formation of Deformation Twins in Titanium Aluminides Containing 48 to 52%Al: *E. Cerreta*[1]; S. Mahajan[1]; T. M. Pollock[2]; [1]Arizona State University, Dept. of Chem. and Matls. Eng., P.O. Box 876006, Tempe, AZ 85287-6006 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., 2300 Hayward St., HH Dow 2042, Ann Arbor, MI 48109-2136 USA

To understand the formation of deformation twins in titanium aluminides, the crystallographic features of twins were investigated. Three compositions ranging in aluminum concentration from 48 to 52at% were examined in the as-processed condition as well as after primary and secondary compression creep at 200MPa and 760°C. Optical microscopy showed a propensity of twinning in all samples and the twinning frequency increased with increasing creep strain and aluminum content. The Burgers vectors of partials bounding non-coherent twin boundaries were analyzed using transmission electron microscopy. Results indicated that in the majority of cases, the Burgers vectors were a/6<112]. Occasionally, twins bounded by a/3<121] partials were observed and in some cases, these twins were associated with a/2<110] dislocations. These results will be discussed in the context of the Mahajan-Chin model previously proposed for the formation of twins in FCC crystals. E.C. acknowledges the ONR support through an ASSERT grant.

10:10 AM Break

10:30 AM +
Microstructures of As-Processed and Crept Titanium Alumindes of Varying Aluminum Concentrations: *E. Cerreta*[1]; S. Mahajan[1]; T. M. Pollock[2]; [1]Arizona State University, Dept. of Chem. and Matls. Eng., Tempe, AZ 85287 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109 USA

Polycrystalline, binary single phase titanium aluminides of compositions 48, 50, and 52at%Al have been crept at 760°C and 200MPa. Microstructures of the as-processed materials as well as those deformed in primary and secondary stages of creep have been examined using optical and transmission electron microscopy. The microstructures of all the as-processed materials consist of subgrains and twins. The 48 and 50%Al alloy have a higher initial dislocation density than the aluminum rich alloy, with the highest defect density in the stoichiometric alloy. The 48%Al alloy in primary creep develops ordinary dislocations that are blocked by subgrain boundaries and twins. Additionally the number of twin intersections increases in primary creep. In secondary creep, more faulted regions develop and the ordinary dislocations are now cusped and bowed. While most twins in these alloys, twin with the a/6<112] partial, in the 50%Al alloy there is some evidence of twinning with a a/3<121] twinning partial. While many grains contain high densities of dislocations, twin intersections are observed in the 50%Al alloy and the amount of twinning increases slightly in the initial stages of creep. Finally, the 52%Al alloy seems to deform primarily by twinning, with an overall increase of 34 to 50% of the grains becoming twinned by secondary creep. Arguments for rationalizing these observations will be developed. E.C. acknowledges the ONR for the ASSERT fellowship which funds this research.

10:50 AM
Influence of Residual Strain upon Initial Oxidation of 3%Si-Fe Alloy: *Hiroi Yamaguchi*[1]; Minoru Takashima[1]; Mitsumasa Kurosawa[1]; Michiro Komatsubara[1]; [1]Kawasaki Steel Corporation, Tech. Rsch. Labs., Kawasakidori 1-chome, Mizushima, Kurashiki, Okayama 712-8511 Japan

The internal oxidation on 3%Si-Fe alloys, which have been pre-strained by cold rolling and then annealed up in H2O-H2 atmosphere, has been studied by scanning electron microscopy and oxygen analysis. The initial oxidation behavior during annealing varied with the following two stages corresponding to the temperature. Below 1073K, the oxidation proceeded along the alloyÕs cell walls (or subgrain boundaries) formed during the recovery process, resulting in a network structure. Therefore, this process was controlled by pipe diffusion of oxygen. Above 1073K, the dendritic shape oxide was formed along <111> direction of recrystallized alloy and internal oxidation grew at the parabolic law. The dendritic growth was controlled by volume diffusion of oxygen. The diffusion coefficient and the activation energy were estimated Do=4.3x10-8cm2/s and E=0.65eV respectively. In contrast, only the dendritic oxide growth was observed for the specimen without pre-strain.

11:10 AM
Effects of Carbon and Manganese Contents on Creep and Creep Fatigue Properties of High Nitrogen Austenitic Stainless Steels: *Takanori Nakazawa*[1]; Masayuki Tendo[2]; Hajime Komatsu[3]; Hitoshi Kaguchi[4]; Masayoshi Yamazaki[5]; [1]Gunma University, Mech. Eng. Dept., 1-5-1 Tenjin-cho, Kiryu 376-8515 Japan; [2]Nippon Steel Corporation, Steel Rsch. Labs., 20-1 Shintomi, Futtsu 293-8511 Japan; [3]Nippon Steel Techno Research, 20-1 Shintomi, Futtsu 293-8511 Japan; [4]Mitsubishi Heavy Industries Limited, 1-1 Wadasaki-cho, Hyogo-ku, Kobe 672-5953 Japan; [5]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305-0047 Japan

The effects of carbon and manganese on creep rupture and creep fatigue properties of high nitrogen Mn-Ni-Cr-Mo austenitic stainless steels were investigated from the viewpoint of phase stability. Microstructures of the tested specimens and aged ones were examined with electron microscope and by analysis of electrolytic extraction residues. Reducing carbon content from 0.010mass% to 0.003% caused decrease in rupture strength and ductility, and also in creep fatigue life. As the manganese content increased from 4% to 8%, the rupture strength at longer term decreased, while the creep fatigue life was not changed. Precipitates, such as Laves and Sigma phases were observed mainly on the grain boundaries. Time-temperature-precipitation diagrams were determined between 550°C and 650°C for up to 10000h as a function of carbon and manganese contents. Reducing carbon content markedly accelerated the precipitation of Laves and Sigma phases, while increasing manganese content somewhat promoted the precipitation of them. Precipitation of Laves phase reduced solute molybdenum content and would result in loss of rupture strength. Sigma phase caused grain boundary embrittlement and would reduce rupture ductility and creep fatigue life.

Interfacial Dislocations: Symposium in Honor of J.H. Van der Merwe on the 50TH Anniversary of His Discovery - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structures
Program Organizers: Gary J. Shiflet, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA; William A. Jesser, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA

Wednesday AM Room: Missouri
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: J. P. Hirth, Ohio State University, Dept. of Matls. Sci. and Eng., Columbus, OH 43210 USA

8:30 AM
Jan van der Merwe and the Theory of Epitaxy: *F. R.N. Nabarro*[1]; [1]University of the Witwatersrand, Dept. of Phys., Johannesburg, South Africa

In his PhD thesis under the guidance of NF Mott and FC Frank, van der Merwe produced the first theory of the role of dislocations in the growth of epitaxial layers. This was probably the first published theory of any commensurate-incommensurate transition. Since then, van der Merwe and his collaborators have expanded the theory in its quantitative aspects to allow for kinetic factors including the coalescence of island deposits, to estimate the cohesion between substrate and epilayer and to treat cases in which substrate and epilayer have different crystal structures. It has now become a useful guide to the fabrication of practical electronic devices.

9:05 AM
The Role of Interfaces in the Nucleation of Crystallization of Amorphous Silicon: *A. Heuer*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. and Eng., Cleveland, OH 44106-7204 USA

Polycrystalline silicon ("polysilicon") thin films for microelectronic and MEMS applications are often made by depositing amorphous silicon (*a*-Si) via low pressure chemical vapor deposition (LPCVD), followed by heat treatment to induce crystallization. If the LPCVD is done between ~550 and ~580°C, and if relatively thick (~0.5mm or greater) films are grown, partial crystallization occurs—the earliest deposited *a*-Si crystallizes while further film growth is taking place. Polysilicon thin films for MEMS applications are usually produced by LPCVD on a SiO_2 "release" layer, the 1-2mm thick SiO_2 film being formed by thermal oxidation. During crystallization, heterogeneous nucleation occurs at the *a*-Si/SiO_2 interface while (apparent) homogeneous nucleation occurs in the bulk of the thin film. We have studied the heterogeneous nucleation kinetics on *a*-Si/SiO_2 and compare them to heterogeneous kinetics of *a*-Si deposited on SiN_x and Al_2O_3, and to the homogeneous nucleation kinetics.

9:40 AM
Why Do Dislocations Assemble into Interfaces?: *Doris Kuhlmann-Wilsdorf*[1]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., Charlottesville, VA 22903-2442 USA

Dislocations form interfaces between crystals, and conversely all interfaces between crystals are analyzable in terms of more or less regular dislocation arrays, whether or not this is morphologically evident. The science of epitaxy, first launched by J. H. van der Merwe's Ph.D. thesis of 1949 in Bristol and immeasurably further enriched by his life-long manifold scientific contributions, is the most successful application of the first of these insights. It has had and continues to have an enormous technological impact. Would we ever have had computers without it? Other scientists have brilliantly developed the implications of the discussed inextricable interrelation between crystal boundaries and dislocations in the areas of grain and phase boundaries. Yet, the corollary thereof in plastic deformation, even though of similarly momentous importance, has remained unappreciated until recently. However, it is the profuse generation of dislocations in the course of plastic deformation, most evidently in sheet rolling, wire drawing and forging, and the mutual trapping of a fraction of these, that causes grain refinement by the production of high densities of grain boundaries. These boundaries in turn may trigger recrystallization. And within the individual grains there are lower angle boundaries that control the materials strength and are similarly formed through the mutual trapping of glide dislocations. And what is the underlying cause of the synergy between dislocations and crystal boundaries? "Nothing but" the second law of thermodynamics. Indeed, boundary formation in the course of plastic deformation takes place for the same reason, i.e. the 2nd Law, also in non-crystalline materials and on all scales, as is finally being recognized under the label of the LES (Low-Energy Structure) hypothesis.

10:15 AM Break

10:30 AM
Interface Dislocations and Deformation Behavior of Nanostructured Metal Multilayers: *Richard G. Hoagland*[1]; *Harriet Kung*[1]; Amit Misra[1]; Terence E. Mitchell[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Atomistic modeling has been performed on interface dislocations and glide dislocations in metal multilayers. For (100) fcc/fcc multilayers, the interface comprises a network of 1/2<110> dislocations in a square grid. The dislocations are not dissociated but their width depends strongly on the two metals. For example, for Cu/Ni multilayers, the dislocations are narrow, while for Cu/Ag multilayers the dislocations have a width comparable with their spacing. Under the action of an applied stress, the wide dislocations are relatively mobile in the interface. At high stresses the interface dislocations can dissociate into Shockley partials and then move in an inclined {111} plane. Glide dislocations approaching a semi-coherent interface have also been studied. Several types of interactions between glide dislocations and misfit dislocations are observed in the simulations and the consequences of these reactions are described. The relevance of the computer simulations to experimental studies of the mechanical properties of various multilayers as a function of layer thickness will be discussed. This work was supported by the Department of Energy, Office of Basic Energy Sciences.

11:05 AM
Interfacial Dislocation Blocking in Strained Epitaxial Fillms: *L. B. Fruend*[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02912 USA

Under non-equilibrium conditions, elastic strain in heteroepitaxial thin films is relaxed through formation of misfit dislocations. This occurs largely through the glide of threading dislocation segments, each of which leaves behind a misfit dislocation segment of ever increasing length. The complexity of the relaxation process during film growth or a subsequent anneal is increased significantly once dislocations began to interact through their elastic fields. The purpose here is to review the mechanics of interaction between an advancing threading dislocation and an interface misfit dislocation which lies across its path. Simple analytical models which serve to estimate the configurational driving force on the threading segments are considered first, followed by a summary of implications of detailed computational procedures. Quantitative understanding of dislocation blocking phenomena has been enhanced substantially in recent years through observations of overall strain relaxation in films under controlled conditions and through real time transmission electron microscopy which is revealing the detailed behavior of interacting dislocations. These observations will be considered within the framework of blocking models, and some emerging issues on blocking phenomena will be noted.

Metastability in Bulk and Thin Film Materials: Amorphous Materials - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Metallurgical & Materials Engineering, Houghton, MI 49931 USA; Frank G. Shi, University of California-Irvine, Department of Chemical Engineering & Materials Science, Irvine, CA 92697 USA

Wednesday AM Room: Field
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Douglas J. Swenson, Michigan Technological University, Dept. of Metall. & Matls. Eng., Houghton, MI 49931 USA

8:30 AM Invited
Energy Landscape, Tunneling Centers and Entropic Barriers in Clusters and Model Glasses: *Gabriele Viliani*[1]; [1]Universita di Trento, INFM and Dipartimento di Fisica, Povo, Trento 38050 Italy

The topology of the potential energy surface in the configuration space of disordered systems determines their thermal and relaxational properties. For example, the low-temperature anomalies of glasses are explained in terms of tunneling of groups of atoms between two local minima, i.e. the so-called two-level systems (TLS), while the high-temperature relaxations that occur in the vicinity of the glass transition are controlled by relaxations among adjacent minima. In order to get information on the very complex configuration space, numerical calculations are necessary. We have set up a numerical procedure for the identification of a large number of minima and saddle points; in this way we were able to identify many minimum-saddle-minimum triples that could in principle produce TLS. The true TLS were found by evaluating the tunneling splitting of the ground state by using the semiclassical WKB approximation: the conditions under which the one-dimensional formula can be used in such multidimensional problems are discussed. We have also investigated the role of entropic barriers in determining relaxational properties.

9:00 AM Invited
Alloying and Amorphization in Systems with Positive Heat of Mixing: *E. Ma*[1]*;* [1]The Johns Hopkins University, Dept. of Matls. Sci. and Eng., Baltimore, MD 21218 USA

Equilibrium phase diagrams of positive-heat-of-mixing (+ΔH) systems, including those composed of common elements such as Ag-Cu, Fe-Cu, and Ag-Ni, show no intermediate phases and little or no mutual solubility in the solid and sometimes even the liquid state. Highly nonequilibrium alloys can be created in such systems through both solid-state and rapid quenching routes. To achieve solid-state alloying in systems immiscible at low temperatures, one would have to i) artificially configure microstructure to alter the energetics of the system, or ii) rely on externally driven processes. Alloying on free surfaces is an example of strategy i). We demonstrate using MD that for a sub-monolayer of Ag on Cu (100), the apparent +ΔH in the surface layers is reduced relative to its value in the bulk, leading to intermixing and a surface alloy well beyond equilibrium solid solubility limits. As an example of ii), we discuss the driven alloying in 3-D Fe-Cu system through severe mechanical deformation during ball milling. The new fcc and bcc solution phases produced have been examined using EXAFS to prove atomic level alloying. Their interesting magnetic properties and atomic volume expansion have been correlated using first-principle calculations. Using a newly developed XANES method, we demonstrate that kinetically forced alloying differs from thermodynamic alloying in a number of ways, one of which is a new type of two-phase (fcc-bcc) coexistence that is polymorphic and represents a solubility overlap. Another mode of driven alloying involves the use of very high strain rate deformation at low temperatures. MD results show that both Ag and Cu are mechanically forced to melt into amorphous metals, which are miscible at <600K again due to the reduction of the +ΔH (in liquid, in this case). Upon unloading, the amorphous Ag-Cu crystallizes into a supersaturated fcc solid solution. Solid solution and amorphous alloys with +ΔH (in liquid, in this case). Upon unloading, the amorphous Ag-Cu crystallizes into a supersaturated fcc solid solution. Solid solution and amorphous alloys with +ΔH can also be obtained by extremely rapid quenching from the vapor phase. We will report the interesting and unique short-range structures of co-sputtered Ag-Ni and Ag-Cu alloys analyzed through EXAFS analysis and computer simulation. Such local structural characteristics will be correlated with the unusually low enthalpy states determined for these unstable alloys in calorimetry experiments.

9:30 AM
Anomalous Small-Angle X-Ray Scattering Study of Phase Separation in Zr-Ti-Cu-Ni-Al Bulk Amorphous Alloys: *Todd C. Hufnagel*[1]; Xiaofeng Gu[1]; Anneli Munkholm[2]; [1]Johns Hopkins University, Mats. Sci. and Eng. Depts., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Argonne National Laboratory, Chem. Dept., 9700 S. Cass Ave., BESSRC-CAT Bldg. 433, Argonne, IL 90439-4858 USA

Bulk glass-forming metallic alloys exhibit rich phase transformation kinetics, including, in some alloys, phase separation in the supercooled liquid state. We have examined the phase separation process in bulk amorphous Zr-Ti-Cu-Ni-Al at temperatures at and above the glass transition, using anomalous small-angle x-ray scattering at the Zr-, Cu-, and Ni-K absorption edges. We see the development of power-law scattering, associated with phase separation by spinodal decomposition, upon annealing; as expected, this process occurs more rapidly at higher temperatures. In addition, a separate maximum in the small-angle scattering develops, possibly due to the precipitation of Zr-rich nanocrystals. We discuss the implications of these results for our understanding of phase separation and crystallization of Zr-based bulk amorphous alloys, and provide a comparison to work by other groups on phase separation in other alloy systems.

10:00 AM
Thermal, Transport and Magnetic Properties of Bulk Pd-Ni-Fe-P Metallic Glasses: *Tongde Shen*[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, Matls. Sci. and Tech. Div., Mail Stop G755, Los Alamos, NM 87545 USA

Bulk amorphous $Pd_{40}Ni_{40-x}Fe_xP_{20}$ (x= 0-20) alloys were prepared. These alloys exhibited a broad supercooled liquid region (53-102K) and a high reduced glass transition temperature (0.57-0.67). The temperature dependence of the electrical resistivity for the alloy of x=17.5 showed no Kondo effect nor strong RKKY interaction. Field dependent measurement at temperatures of 13 and 40K showed a negative magnetoresistance, the absolute value of which increases with increasing field. With decreasing temperature and under a weak field, the glassy alloy of x=17.5 exhibited paramagnetic, superparamagnetic and spin glass behaviors. At higher field, the metallic glass showed a field-induced ferromagnetic state. The field dependence of the spin-freezing temperature for the metallic glass differs from the prediction of Neel's model. Both the temperature dependence and the field dependence of magnetic entropy change were investigated.

Powder Metallurgy Alloys and Particulate Materials for Industrial Applications: Porous Materials and Light Alloys - II

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: David E. Alman, US Department of Energy, Albany, OR 97321 USA; Joseph W. Newkirk, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday AM Room: Clark
October 11, 2000 Location: Regal Riverfront Hotel

Session Chairs: Omer N. Dogan, U.S. Department of Energy, Albany Research Center, Albany, OR 97321 USA; F. C. Chang, Argonne National Laboratory, Argonne, IL USA

8:30 AM
Development of Advanced Metallic Filters for Hot Gas Cleanup: *I. E. Anderson*[1]; B. K. Lograsso[1]; B. Gleeson[1]; [1]Iowa State University, Ames Lab., 122 Metals Dev. Bldg., Ames, IA 50011-3020 USA

Hot gas cleanup is one of the key technological barriers remaining in the development of advanced coal-fired power cycles. The successful development of such high efficiency, low emission power plant concepts is premised on efficient removal of fine particles and gaseous contaminants from high temperature, high pressure combustion gas streams. Despite research and development over the last two decades, the problems of hot gas cleanup have yet to be resolved. Recent analysis suggests that the metallic candle filter is a more promising approach to hot gas cleanup than the current ceramic filters. A unique spherical powder processing and sintering technique is being developed to fabricate metallic filters having uniform, closely controlled porosity. High pressure gas atomized powder samples of Haynes 214-alloy were tap densified and sintered to densities ranging from 64% to 74% with open permeable pore networks and transverse rupture strengths from 270 MPa to 613 MPa, respectively. In addition to microstructure analysis, the corrosion resistance of the filter materials will be evaluated under simulated gaseous combustion environments in order to determine the suitability of this alloy composition and filter structure. Funding for different aspects of this project provided by USDOE-BES and USDOE-FE (contract no. W-7405-ENG-82).

8:50 AM
A New Method of Controlling Porosity Using Carbon Fullerenes: *Xia Yang Shen*[1]; Enrique V. Barrera[1]; [1]Rice University, Dept. of Mech. Eng. and Matls. Sci., MS-321, 6100 Main St., Houston, TX 77252-1892 USA

A novel method of producing and controlling porosity in powder metallurgical parts has been identified where carbon fullerenes aid in the development of non-spherical porosity. Copper with fullerene additions have been PM processed over an extended range of temperatures, in some cases that are very close to the melting temperature of copper, to form porous materials. Both wet and dry routes of processing in fullerenes have been achieved and studied as a function of final grain size of the consolidated parts. The porosity is typically of an intergranular nature and is not in the form of spherical voids. Typically the porosity is produced by the gasification of the carbon molecules and their interaction with the copper matrix. These factors contribute to an irregular porosity being formed which is not common to most porosity forming processes. It is evident that this process is controllable via fullerene concentration, its interaction with the matrix, copper alloy additions, and processing parameters. Degrees of porosity have been identified and a model for porosity formation will be discussed. This research has been supported by the National Science Foundation under grant no. DMR 9357505 and by the Shell Foundation.

9:10 AM +
Aluminum Foams Produced in Semisolid State: Mei Yin[1]; Roger Doherty[1]; Antonios Zavaliangos[1]; [1]Drexel University, Dept. of Matls. Eng., 3141 Chestnut St., Lebow 344, Philadelphia, PA 19104 USA

Aluminum foams with high levels of porosity have become a class of high strength-to-weight ratio materials that are used in a wide range of application. Although the production of foams by liquid state has been characterized extensively, the processing of foams by semisolid state is less investigated. In this study, Al-7Si foams were made in semisolid state. The processes of bubbles nucleation, growth, coarsening, coalescence and drainage are discussed. The influence of processing parameters such as foaming temperature, foaming time and cooling rate on the cellular structural performance of the material is investigated. For aluminum foams produced in semisolid state, the existence of solid grains increases the viscosity of the melt. It plays an important role in bubbles formation. In addition, the decomposition behavior of foaming agent TiH2 under inert atmosphere was analyzed by differential scanning calorimetry (DSC) and X-ray diffraction method.

9:30 AM +
Production and Characterization of Fully Dense Titanium and Ti-6Al-4V Parts from Titanium Turnings: *Javaid I. Qazi*[1]; Charles F. Yolton[2]; Jawad Rahim[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; Valadmir S. Moxson[3]; [1]University of Idaho, Instit. for Matls. and Adv. Process., Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]P/M Speciality Materials, Crucible Research Division, 6003 Campells Run Rd., Pittsburgh, PA 15205-1022; [3]ADMA Products, Inc., 8180 Boyle Park Way, Twinsburgh, OH 44087 USA

Commercially pure (CP) titanium and Ti-6Al-4V alloy powders were produced from their respective turnings. Turnings were initially cleaned, hydrogenated and then milled to produce the powders. Oxygen and nitrogen contents of the powders were within the allowable limits. Fully dense compacts were produced from these powders by hot isostatic pressing. Scanning electron microscopy and optical microscopy were used to study the powder morphology and the microstructure of the compacts. Phase analysis of the powders and compacts was carried out using X-ray diffraction technique. Mechanical properties were studied and compared with the properties of parts made of more expensive powders.

9:50 AM Break

10:10 AM
Processing of Nanocrystalline Al-Based Alloys by Mechanical Attrition in Liquid Nitrogen: *Fei Zhou*[1]; *Enrique J. Lavernia*[1]; [1]University of California at Irvine, Depts. of Chem. Biochem. Eng. and Matls. Sci., 916 ET, Irvine, CA 92697-2575 USA

Nanostructured Al-based alloy powders have been synthesized by mechanical attrition technique under liquid nitrogen in a process known as cryomilling. Several types of Al-rich alloys, such as Al-Mg, Al-Ti-Cu, in the forms of either spray atomized powders or a mixture of elemental blends, were chosen as the model systems. The crystal refinement and the development of the microstructure during the milling process have been investigated in detail. The mechanism responsible for the crystal refinement is discussed. The nanocrystalline alloys in as-milled state are found to exhibit a high thermal stability against grain growth, which may provide the opportunity to compact these powders into high dense bulk materials without significantly coarsening of the ultrafine microstructure. Our results demonstrate a unique approach to the processing of nanostructured Al-based materials with novel mechanical properties.

10:30 AM
Development of Automotive Al-Alloy Using Hot Isostatic Pressing and Constraint Rolling: *Somesh K. Mukherjee*[1]; [1]Michigan Technological University, Metall. and Matls. Eng. Depts., 1400 Townsend Dr., Houghton, MI 49931 USA

High Strength aluminum alloys with outstanding corrosion resistance properties have wide applications in the automotive industry. 6000 series Al-alloys are a prime candidate in this respect, provided its strength can be increased further. Such high strength Al-alloys can be developed by alloy modification using Mechanical Alloying and Rapid Solidification processes. In addition, high deformation during secondary fabrication can be used to improve mechanical properties. In the present work, Mechanical Alloying and the conventional powder metallurgy route are used to prepare modified 6111 Al-alloy. Then Cold Isostatic Pressing and Hot Isostatic Pressing are used to obtain a dense Al-alloy. Secondary operation like hot rolling and novel 'Constraint Rolling' have been used for introducing strain and to obtain sheet from the dense Al-alloy. In addition, the direct hot rolling route was also used in some cases. It has been found that the mechanical properties, like tensile strength, yield strength and also ductility of the alloy, are improved to a great extent when the alloy is subjected to high deformation under 'Constraint Rolling'. Although, direct rolling give almost 100 percent density and very high elongation but lower strength.

10:50 AM
Sintering Process Characterization Using Non-Contact EMAT Generated Acoustic Waves: *David K. Rehbein*[1]; James C. Foley[1]; [1]Ames Laboratory, 211A Metals Dev., Ames, IA 50011 USA

Initial work has shown that in-situ evaluation of the mechanical strength of a powder metal compact during sintering can successfully be accomplished using a non-contact EMAT (Electromagnetic Acoustic Transducer) system. An excellent correlation between the ultrasonic echo amplitude obtained during sintering and the subsequent room temperature transverse rupture strength has been demonstrated both for solid state and liquid phase sintering. The technique developed was used to further the understanding of the sintering process and development of the mechanical strength through

monitoring of the acoustic energy transmitted through the sample as sintering progresses at varying conditions. Experiments performed at various green state densities as well as changes in the sintering temperature will be shown as measured by noncontact acoustic methods and will be compared with results of standard mechanical strength tests. This work is funded by DOE-BES under contact W-7405-Eng-82.

11:10 AM
Characterizing Strength and Sintering Progress with Acoustic Harmonic Generation Measurements: *Daniel J. Barnard*[1]; James C. Foley[1]; [1]Iowa State University, Ames Lab., Metall. and Cer. Pgm., 205 Metals Dev. Bldg., Ames, IA 50011-0001 USA

Previous experimental work on sintered powder metallurgy compacts demonstrated high levels of acoustic harmonic generation, where the fundamental amplitude of the peak in harmonic generation efficiency was found to correlate with higher transverse rupture strength. The relatively narrow peaks in harmonic generation efficiency are believed related to the initial powder sizes used in the compacts and their effect on sintering. Results of experiments examining PM compacts with varying initial powder sizes will demonstrate the effect of powder size on sintering, strength and acoustic harmonic generation efficiency. The application of acoustic harmonic generation measurements for characterizing the sintering process will be discussed. This work is funded by DOE-BES under contract W-7405-Eng-82.

Testing, Characterization and Standards for Composite Materials: Testing and Standards - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Benji Maruyama, WL/MLLM, Wright Laboratory Materials Directorate, WPAFB, OH 45433; Nikhilesh Chawla, Arizona State University, Department of Chemical, Bio and Materials Engineering, Tempe, AZ 85287-6006 USA; Awadh B. Pandey, AFRL/MLLM, Universal Energy Systems, WPAFB, OH 45433 USA

Wednesday AM Room: Lewis
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Nikhilesh Chawla, Arizona State University, Matls. Sci. and Eng. Pgm., Tempe, AZ 85287-6006 USA

8:30 AM Invited
ASTM Standard Tests for Continuous Fiber Ceramic Composites–An Overview: *Stephen T. Gonczy*[1]; [1]Gateway Materials Technology, Inc., 221 S. Emerson, Mt. Prospect, IL 60056 USA

Continuous fiber ceramic composites (CFCCs) have been under development by numerous corporations, government labs and academic institutions because they offer many potential benefits: damage-tolerant, high strain (>0.5%) failure; weight savings compared metals; and thermal and corrosion resistance at conditions where metals fail. As an expanding technology in the 1990s, CFCCs are currently being developed for a wide range of commercial applications (aerospace, industrial power, environmental control, refractories, chemical/material processing, and automotive). The CFCCs are produced by a range of different fabrication methods–chemical vapor infiltration, preceramic polymers, directed metal oxidation, sintered oxides, and reaction melting. One of the challenges in moving any technology into the market-place is to develop standardized tests that are recognized by manufacturers and users as valid, useful, and reliable. Recognizing that need for CFCCs, the ASTM C28 Committee on Advanced Ceramics formed a Ceramic Matrix Composites subcommittee in 1989 with participation from industry, government laboratories, and academia. Over the last nine years, the subcommittee has written and published nine standards for testing the mechanical properties of CFCCs in tension, flexure, shear, compression, creep, and cyclic fatigue under ambient and high temperature conditions. Draft standards are currently in process for testing transthickness tensile strength, shear strength of joints, tensile strength of single filaments, and thermal properties. This presentation will review the scope, application, and approaches for the current CFCC test methods and discuss the challenges involved in writing test standards for these challenging and complex materials.

9:15 AM
Fracture Toughness of Particulate-Reinforced Aluminum Alloy Composites: *A. B. Pandey*[1]; B. S. Majumdar[2]; D. B. Miracle[3]; [1]Pratt and Whitney, Liquid Space Propulsion, P.O. Box 109600, M/S 706-06, West Palm Beach, FL 33410 USA; [2]New Mexico Tech, Depts. of Matls. Sci. and Metall. Eng., Socorro, NM 87801 USA; [3]Air Force Research Laboratory, Matls. and Manufact. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Discontinuously reinforced aluminum (DRA) composites have been used in certain aerospace, automotive and electronic components and are being considered for more applications in aerostructures, aero-propulsion, and space propulsion components. Most of the applications of DRA were primarily driven by the superior specific stiffness and strength as compared to monolithic alloys. In order to use the DRA in load bearing applications, fracture toughness needs to be improved. This consist of two components: (a) careful measurement of fracture toughness and (b) understanding of the factors controlling fracture toughness. The purpose of this paper is to present the influence of specimen thickness and precracking on the fracture toughness of DRA composite. Elastic-plastic fracture toughness, J_{Ic} measurement was performed using ASTM E-813 technique. The toughness varied significantly with specimen thickness and precracking indicating that the constraint plays an important role in DRA similar to other materials. This was modeled in terms of void growth mechanism coupled with critical strain criterion. The experimental results also indicated that the plane strain fracture toughness criterion given in ASTM E-813 might not be valid for DRA materials.

9:45 AM
Properties for Thermal Management Applications: *Warren H. Hunt*[1]; [1]Aluminum Consultants Group, Inc., 4530 William Penn Hwy., #3900, Murrysville, PA 15668-2002 USA

Aluminum-based materials, including AlSiC and Si-Al alloys, are of great interest in electronics packaging and other thermal management applications due to the ability to tailor coefficient of thermal expansion while also obtaining high thermal conductivity and low density. Measuring the key physical properties of interest and understanding how they are affected by compositional and process variations are important to the designer. In this presentation, test techniques for assessing key physical properties will be described, and sources of variation both from test method as well as material variations discussed.

10:15 AM Break

10:35 AM
Qualification of 2009Al DRA Forging Stock for Rotating Helicopter Parts: *Cory A. Smith*[1]; Kenneth G. Davis[1]; Mark R. van den Bergh[1]; [1]DWA Aluminum Composites, 21130 Superior St., Chatsworth, CA 91311 USA

A Discontinuously Reinforced Aluminum (DRA) composite with a 2009/SiC/15p composition has entered service as forged blade sleeves on commercial helicopters. The blade sleeves are responsible for attaching the main rotor blades to the rotor; therefore these components are classified as critical "man-rated" parts. A comprehensive evaluation of extruded forging stock is required to qualify each and every production lot. This presentation will describe the development of the destructive and non-destructive test methodologies used, primarily tensile testing, electrical conductivity, chemical composition, reinforcement volume loading and ultrasonic inspection, as well as review archived data for current production material. Particular emphasis will be placed on the evolution of ultrasonic inspection to Class AA of MIL-STD-2154.

11:05 AM
Adaptation of Testing and Analysis for Discontinuously Reinforced Aluminum (DRA) Characterization: *Kenneth G. Davis*[1]; Cory A. Smith[1]; Mark R. van den Bergh[1]; [1]DWA Aluminum Composites, 21130 Superior St., Chatsworth, CA 91311 USA

Discontinuously Reinforced Aluminum (DRA) materials have been produced commercially for over 20 years for a variety of applications. The presence of the SiC particulate reinforcement in the aluminum matrix has required producers and end-users of DRA to adapt the tests and analyses used for routine characterization and acceptance. The necessary adaptations of five (5) areas will be discussed: 1) tensile testing, 2) chemical composition, 3) volume percent (v/0) analysis, 4) Metallography, and 5) verification of heat treatment. Additionally, comparisons to conventional aluminum alloy characterization will be made to illustrate the differences between the two classes of material.

The Mechanisms of the Massive Transformation - V

Sponsored by: ASM International: Materials Science Critical Technology Sector, Phase Transformations Committee
Program Organizers: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; V. K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Wednesday AM Room: Mississippi
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Barry C. Muddle, Monash University, Dept. of Matls. Eng., Victoria 3800 Australia

General Discussion

Under the chairmanship of Prof. Muddle, this session will provide time for more detailed discussions of the various papers presented in the preceding sessions. Comments bearing on multiple papers would be particularly appropriate during this session. The first portion of the General Discussion will be focused on the role of crystallography and interphase boundary structure in the massive transformation. The remainder of this session will be devoted to a discussion of whether the massive transformation can take place only within the single phase region of the product phase or, alternatively, can occur below T_o in the matrix + product region. Although achievement of general agreement in respect of the first topic seems unlikely, it is hoped that such can be secured about the second topic. Following the model used in the ASM Phase Transformations Committee's Hawaii conference on the shear vs. diffusion problems (H.I. Aaronson, J.P. Hirth, B.B. Rath, and C.M. Wayman, editors, "General Discussion Sessions of the 'Pacific Rim Conference on the Roles of Shear and Diffusion in the Formation of Plate-Shaped Transformation Products'", Metallurgical Materials Transactions A, 25A. 2655 (1994)), the General Discussion session will be recorded, transcribed, edited by discussers and organizers and submitted to Metallurgical Materials Transactions A as the "anchor paper" for the proceedings.

Fatigue and Fracture Behavior of High Temperature Materials - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Mechanical Behavior of Materials
Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Miinshiou Huang, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA

Wednesday PM Room: Mississippi
October 11, 2000 Location: Regal Riverfront Hotel

Session Chairs: P. K. Liaw, University of Tennessee, Knoxville, TN 37996-2200 USA; Walter W. Milligan, Michigan Technological University, Houghton, MI USA

2:00 PM
Mechanisms of High-Temperature Fatigue in Silicon Carbide Ceramics: *Robert O. Ritchie*[1]; Da Chen[1]; X. F. Zhang[1]; [1]University of California at Berkeley, Matls. Sci. and Eng. Depts., 463 Evans Hall #1760, Berkeley, CA 94720-1760 USA

Mechanisms of crack growth under cyclic loading in a monolithic silicon carbide ceramic are examined at elevated temperatures, with emphasis on the role of grain-boundary phases. It is shown that the damage and shielding mechanisms governing high-temperature fatigue-crack growth behavior up to ~1300°C are essentially unchanged from those at ambient temperatures. Specifically, the elastic and frictional tractions generated via the contact of opposing crack faces act to reduce the near-tip crack-driving force and hence retard subsequent crack extension. The basis for the cyclic component to fatigue-crack growth in these instances is the cycle-dependent, progressive suppression of such crack-tip shielding in the crack wake. The absence of creep-induced softening and cavitation in the grain-boundary glassy layer, together with the occurrence of viscous-ligament bridging, is attributed to the in situ crystallization of the grain-boundary phase, which was confirmed by high-resolution electron microscopy observations. It is concluded that ceramic fatigue at both low and high temperatures is critically affected by the nature of grain-boundary phase; mechanistically, fatigue-crack growth reflects a balance between intrinsic damage, in the form of intergranular cracking, high-temperature softening and creep cavitation within the glassy phase, and extrinsic crack-tip shielding from grain/viscous bridging. Supported by the Office of Science, Office of Basic Energy Sciences (Materials Sciences Division) of the Department of Energy.

2:20 PM
High Frequency FCP Behavior of a Nickel-Base Turbine Disk Alloy: *Santo A. Padula*[1]; Amit Shyam[1]; Walter W. Milligan[1]; [1]Michigan Technological University, Matls. Sci. and Eng. Depts., 1400 Townsend Dr., Houghton, MI 49931 USA

Fatigue crack propagation behavior and fatigue thresholds were measured for KM4, a nickel-base turbine disk alloy, at 550 and 650°C. A variety of frequencies (up to 1,000 Hz), load ratios, and microstructures were investigated. Threshold behavior was found to be a complex function of all variables. Mechanisms of threshold behavior will be discussed.

2:40 PM
Modelling the Effects of Grain Size and Grain Orientation Distributions on Short Fatigue Crack Growth: *Angus J. Wilkinson*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

A physically based model for the growth of a short fatigue cracks through a local grain structure is presented. The growth rate is evaluated using an existing solution for the distribution of dislocations ahead of a crack in a slip band that is blocked by a grain boundary. The effect of the local microstructure and microtexture are incorporated using orientation factors relating the applied far-field stress to the mode II shear loading of the crack tip. Probability distributions describing the grain sizes and orientations are used within a Monte-Carlo framework to calculate the growth rates of many individual cracks passing through different local microtextures. This allows not only the average behaviour of the short cracks, but also the statistical variation in the behaviour to be examined. Curves showing the probability of propagating cracks reaching a given crack length in a given number of cycles are presented.

3:00 PM
In-Situ TEM Investigation of a Crack Propagation in Single Crystal Ni3Al: *Xu Yongbo*[1]; Shan Zhiwei[1]; [1]Institute of Metal Research, Chinese Acad. of Sci., Shenyang 110015 China

The iniation and propagation of a crack in(110)[110] oriented Ni3Al single crystals have been studied by in-situ tension in TEM. The results show that the crack propagation appears to be in a zigzag manner and the main crack propagation orientation is along the tensile axis direction. The trace analysis shows that the two major slip planes, (111) and (111) are activated firstly, and then the other two primary slip planes, (111) and (111) are activated. A model was proposed to explain the observed results. The calculated results of the stress fields shows that the choice of the secondary slip system is dictated by stress concentration which arises from the dislocation pileup and makes the main crack propagation orientation parallel to the tensile axis.

3:20 PM Break

3:40 PM
Cyclic Fatigue-Crack Growth and Fracture Behavior of Mo-12Si-8.5B Intermetallics Both at Ambient and Elevated Temperatures: *Heeman Choe*[1]; Da Chen[1]; Joachim H. Schneibel[2]; Robert O. Ritchie[1]; [1]University of California at Berkeley, Matls. Sci. and Eng. Depts., 1 Cyclotron Rd., LBNL MS 62-203, Berkeley, CA 94720 USA; [2]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

Boron-containing molybdenum silicides have received some interest of late due to their superior low temperature "pest" resistance and a high-temperature oxidation resistance comparable to that of $MoSi_2$-based silicides, although like many intermetallics, they are generally plagued by poor toughness properties. Among various multiphase Mo-Si-B intermetallic systems available, Mo-12Si-8.5B (at.%), which contains Mo, Mo_3Si, and T2 phases, is expected to have somewhat higher fracture toughness because of the presence of the relatively ductile Mo phase. In this study, we examine the fracture toughness (R-curve) and fatigue-crack growth characteristics of this alloy, at temperatures from ambient to 1200°C, with the objective of discerning the salient mechanisms governing crack growth. It is found that this material displays quite high intrinsic (crack-initiation) toughness at 1200°C (exceeding 10 MPa$\sqrt{m}$), but with only limited extrinsic R-curve (crack-growth) toughening. Although the lack of extrinsic toughening mechanisms is not necessarily beneficial to quasi-static properties, it does imply that the material should show only minimal susceptibility to premature failure by fatigue. This relative insensitivity to fatigue was confirmed by fatigue-crack growth testing at temperatures up to 1200°C.

Research sponsored by the Office of Fossil Energy, Advanced Research and Technology Development (AR&TD) Materials Program, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

4:00 PM
Thermographic Detection of Fatigue Damage of Reactor Pressure Vessel (RPV) Steels: *Bing Yang*[1]; Peter K. Liaw[1]; Hsing Wang[2]; Liang Jiang[1]; Jiunn-Yuan Huang[3]; Roang-Ching Kuo[3]; J. G. Huang[4]; [1]The University of Tennessee at Knoxville, Matls. Sci. and Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research (INER), P.O. Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, 18f #242, Roosevelt Rd. Sec 3, Taipei 00142 Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 20 Hz and 1,000 Hz fatigue testing. Five stages of temperature profiles were observed: an initial decrease of the average specimen temperature, a followed temperature hump, and equilibrium (steady-state) temperature region, an abrupt increase of the temperature, and a drop of temperature following specimen failure. The relationship among the temperature, stress-strain state, and fatigue behavior is discussed. Both thermodynamics and heat transfer theories are applied to model the observed temperature variation during fatigue. The predicted and measured temperature evolutions during fatigue were found to be in good agreement.

4:20 PM
Effect of Frequency and Specimen Self-Heating on the Fatigue Life of 316 LN Stainless Steel: *Hongbo Tian*[1]; Peter K. Liaw[1]; Hsin Wang[2]; Douglas E. Fielden[1]; Liang Jiang[1]; Bing Yang[1]; Charlie R. Brooks[1]; Joseph P. Strizak[2]; Louis K. Mansur[2]; James R. DiStefano[2]; Kenneth Farrell[2]; Steven J. Pawel[2]; George T. Yahr[2]; [1]The University of Tennessee at Knoxville, Matls. Sci. and Eng. Dept., 434 Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

During high-cycle fatigue tests, the temperature on specimens may rise up to different levels at different frequencies due to the thermoelastic and inelastic phenomena. Temperature can reach 275°C on 316 low-carbon nitrogen-contained (LN) stainless steel (SS) during 700 Hz fatigue tests, and the temperature increase can reduce the fatigue life of 316 LN SS in air and mercury environments. Recent work on the frequency effect in air and mercury environments by controlling the specimen temperature with cool nitrogen gas revealed that the temperature increase induced by specimen self-heating played an important role in determining the fatigue life of 316 LN SS. Acknowledgements: This research was sponsored by the Division of Materials Sciences, US Department of Energy under contract DE-AC05-96OR22464 with the Lockheed Martin Energy Research Corporation. The authors acknowledge Dr. L. Chen and Dr. Y. He for their careful instructions and helpful suggestions on the fatigue tests. We also acknowledge the financial support of the National Science Foundation, the Division of Design, Manufacture, and Industrial Innovation, under Grant No. DMI-9724476, and the Combined Research-Curriculum Development Program, under EEC-9527527, to the University of Tennessee, Knoxville, with Dr. D. R. Durham and Ms. M. Poats as program managers, respectively. We appreciate the financial support of the Center for Materials Processing and Office of Research Administration at the University of Tennessee, Knoxville.

4:40 PM
Orientation Dependence of Directional Coarsening Behavior in a Nickel-Base Single Crystal Superalloy: *Xu Yongbo*[1]; Sha Yuhui[1]; Zhang Jinghua[1]; [1]Institute of Metal Research, Chinese Acad. of Sci., Shenyang 110015 China

Finite element analysis was applied to the amplitude and distribution of internal stress before and after loading. The results showed that the stress distribution in matrix channels is closely related with the directional coarsening, and the driving force for directional coarsening is the anisotropic relaxation of lattice misfit resulted from the inhomogeneous distribution of plastic deformation.

General Abstracts: Alloy Phases: Alloy Phases

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Wednesday PM Room: Jefferson A
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Dan Branagan, Bechtel BWXT Idaho LLC, Idaho Nat. Eng. and Environ. Lab., Idaho Falls, ID 83415-2218 USA

2:00 PM
Chemical Bonding by Metallic Covalentivity: *William F. Smith*[1]; [1]University of Central Florida, Dept. of Mech. Matls. and Aero. Eng., Alafaya Trail, Orlando, FL 32816-2450 USA

Linus Pauling has left us with an electronegativity table which appears in most general chemistry books and is associated with ionic-covalent bonding. It is proposed that this idea be extended to covalent-metallic bonding also. By using covalent carbon (diamond) which melts at 3500°C and which is assumed to be 100% covalent bonding, assigning this melting point 4.0 on the C-M scale. In this manner the C-M values of pure iron which melts at 1535°C would be assigned a value (C-M) 1.75. In this manner we could explain the low strength and metallic behavior of potassium vs. the higher strength and metallic behavior of copper. A Table of proposed C-M values will be presented. It is sad that Linus is not with us to favor this extension of his bonding work. It is also proposed that electronic structure be substituted for valence in the famous four rules for solid solubility proposed by Professor Hume-Rothery.

2:20 PM +
Phase Transformation in Ti-6Al-4V-xH Alloys: Jawad Rahim[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; *Javaid I. Qazi*[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., Mines Bldg. Rm. 321, Moscow, ID 83844-3026 USA

Ti-6Al-4V alloy samples were alloyed with 10, 20 and 30 at.% hydrogen by holding the samples in hydrogen atmosphere with different hydrogen partial pressures at 780°C for 24 hours. Hydrogen being strong b stabilizer has increased the amount of β phase at room temperature. An increase of the hydrogen content from 0 to 30at.% decreased the β to α+β transus temperature from 1000°C to 815°C. The beta transus temperatures were determined for all four compositions by microstructural analysis. TTT diagrams for decomposition of metastable beta and martensitic phases have also been determined using microstructural analysis and X-ray diffraction technique. 30at.% hydrogen has increased βTTT nose time from ~12seconds to ~16minutes. The nose temperature has also been lowered for 30at.% hydrogen from ~725°C to 580°C. Using the results obtained, conditions of heat treatment of the hydrogenated samples were optimized leading to a refined microstructure and improved mechanical properties after dehydrogenation.

2:40 PM
Identification of a New Tetragonal Phase in the Nb-Ti-Al System: *Keith J. Leonard*[1]; Joseph C. Mishurda[1]; Bryan Molloseau[2]; Marc De Graef[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. and Eng. Depts., 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA; [2]Carnegie Mellon University, Matls. Sci. and Eng. Depts., Pittsburgh, PA 15213 USA

During the course of investigation of the phase equilibria in the Nb-Ti-Al system, a new phase, designated θ, was observed in three Nb-Ti-40Al (at.%) alloys following aging at low temperatures (<1000°C). Using X-ray diffraction and transmission electron microscopy techniques, this phase was determined to have a body-centered tetragonal structure, with lattice parameters of a_o=5.106 and c_o=28.168 angstrom, space group of $I4_1/amd$ (#141) and composition near 45Nb-25Ti-30Al. Neutron diffraction and high-resolution electron microscopy were also used to determine atomic site occupancies. These results are presented and discussed.

3:00 PM
Coarsening Kinetics and Morphology of Ni3Al Precipitates in Alloys Aged Under Uniaxial Compression: Sergey Prikhodko[1]; *Alan J. Ardell*[2]; [1]University of California, Matls. Sci. and Eng. Depts., 6532 Boelter Hall, Los Angeles, CA 90095-1595 USA; [2]University of California, Matls. Sci. and Eng. Dept., 6531-G Boelter Hall, Los Angeles, CA 90095-1595 USA

Coarsening of Ni_3Al precipitates at 640°C under uniaxial compression was investigated in [001]-oriented monocrystals of a Ni-13.36Al alloy. The specimens were doubly tapered cylinders, with stresses varying from 30 to ~130 MPa and aging times to 1021 h, or cylindrical cylinders aged for 120 h under stresses from 0 to 150 MPa. Microstructures were examined by transmission electron microscopy in thin foils oriented [001]. The average particle radius, aspect ratio, and a shape parameter were measured. In general the applied stress retards the kinetics of coarsening, and the exponent n increases with applied stress (n = 3 at zero stress). Coarsening kinetics are accelerated in doubly-tapered specimens at intermediate stresses. Applied stress promotes the loss of 4-fold symmetry and stimulates the occurrence of groupings commonly associated with splitting. These observations are considered in light of current theoretical considerations. This research is sponsored by the Department of Energy.

3:20 PM
The Volume Fraction Dependence of Ni3Ge Coarsening Kinetics: Dongman Kim[1]; *Alan J. Ardell*[1]; [1]University of California, Matls. Sci. and Eng. Depts., 6532 Boelter Hall, Los Angeles, CA 90095-1595 USA

Coarsening kinetics of Ni_3Ge precipitates were investigated in alloys containing 12.15, 13.02 and 14.03 at. % Ge aged at 724°C. The volume fraction, f, varied from ~0.02 to 0.20. The purpose was to determine whether coarsening of Ni_3Ge is anomalous, i.e. whether the rate constant for the kinetics of growth decreases with increasing f in the low f regime (< 0.04). Strong anomalous coarsening behavior is found in Ni-Al and Ni-Ti alloys, but is nearly absent in Ni-Ge alloys despite the relatively large lattice mismatch (0.0063, cf. 0.0047 and 0.0085, respectively). Instead, the behavior of Ni_3Ge and Ni_3Si precipitates is similar, despite the much smaller Ni_3Si lattice mismatch (-0.0023). Possible reasons for this will be discussed, as will other aspects of coarsening behavior in Ni-Ge alloys, such as the kinetics of solute depletion of the matrix and particle size distributions. This research is sponsored by the National Science Foundation.

3:40 PM Break

4:00 PM
Influence of Cold-Rolling on the Devitrification of Al-Based Marginal Glass Formers: *Rainer J. Hebert*[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Matls. Sci. and Eng. Dept., 1509 University Ave., Madison, WI 53706 USA

Results are presented for melt-spun Al-Sm, Al-Y-Fe and Al-Ni-Y marginal glass-forming alloys that elucidate the impact of cold-rolling on quenched in nuclei and primary crystallization. For the Al-Sm system rolling can induce the crystallization of an intermetallic phase and a reduction of primary crystallization until a fully crystalline structure is obtained yielding essentially the same phases as obtained by annealing of the as-spun material. Rolling of the ternary systems reveals the crystallization of mainly metastable crystalline phases. Moreover, the experiments show that changes in the processing conditions are sufficient to change the crystallization pathway. Based on the possibility that the short range order in the amorphous phase is correlated to the structure in the melt prior to quenching, it appears that the crystallization pathway can be tailored by manipulating the melt-spinning conditions. Aside from providing new insight into the devitrification behavior of metallic glasses a comparison with rolling of crystalline multilayers will show that these results are also relevant for a more complete understanding of the rolling induced crystalline to amorphous transformation. The support of the ARO (DAAG 55-97-1-0261) is gratefully acknowledged.

4:20 PM
The Catalytic Effect of Pb on the Nanocrystallization in Al-Based Amorphous Alloys: *Robert I. Wu*[1]; Zhenfu Dong[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Matls. Sci. and Eng. Dept., 1509 University Dr., Madison, WI 53706 USA

Al-based alloys containing transition and rare earth elements with a finely mixed microstructure of dispersed Al nanocrystals in an amorphous matrix have attracted considerable attention for their superior mechanical properties compared with conventional Al-based alloys. Through primary crystallization of amorphous melt-spun ribbons, an ultrahigh number density ($> 10^{20}$ m^{-3}) of Al nanocrystals (~20nm in diameter) form within the amorphous phase and provide effective dispersion strengthening. The properties can be enhanced further by increasing the number density of Al-nanocrystals. An approach to attain this goal has been developed through the incorporation of insoluble Pb in the amorphous matrix. It is discovered that in $Al_{87}Y_7Fe_5Pb_1$ melt-spun ribbons, nano-sized Pb crystalline particles in the amorphous matrix are effective in catalyzing the crystallization of Al-nanocrystals and yield an increase in the total number density of nanophases by an order of magnitude. Microstructure analyses is utilizing high resolution transmission electron microscopy (HRTEM), energy dispersive x-ray (EDX) analyses, and XRD have been carried out on annealed melt-spun ribbon samples. DSC has been applied on melt-spun ribbon samples to probe into the thermal response and kinetics of primary crystallization of Al-nanocrystals with the presence of Pb. The HRTEM observation of the co-existence of the lattices for Al and Pb within one particle suggests that Al nanocrystals tend to precipitate at the Pb particle/amorphous matrix interface. The catalytic effect of Pb upon nanocrystallization along with the detail results of the HRTEM analysis will be presented. The support of the ARO (DAAG55-97-1-0261) is gratefully acknowledged.

4:40 PM +
Perspective Systems of Ion-Plasma Coatings: *Tatiana L. Loumacheva*[1]; Alexey V. Sverdlin[2]; Valery I. Bogdanovich[3]; Vitaly A. Barvinok[3]; [1]Bradley University, Mktg. Dept., 1530 W. Bradley Ave., Peoria, IL 61625 USA; [2]Bradley University, Indust. and Manufact. Eng. and Tech., 1501 W. Bradley Ave., Peoria, IL 61625 USA; [3]Samara State Aerospace University, Dept. of Aircrafts, Gaya St. 59, Samara 443086 Russia

This study deals with possibilities of vacuum ion-plasma technology for production of coatings with anomaly in microhardness and melting points values. These aspects are considered on the basis of extreme characteristics of carbide and nitride systems to be the result of the composition of certain amount of valence electrons per atom in metal lattice. The study discusses the perspectives in production of new types of coatings for different applications.

General Abstracts: Ferrous Metallurgy

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Howard W. Sizek, Special Metals Corporation, Dunkirk, NY 14048 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Richard N. Wright, Idaho National Engineering Laboratory, Idaho Falls, ID 83415-2218 USA

Wednesday PM Room: Field
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Karol Schrems, U.S. Department of Energy,Matls. Conserv. Div., Albany Rsch. Ctr., Albany, OR 97321 USA

2:00 PM
Mechanical Properties of Thin-Wall Ductile Iron: *Karol K. Schrems*[1]; Omer N. Dogan[1]; Jeffrey A. Hawk[1]; Alan P. Druschitz[2]; [1]U.S. Department of Energy, Matls. Conserv. Div., Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]Intermet Corporation, Matls. Dev., 939 Airport Rd., Ste. 200, Lynchburg, VA 24502-3506 USA

The use of cast iron in automotive applications in this era of increasing fuel efficiency requires the ability to cast very thin sections (2-7 mm). Although thin-wall iron castings have been produced, difficulty arises in predicting the mechanical properties of these castings because mechanical behavior is closely related to thickness, which in turn is a direct consequence of the section cooling rate. Experiments relating casting thickness with ultimate tensile strength, elongation, reduction in area, and hardness were performed. An inverse relationship was found between ultimate tensile strength and thickness. Elongation was found to depend only on the thickness of the sample and approached zero as the thickness of the sample decreased below 1.5 mm. Percent reduction in area was found to depend linearly on thickness. Although average hardness also correlated with the inverse of thickness, it was not found to be a useful measure of ultimate tensile strength. The results of this study show that cooling rate of the thin wall casting very much affects the mechanical properties.

2:20 PM
Microstructure of Thin-Wall Ductile Iron Castings: *Omer N. Dogan*[1]; Karol K. Schrems[1]; Jeffrey A. Hawk[1]; Alan P. Druschitz[2]; [1]U.S. Department of Energy, Matls. Conserv. Div., Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]Intermet Corporation, Matls. Dev., 939 Airport Rd., Ste. 200, Lynchburg, VA 24502-8749 USA

The automotive industry is seeking to replace current car parts made of aluminum and iron castings with thin wall (down to 2 mm) iron castings to reduce the cost and weight of automobiles. The mechanical properties of thin wall ductile iron castings are affected strongly by the thickness of the castings. The thinner castings cool at a faster rate, and microstructural features that form during solidification, and subsequently, transform in the solid state, are strongly dependent on a geometrical parameter related to

the ratio of surface area-to-volume of the casting. As this ratio becomes larger, castings cool faster. As a result, the nodule count on the observation plane of the specimens increases dramatically (>2000 nodules/mm2 in most specimens), i.e. as the thickness of castings decreases. Also, the matrix of the thin walled ductile iron castings becomes more ferritic as the ratio of surface area-to-volume decreases.

2:40 PM
Texture Development during Annealing of a Cold Rolled Magnetic Lamination Steel: *Soonwuk Cheong*[1]; Anthony D. Rollett[1]; Erik Hilinski[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng. Depts., Wean Hall 3325, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Texture development during the final annealing step (the Quality Development Anneal) of a cold rolled magnetic lamination steel is being investigated. Previous work conducted on a 0.3Si and 0.6Mn cold roll magnetic lamination steel has shown that the magnetic properties and the texture are sensitive to the processing, more specifically to the temper rolling reduction and the Quality Development Anneal. Orientation Imaging Microscopy (OIM) by means of electron back scatter diffraction patterns has been used to characterize the relationship between the growing grains and the matrix in partially (secondary) recrystallized material. As has been observed in the Fe-3Si system, the grain boundaries of the Goss grains have a higher frequency of Σ9 boundaries than is present in the matrix. The presence of Goss-related components causes pronounced anisotropy in the magnetic and elastic properties. The OIM analysis techniques used to characterize the grain boundary orientation relationships in the 0.3Si and 0.6Mn steel has been extended to a steel with a composition of 0.003C, 0.6Mn, 1.1Si, 0.31Al and 0.003N (weight %). Laboratory temper rolling experiments have been performed on a mill coil that was conventionally processed through batch annealing and then slit into mults. Variations in temper rolling included reductions between 2 and 10%, the number of passes to attain a given reduction, roll roughnesses between 20 and 180 µin, and variable front and back tension. Characterization methods including optical metallography, X-ray pole figure measurement, Epstein pack measurement along with OIM using electron backscatter diffraction pattern analysis in a scanning electron microscope have been used to compare the microstructure, texture, and grain boundary orientation relationships between these two steels.

3:00 PM Break

3:20 PM
On the Growth of Grain Boundary Ferrite in Fe-C-X Steels: *R. E. Hackenberg*[1]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., 116 Engineer's Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA

The growth kinetics of grain-boundary nucleated ferrite (with or without carbides) in steels is not well understood. Traditional growth kinetics determinations focused on the thickness measurement of the thickest grain boundary allotriomorph in a given sample, with data from different times plotted on a log-log scale to determine the thickening constant and time exponent. Samples measured in this fashion must be carefully heat treated to render the austenite grain boundaries perpendicular to the surface of measurement, to avoid stereological errors. A new technique, involving three-dimensional sectioning to determine the angle of the grain boundary to the plane of polish, is demonstrated. This is applied to Fe-0.24C-4.00Mo isothermally transformed at its bay temperature, where data from the previous method showed a time interval of growth stoppage. Factors which may affect the correct interpretation of this data, especially crystallography, interfacial structure and carbide precipitation, are discussed.

3:40 PM
Effects of Nickel Content and Heat Treatment on the Strength and Toughness of a 12Cr/12Co/5Mo Martensitic Precipitation Strengthened Stainless Steel: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng. Dept., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In this work, nickel additions of 4.5, 5, 5.5 and 6.0 wt.% have been made to a 0.01C /12Cr/12Co/5Mo base composition in order to study the effects of nickel on age-hardening response and on the ductile-to-brittle transition behavior as determined by Charpy impact energies. It has been found that nickel additions of up to 5.5 wt.% enhance the age-hardening response and, at the highest level, lower the aging temperature at which the peak yield strength is observed. It was also found that the ductile-to-brittle transition temperatures are decreased significantly after aging at both 500°C and 525°C when the nickel content is increased to 6 wt.%. Austenite contents were determined by X-ray for the four alloys as a function of aging temperature and the results indicate that the decreased ductile-to-brittle transition temperatures associated with the highest nickel content are due to this alloy containing substantially more austenite than the other three alloys. The precipitation processes in this system have been investigated as a function of aging temperature for the 4.5 wt. % alloy and the results show that R-phase is formed at aging temperatures of 550°C and higher but that another precipitate type may be responsible for age-hardening at lower aging temperatures where the highest yield strength strengths are observed.

Interfacial Dislocations: Symposium in Honor of J.H. Van der Merwe on the 50TH Anniversary of His Discovery - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structures
Program Organizers: Gary J. Shiflet, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA; William A. Jesser, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA

Wednesday PM Room: Missouri
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: F. R.N. Nabarro, University of Witwatersrand, Dept. of Phys., Johannesburg 0001 South Africa

2:00 PM
Diffusional Accommodation of Misfit at Dislocation Interfaces in Alloys: *John W. Cahn*[1]; [1]NIST, Matls. Sci. and Eng., Gaithersburg, MD 20899-8555 USA

Diffusional accommodation in alloys creates compositional inhomogeneities around single dislocations, as e.g. in a Cottrell atmosphere, and around the dislocations in an interface. The elastic free energy is reduced if the atoms that expand or contract the lattice concentrate in the tensile or compressive regions respectively; this reduction is balanced by the free energy changes accompanying the concentration changes. Equations are given for the elastic accommodation in the neighborhood of a the dislocation interface and the composition changes. When the equations are linearized, the use of the Larché-Cahn open system elastic constants results in quick estimates for the composition and elastic fields and for most of the interfaces examined by van der Merwe where continuum concepts apply, when extended to many types of multicomponent alloys. Nonlinear effects become important at the dislocation cores and for phases near phase transitions. The linear theory gives estimates of reduction of the interfacial energy from this accommodation, but a nonlinear theory must be used to achieve consistency with the Gibbs adsorption equation.

2:35 PM
Misfit Compensating Mechanisms during Lamellar Phase Transformations: *Gary J. Shiflet*[1]; Chris Hutchinson[1]; Kenji Matsuda[2]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., Charlottesville VA 22903-2442 USA [2]University of Toyama, Dept. of Matls. Sci., Toyama, Japan

This talk will discuss the role of misfit compensating dislocations on interfacial structure and subsequent phase transformations. This is an area that Professor van der Merwe has had such a tremendous influence over the past 50 years. These defects will be examined in the context of growth and development of phase transformation plates. Plates will include those occurring separately and those forming during lamellar transformations in pearlite and discontinuous transformations. Plates examined include g and g' in Al-Ag alloys. It will be shown that the appearance of misfit compensating dislocations is concurrent with a change of silver concentration in g. Likewise, the misfit dislocations have a significant and deleterious effect on the coarsening of W in Al-Cu-Mg-(Ag) alloys. In lamellar phase transformations, discussion of the misfit dislocation patterns that can occur when combined with structural ledges and direction steps. The National Science Foundation is acknowledged for support.

3:10 PM
Comparison of Central Interfacial Structure Mechanisms in Diffusional and Martensitic Transformations: *H. I. Aaronson*[1]; J. P. Hirth[2]; B. C. Muddle[3]; J. F. Nie[3]; W. Z. Zhang[3]; [1]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Road, Columbus, OH 43210 USA; [3]Monash University, Dept. of Matls. Eng., Mail Code 00139, Clayton, VIC 3800 Australia

Some central problems in understanding the similarities and the differences between ledgewise martensitic and ledgewise diffusional growth are examined. Martensitic growth can be described in terms of a lattice correspondence and a plane undistorted by the shear transformation. Diffu-

sional growth can be similarly described in some cases but not in others. Only misfit dislocations (on terraces or risers) or orthogonal sets of disconnections provide a truly sessile interface. Closely spaced structural ledges (disconnections) present during diffusional growth must have been mobile during early growth stages. Once these ledges are in local equilibrium, they can only move synchronously because of their local strain interaction; such concerted movement by diffusional processes is unlikely. Under these circumstances extrinsic sources of growth ledges are required to move such interfaces in a diffusional manner. During martensitic growth, however, disconnections (transformation dislocations) can move freely in a synchronous manner.

3:45 PM Break

4:00 PM
Role of Interface Structure and Interfacial Defects in Oxide Scale Growth: *Robert A. Rapp*[1]; Bernard Pieraggi[2]; John P. Hirth[3]; [1]Ohio State University, MSE Dept., Columbus, OH 43210 USA; [2]INP-ENSC, Matls. Lab., Toulouse, France; [3]Washington State University, Dept. Mech. & Matls. Eng., Pullman, WA USA

The action of interfacial defects (misfit dislocations, misorientation dislocations and disconnections) in the creation and annihilation of the point defects supporting the diffusional growth of scales on metals and alloys is considered. Scales grown by dominant anion diffusion and those grown by dominant cation diffusion are treated. The consequences of blocking the interfacial reaction step, e.g. by dislocation pinning through the adsorption of large, highly charged impurity ions, may explain the important "Reactive Element Effect" in scale growth on metals.

4:35 PM
Misfit Dislocation Introduction in Large Lattice Mismatched Epitaxy: Vidyut Gopal[1]; *Eric P. Kvam*[1]; [1]Purdue University, Matls. Eng. Dept., 1289 MSEE Bldg., W. Lafayette, IN 47907-1289 USA

Large mismatch InAs/GaP exhibits strain relaxation which differs from low misfit systems. TEM imaging showed initial island growth followed by rapid coalescence. Stress concentrations from initial vertical (Stranski-Krastanov) growth caused introduction of edge dislocations at/near the island perimeter, allowing a reduction in island aspect ratio (height:width). After continuous layer formation at a nominal thickness of 5 nm, direct edge dislocation introduction was no longer possible. Further relaxation occurred by pairs 60° dislocations, which were complementary and thus reacted to form interfacial edge dislocations. By 30 nm thickness, strain relaxation was complete, and the mean edge dislocation spacing was 3.85 nm. Further growth or annealing homogenized but did not reduce this spacing. (In,Al)As capping layers atop the InAs layer exhibited their own unique misfit dislocation introduction at the cap/layer interface. Only edge segments were observed, introduced by climb rather than conventional glide mechanisms.

Microcharacterization and Microtexture - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee
Program Organizers: Dierk Raabe, Max Planck Institut fuer Eisenforschung, Department for Microstructure & Metal Forming, Duesseldorf 40237 Germany; Wei Tong, Yale University, Department of Mechanical Engineering, New Haven, CT 06520-8264 USA; Hasso Weiland, Alcoa Technical Center, Alcoa Center, PA 15069 USA

Wednesday PM Room: Meramac
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Dierk Raabe, Max-Planck-Institut fuer Eisenforschung, Dusseldorf 40237 Germany

2:00 PM
Experimental Investigation of Microtexture and Microstrains: *M. Sachtleber*[1]; Z. Zhao[1]; H. Faul[1]; H. -G. Sattler[1]; D. Raabe[1]; [1]Max-Planck-Institut fur Eisenforschung, Max-Planck-Str. 1, Dusseldorf 40237 Germany

We present microtexture experiments on polycrystalline samples in conjunction with microstrain experiments. We aim at correlating kinematics and micromechanics pertaining to the various crystals with the accessible components of the local displacement vector and strain tensor measured at the sample surface. We use 2D and 3D digital image surface characterization and analysis methods based on stochastic pattern recognition before and after straining. The microtexture experiments are conducted using the SEM EBSP method. The deformation experiments are conducted using a plane strain compression set-up with solid lubrication.

2:20 PM
Slip System Analysis at Free Surface of Aluminum Alloys: *Hasso Weiland*[1]; L. G. Hector[1]; W. Tong[2]; [1]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]Yale University, Dept. of Mech. Eng., New Haven, CT 06520-8264 USA

The deformation behavior of individual grains within a polycrystalline aggregate controls the visual appearance of the material when it is plastically deformed. Quite often a visual defect on a metal surface known as "orange peel" results from free grain roughening during tensile deformation. A fundamental understanding of this type of defect requires an understanding of the microstructural attributes that contribute to the development of grain roughening during plastic deformation. Specifically, it is necessary to understand how slip is distributed within the grains, how differences in slip activity between neighboring grains affect the respective slip in each grain, and how all these effects are influenced by the type of solid solutions present in the material matrix. Several binary aluminum alloys were fabricated with a columnar grain structure. Subminiature tensile samples were prepared such that the long grain direction was normal to the sheet plane. Hence, each grain was extended from one sample surface to the other. The grain structure was characterized by Orientation Imaging Microscopy (OIM). The samples were subsequently strained in-situ in an Atomic Force Microscope (AFM). Changes in the slip step topography on grains of known crystallographic orientation were measured in the AFM. Analysis of the AFM scans gave an indication of the active slip systems on each grain as well as the magnitude of slip occurring on each slip plane. From these data, local surface Taylor factors were determined.

2:40 PM
Theoretical and Experimental Investigation on Grain Interaction: *J. Park*[1]; Z. Zhao[2]; M. Sachtleber[2]; K. Oh[1]; D. Raabe[2]; [1]Seoul National University, Sch. of Matls. Sci. and Eng., College of Eng., Seoul 151-742 Korea; [2]Max-Planck-Institut fur Eisenforschung, Max-Planck-Str. 1, Dusseldorf 40237 Germany

We present theoretical simulations of selected grain-grain interaction configurations based on a viscoplastic crystal plasticity finite element formulation. We aim at elucidating the influence of grain orientation on kinematic stability, i.e. on reorientation rate, shape change, and hardening behavior during deformation. Selected grain pair interactions are also investigated by experimentation using microtexture determination and microstrain measurement.

3:00 PM Break

3:20 PM
Effect of Alloying Element and Grain Orientation During Single Asperity Plowing of Aluminum Alloys: S. M. Opalka[1]; *Hasso Weiland*[1]; L. G. Hector[1]; S. R. Schmid[2]; [1]Alcoa Technical Center, Alcoa Center, PA 15069 USA; [2]University of Notre Dame, AME Dept., Notre Dame, IN USA

The abrasive action in a tooling/workpiece interface at the scale of a single asperity is simulated with an atomic force microscope (AFM). A sharp diamond tip, which is affixed to a stainless steel cantilever, is directed to plow the surface of selected aluminum alloys. The AFM piezos control the cantilever speed and normal force applied to the surface through a software macro. This leaves a plow track consisting of a groove and displaced material, or ridges, along the banks of the groove. The track length is typically 10 mm which is smaller than the average grain size of wrought aluminum products. Consequently, such an analysis is restricted to areas within a single grain. The purpose of this work is to quantitatively characterize the degree to which the crystallographic planes of the underlying crystal structure affect the surface flow strength. For several binary solid solutions of aluminum, the tip was directed to plow along 001, 011, and 111 directions of selected grains. The grain orientations were determined prior to the plowing experiments from Orientation Imaging Microscopy (OIM) analysis. The resulting plow tracks were analyzed for average ridge height, ridge angles, and groove depth. Along with the cantilever parameters of the AFM tip and the tip geometry, the surface flow strength was calculated. The results show that alignment of the plow directions with the crystallographic direction affects the value of the flow stresses determined. Additionally, the experiments showed a dependence of the flow stress on the elements in solid solution, which is different in magnitude for the selected crystallographic directions.

3:40 PM
Microstructural and Micromechanical Characterization of LIGA Fabricated Nickel: *Thomas E. Buchheit*[1]; Joseph R. Michael[2]; David A. LaVan[3]; Todd R. Christenson[4]; Steven D. Leith[5]; [1]Sandia National Laboratories, Dept. 1835, MS: 0333, P.O. Box 5800, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 1822, MS: 1405, P.O. Box

5800, Albuquerque, NM 87185 USA; [3]Sandia National Laboratories, Dept. 1835, MS: 1407, P.O. Box 5800, Albuquerque, NM 87185 USA; [4]Sandia National Laboratories, Dept. 1713, MS: 0603, P.O. Box 5800, Albuquerque, NM 87185 USA; [5]Sandia National Laboratories, Dept. 8729, MS: 9401, P.O. Box 969, Livermore, CA 94551 USA

LIGA micro-fabrication utilizes an additive process in which structural material is electrodeposited into a precision mold of PMMA realized through deep x-ray lithography. This fabrication method is regarded as a microelectromechanical system (MEMS) technology as representative dimensions of LIGA components range from a few microns to several millimeters. A consequence of these small length scales is that material microstructural features, such as grain size, can approach the critical dimensions of the components themselves. In addition, LIGA components and materials share a characteristic common to all electrodeposited films; their microstructure and mechanical properties are very sensitive to several electroplating processing variables including bath chemistry and agitation, applied current density (and waveform) and feature geometry. These issues require a detailed and careful approach to LIGA materials characterization. To understand and control the processing-microstructure-properties relationships in LIGA electroformed nickel, a concurrent mechanical testing-microstructural analyses study was conducted using a mini-servohydraulic load frame and the Electron Backscattered Diffraction (ESBD) measurement technique. This study focussed on LIGA-fabricated nickel parts plated from two common bath chemistries, a Watts bath and a Nickel Sulfamate based bath. The variation in microstructure and properties of the nickel with annealing treatments was also characterized to determine the viability of high temperature diffusion bonding as a method of assembly for LIGA structures. The electroformed structures were found to have fine grain, highly textured microstructures oriented with a lenticular or columnar morphology relative to the plating direction. Previously uncharacterized, anomolous local spatial variations in the crystallographic texture of the as-deposited microstructures were identified by ESBD analyses and will be presented. Also, microstructure characterization results will be presented from the annealing study that reveal a recovery, recrystallization, rapid anomalous grain growth microstructural evolution mechanism. The evolution of microstructure in the annealed samples corresponded with a dramatic drop in strength of the LIGA samples and determined the limiting diffusion bonding temperature for LIGA-fabricated nickel components. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

4:00 PM
Effects of Grain Boundary Crystallography on the Retardation of Short Fatigue Cracks: Tongguang Zhai[1]; John W. Martin[1]; *Angus J. Wilkinson*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

This paper presents a crystallographic model for the retardation of short fatigue crack propagation at grain boundaries. Experimental observations of short fatigue crack growth in Al-Li 8090 alloys used to construct the model will be given. The variation in crack growth rate as the crack tip passes different grain boundaries was measured using interrupted 4 four point bend fatigue tests and replication methods. The crystallographic orientations of grains along the crack path were measured using electron backscatter diffraction. The cracks were found to propagate mainly on {111} planes, with high but not necessarily maximal Schmid factors. The crack plane deflections at grain boundaries could be resolved into tilt and twist components. Successful propagation of the crack across a grain boundary was onto the slip plane that minimised the twist deflection. Grain boundaries imposing large crack twist deflections tended to show a strong retardation of the short fatigue crack.

Powder Metallurgy Alloys and Particulate Materials for Industrial Applications: Nickel Alloys and Intermetallics - III

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: David E. Alman, US Department of Energy, Albany, OR 97321 USA; Joseph W. Newkirk, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday PM Room: Clark
October 11, 2000 Location: Regal Riverfront Hotel

Session Chairs: David E. Alman, U.S. Department of Energy, Albany Research Center, Albany, OR 97321 USA; J.-Y. Chen, National Research Council Canada, London, Canada

2:00 PM Invited
Manufacturing Constraints for Industrial Applications of P/M Superalloys: *Anthony Banik*[1]; [1]Special Metals Corporation, 100 Industry Ln., Princeton, KY 42445 USA

Traditional powder metal superalloy applications have been focused on high temperature rotating components in military turbine engine applications while conventional ingot metallurgy products have been widely used in commercial and industrial applications. Military turbine engine applications demand extremely rigid process control standards to assure the powder integrity is maintained throughout processing. A comparison between ingot metallurgy and powder metallurgy process routes for turbine components will be presented. Process control methodologies imposed for the powder metallurgy aerospace applications provide a means to meet the production demands for industrial applications. Therefore, powder testing methodologies and the applications to production alloys will also be reviewed.

2:25 PM Invited
Industrial Applications of Intermetallic Alloys: *V. K. Sikka*[1]; M. L. Santella[1]; [1]Oak Ridge National Laboratory, Mets. and Cers. Div., P.O. Box 2008, Oak Ridge, TN 37831 USA

Intermetallic, especially the aluminides of nickel, iron, and titanium have reached the stage of significant advance for them to be considered for a variety of applications. This paper will present the advances made to date in the compositions, processing, properties, and fabrication of nickel and iron aluminides. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

2:50 PM Invited
Plasma Spray-Forming of Advanced Silicide-Based Materials for High Temperature Industrial Applications: *Richard G. Castro*[1]; Rajendra U. Vaidya[1]; John J. Petrovic[1]; Kendall J. Hollis[1]; [1]Los Alamos National Laboratory, Matl. Sci. and Tech. Depts., P.O. Box 1663, Mail Stop G741, Los Alamos, NM 87545 USA

Plasma spray forming of MoSi2-base materials is being investigated as method for producing near-net-shape components for applications in glass, heat-treating and melt processing industries. Plasma spray processing allows direct powder to product consolidation of advanced materials that are difficult to consolidate by other processing methods. The environmental performance of plasma spray-formed MoSi2-based materials has been investigated in molten aluminum, copper and alkali borosilicate glass and in combustion and endothermic (40% H2, 40% N2 and 20% CO) heat treating environments. Information will be presented on the performance of MoSi2-based materials in these industrial environments and how it compares to other refractory materials that are currently being used.

3:15 PM +
Examination of Cr-(CrxMoy)3Si Alloys for Industrial Applications: *Martin Gerardo Perez*[1]; Joseph W. Newkirk[1]; [1]University of Missouri-Rolla, Metall. Eng. Dept., 223 McNutt Hall, Rolla, MO 65409 USA

Refractory metal silicides have many desirable properties such as a high melting point, good corrosion resistance, low densities, and low wear rates. Ganapathi and Newkirk have successfully produced a series of Cr-Cr3Si-NbC alloys with hardness values between 956 and 1338 Hv and corrosion rates in sulfuric acid of 50 to 121 mpy, but with a fracture toughness of only 3 to 6.6 MPa-m-1/2. Cruse and Newkirk demonstrated that the fracture

toughness of the Cr-Cr3Si alloys can be improved to 8.4 MPa-m-1/2 by the addition of molybdenum. A series of alloys have been created in an attempt to improve on wear, corrosion resistance, hardness and fracture strength. The new alloy series will be a Cr- (CrxMoy)3Si compound with 5, 10, and 15 wt.% NbC additions. All alloys were created via a powder metallurgy route. Cr, Mo, Si and NbC powders were mechanically alloyed and then hot pressed to produce the alloys.

3:35 PM Break

3:50 PM Invited
An Assessment of Powder Processing of Intermetallic Compounds: *N. S. Stoloff*[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. and Eng. Dept., MRC-110, Troy, NY 12180-3590 USA

Powder techniques are widely used to consolidate intermetallic compounds and their composites, due in many cases to lack of workability and/or extreme sensitivity to compositional variations. Another factor favoring powder techniques is the exothermic reaction between aluminum or silicon with transition metals, thereby allowing use of reactive processing methods. Mechanical alloying is widely used, as are thermal spray techniques. The relative advantages and disadvantages of the various powder processes are discussed, and prospects for further development of powder techniques are described.

4:10 PM Invited
Powder Processing and Properties of Fe-40Al Sheets: *S. C. Deevi*[1]; B. Reddy[1]; S. Gedevanishvili[1]; [1]Philip Morris, Rsch. Ctr., 4201 Commerce Rd., Richmond, VA 23264 USA

Intermetallics based on iron aluminides are attractive candidates for high temperature applications due to their high strength to weight ratio, and excellent oxidation and corrosion resistances. As part of the Philip Morris program on intermetallics, we carried out alloy design of FeAl to optimize the room temperature ductility and high temperature strength of FeAl alloys. Alloys with an optimum combination of properties were powder processed to obtain thin sheets of FeAl by employing a roll compaction and annealing techniques. At present, excellent sheets of FeAl are commercially manufactured by roll compaction followed by annealing techniques using water atomized FeAl powders. In this paper, we will discuss the evolution of microstructure during the rolling process, and the importance of intermediate and final sintering/annealing steps for the recovery and recrystallization processes. In addition, we summarize the physical, electrical, and tensile properties of FeAl sheets obtained by roll compaction.

4:30 PM +
Production and Characterization of Fully Dense Hydrogenated γ-TiAl by Blended Elemental Approach: A. Genc[2]; M. L. Ovecoglu[2]; O. N. Senkov[1]; F. H. Froes[1]; *Javaid I. Qazi*[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., Mines Bldg. Rm. 321, Moscow, ID 83844-3026 USA; [2]Istanbul Technical University, Metall. Eng. Dept., Maslak, Istanbul 80626 Turkey

Fully dense nanocrystalline γ-TiAl alloys were produced by mechanical alloying (MA) of elemental titanium and aluminum powders in a high-energy ball mill for sixteen hours followed by hot isostatic pressing (HIP'ing) at 850°C or 1050°C and a 207-MPa pressure. Some amounts of titanium powder were replaced with titanium hydride to obtain the alloys with 0, 10 and 20at.% hydrogen. After HIP'ing hydrogen was removed by vacuum annealing. Scanning electron microscopy (SEM) was used to study the powder morphologies before and after MA. Phase and microstructural analyses were carried out using X-ray diffraction (XRD) and transmission electron microscopy-energy dispersive spectroscopy (TEM-EDS) techniques. Differential thermal analysis (DTA) was used in conjunction with XRD to determine phase transformations in powders and compacted materials on heating. Mechanical properties of the compacts were also determined. This far from equilibrium process allowed production of hydrogenated & nanocrystalline γ-TiAl alloy, which has very low hydrogen solubility otherwise.

4:50 PM
Fabrication, Microstructure and Creep Properties of Composites for Compositional Gradation In Turbine Blades: *Ramkumar K. Oruganti*[1]; Amit K. Ghosh[1]; [1]University of Michigan, Dept. of Matls. Sci., 2300 Hayward St., Ann Arbor, MI 48109 USA

Thermal barrier coatings for turbine blades suffer from the problem of spalling due to residual stresses developed as a consequence of mismatch in coefficients of thermal expansion between the superalloy substrate and the coating. Compositional gradation between the coating and the substrate has been proposed as a means to alleviate this problem. In this investigation powder metallurgy has been explored as a possible means to fabricate such materials. A model superalloy material selected for use in this study was Rene' 95. Composites of Rene '95 and partially stabilized zirconia, which is commonly used as a thermal barrier coating were fabricated by ball milling and subsequent hot pressing. The microstructural features of such composites and their creep strengths were studied. The creep strength of this material was evaluated in compression using decremental step strain rate tests. It is found that the composite exhibits higher strength than the unreinforced superalloy in the creep regime. A graded material was also fabricated by hot pressing slices of material of varying composition produced through the powder metallurgy route. Microstructural studies show good bonding between the layers and a theoretical calculation has been made to demonstrate the beneficial stress reductions that can be achieved in such a material over an abrupt ceramic-superalloy interface.

5:10 PM
Effects of Temperature and Shot-Peening on LCF Behaviour of a PM Ni-Base Superalloy Udimet 720: *Jiaming Luo*[1]; Paul Bowen[1]; [1]The University of Birmingham, Sch. of Metall. and Matls., Edgbaston, Birmingham, England B15 2TT UK

Fatigue S-N curve behaviour for a PM Ni-base superalloy Udimet 720 in both as extruded and shot-peened conditions was investigated at room temperature and a temperature of 600°C in air. Tests were carried out under four point bending condition at a load ratio of 0.1 and a frequency of 10 Hz. At room temperature, a bilinear S-N curve for shot-peened material was found, and at a temperature of 600°C, an offset "bilinear" S-N curve behaviour was found for both as-extruded and shot-peened materials. This offset "bilinear" S-N curve behaviour is deduced to be due to the transference of crack nucleation site location. Information obtained on crack nucleation sites, crack nucleation life, crack closure, environmental attack and cyclic softening and hardening has been used to explain the experimental results.

Testing, Characterization and Standards for Composite Materials: Characterization - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Nikhilesh Chawla, Arizona State University, Department of Chemical, Bio & Materials Engineering, Tempe, AZ 85287-6006 USA; Awadh B. Pandey, AFRL/MLLM, Universal Energy Systems, WPAFB, OH 45433 USA

Wednesday PM Room: Lewis
October 11, 2000 Location: Regal Riverfront Hotel

Session Chair: Benji Maruyama, Air Force Research Laboratory, Matls. and Manufact. Direct., WPAFB, OH 45433 USA

2:00 PM Invited
Characterization of Discontinuously Reinforced Composite Microstructures: *Benji Maruyama*[1]; Jonathan E. Spowart[2]; [1]Air Force Research Laboratory, Matls. and Manufact. Direct., AFRL/MLLM, 2230 10th St., Wright Patterson AFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The microstructure of a composite (i.e., reinforcement size and distribution, particle shape and spatial distribution or degree of clustering) can have a significant impact on its processing behavior as well as its mechanical and physical behavior. Traditional means of characterization, such as volume fraction, average particle size and aspect ratio are limited in their ability to capture important microstructural phenomena such as clustering. More advanced techniques such as point tessellation and radial distribution functions are better, but still lack meso- or multiple length scale information. This work reviews the capabilities and limitations of current microstructural characterization techniques and explores newly-proposed techniques. Also, the matrix-coated particle process for composites with improved distribution is briefly discussed.

2:45 PM
Yield Surface Estimation of Particulate-Reinforced Aluminum Composites by Microtomography and FEM Simulation: A. B. Geltmacher[1]; K. E. Simmonds[1]; *R. K. Everett*[1]; P. Matic[1]; C. T. Dyka[2]; [1]Naval Research Laboratory, Multifunct. Matls. Brnch, Code 6352, Washington, DC 20375-5343 USA; [2]GEO-Centers, Inc., Ft. Washington, MD 20749 USA

The process of meso-scale deformation localization and percolation of yield has been investigated for titanium diboride particulate reinforced 1100 aluminum composite materials. High resolution, synchrotron x-ray computed tomography (XCMT) was performed to determine the spatial arrangements of the particles. These data were used to construct a 3D

uniform-mesh finite element method (FEM) model of the actual composite material microstructure. Linear elastic and non-linear plastic FEM analyses were performed to identify regions of high stress and strain under various loading conditions. Superposition of the linear elastic cases was used to estimate the three-dimensional yield surface by tracking percolation of clusters of yielded elements throughout the model, and to understand the statistics of yield percolation in material failure. The non-linear plastic cases help to identify microstructural features which promote local microyielding.

3:15 PM
Object Oriented Finite Element Method (OOF) for Microstructure-Based Analysis of Metal Matrix Composites: *Rajarshi Saha*[1]; J. Roberts[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Matls. Sci. and Eng. Pgm., Tempe, AZ 85287-6006 USA

Conventional Finite Element Method (FEM) techniques have been used to extensively to describe and predict the behavior of metal matrix composites (MMCs). Unfortunately, these techniques are unable to take the complex nature of microstructure in these materials into the consideration. In particulate reinforced materials, for example, clustering and the morphology of the particles can significantly affect composite response to an applied stress. To obtain a true microstructure-based model, we have employed a new object oriented finite element analysis (OOF). OOF relies on objected-oriented design of the microstructure, where digitized images are assigned material properties by pixel and gray levels and Boolean operations. Numerical solutions, similar to conventional FEM technology, are then used to calculate macroscopic properties from the images of real or simulated microstructures. In this manner, complex microstructures in MMCs were analyzed. Results from the analysis, as well as future directions in object-oriented modeling will be presented.

3:45 PM Break

4:05 PM
Determination of Elastic Moduli in Composites by Resonant Ultrasound Spectroscopy and Impulse Excitation: *Charles P. Coffin*[1]; Krishan K. Chawla[1]; Mark C. Koopman[1]; Burton R. Patterson[1]; Xin Deng[1]; *Zak Fang*[2]; Greg Lockwood[2]; [1]University of Alabama at Birmingham, Dept. of Matls. and Mech. Eng., BEC 254, 1530 3rd Ave. South, Birmingham, AL 35294-4461 USA; [2]Smith International, Smith Tool, 16740 Hardy St., P.O. Box 60068, Houston, TX 77205-0068 USA

Resonant ultrasound spectroscopy (RUS) and impulse excitation are non-destructive techniques that can be used to determine the elastic moduli of a solid. In addition, the RUS technique has the capability of determining a complete set of elastic constants for a given specimen. Quantification of microstructural damage is also possible with these methods. Descriptions of these techniques will be given as well as examples of modulus determination of some WC/Co composites (cemented carbides) by these techniques will be provided. In the cemented carbides examined, the volume fraction and morphology of the particulate phase were varied. Comparison between experimental and theoretical values of elastic moduli showed them to be in very good agreement. Project funded by NSF, DMR-9904352, Dr. Bruce MacDonald, Program Director.

4:35 PM
Correlating Hardness and Tensile Behavior of Reinforced Metal Matrix Composites: *Yu-Lin Shen*[1]; Nikhilesh Chawla[2]; [1]The University of New Mexico, Dept. of Mech. Eng., Albuquerque, NM 87131 USA; [2]Arizona State University, Depts. of Chem. Bio. and Matls. Eng., Tempe, AZ 85287 USA

Hardness tests are routinely employed to estimate the mechanical strength of metallic materials because of their ease of sample preparation and non-destructive nature. For a variety of engineering metals and alloys, the correlation between various hardness scales and tensile strength has been documented. As metal matrix composites are generating increased interest for applications where quality control may be important, it is essential to understand if such a correlation is existent in MMCs. In this work we have performed tensile and hardness tests on discontinuously reinforced aluminum composites, namely 2080/SiC/10p and 2080/SiC/20p, as well as the monolithic 2080 alloy. No unique correlation between hardness and tensile strength was found. The reinforcement fraction and matrix strength appear to play an important role in affecting the hardness-strength relationship. The different loading modes of the tensile test compared to the hardness test, along with the local increase in particle concentration directly underneath the indenter during indentation, result in a significant overestimation of the tensile strength by the hardness test, especially when the matrix strength is relatively low. Micromechanical modeling using the finite element method was also undertaken to rationalize the experimental findings. Implications to the establishment of a standard procedure for characterizing the hardness-strength relation in particle reinforced metal matrix composites will be discussed.

NOTES

Thursday, October 12, 2000

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
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Fatigue and Fracture Behavior of High Temperature Materials - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Mechanical Behavior of Materials
Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, TN 37996-2200 USA; Miinshiou Huang, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA

Thursday AM Room: Mississippi
October 12, 2000 Location: Regal Riverfront Hotel

Session Chairs: R. O. Ritchie, University of California, Berkeley, CA 94720-1760 USA; W. O. Soboyejo, Princeton University, Princeton, NJ USA

8:30 AM
An Investigation of the Effects of Temperature on Fatigue Crack Growth in Cast Lamellar Gamma Titanium Aluminides: *J. Lou*[1]; C. Mercer[1]; W. O. Soboyejo[1]; [1]Princeton University, The Princeton Matls. Instit. and the Dept. of Mech. and Aero. Eng., Olden St., Princeton, NJ 08544 USA

This paper examines the effects of temperature on fatigue crack growth on cast lamellar gamma-based titanium aluminides. The paper attempts to provide mechanistic explanation of the so-called crack growth rate anomalies that have been reported previously in the intermediate-temperature regime between 450 and 550°C. Faster (compared to those at 25 and 750°C fatigue crack growth rates are attributed to the high incidence of irreversible deformation-induced twinning phenomena. However, slower (compared to those at 450°C) fatigue crack growth rates at 750°C are explained by oxide-induced closure phenomena. The shielding contributions from oxide-induced crack closure are also quantified using a modified Dugdale-Barrenblatt model. The effects of temperature on fatigue crack growth are then rationalized by considering the combined effects of oxide-induced closure and crack-tip deformation on fatigue crack growth.

8:50 AM
Fatigue Crack Initiation and Short Crack Growth in Cast Lamellar Gamma Titanium Aluminides: *C. Mercer*[1]; D. Mumm[1]; V. Sinha[2]; J. Lou[1]; W. O. Soboyejo[1]; [1]Princeton University, Dept. of Mech. and Aero. Eng., Olden St., The Princeton Matls. Instit., Princeton, NJ 08544 USA; [2]The Ohio State University, Dept. of Matls. and Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

This paper presents the results of an experimental study of fatigue crack nucleation and growth mechanisms in cast gamma-based titanium aluminides with lamellar microstructures. Natural fatigue crack nucleation from smooth surfaces is shown to occur from slip offsets associated with twin boundaries across lamellar colonies. The crystallographic orientations of the deformed colonies, and the misorientations across colony boundaries are elucidated via orientation imaging microscopy. The micromechanisms of fatigue crack nucleation and short crack growth are then described for near-commercial alloys at room temperature. The implications of the results are then discussed for the prediction of fatigue life.

9:10 AM +
Damage Assessment of Ceramic Matrix Composites by Nondestructive Evaluation Techniques: *Jeongguk Kim*[1]; Peter K. Liaw[1]; Hsin Wang[2]; You-Tae Lee[3]; [1]The University of Tennessee, Depts. of Matls. Sci. and Eng., 434 Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Mets. and Cers. Div., Oak Ridge, TN 37831 USA; [3]Kyongdo College, Auto. Dept., Kyongpook 757-800 South Korea

Nondestructive Evaluation (NDE) techniques, such as acoustic emission (AE), infrared (IR) thermography and ultrasonic testing (UT), were used to characterize the mechanical behavior of ceramic matrix composites (CMCs). Before, during and after the tensile tests, NDE techniques were performed on CMCs to characterize the damage evolution. Prior to mechanical testing, UT and thermography were used to evaluate initial defect distributions of samples. UT provided 2-dimensional C-scans, and IR thermography was used for generating thermal diffusivity maps. NDE results showed defect distributions that may affect the damage evolution process. Both AE sensors and IR camera were used to detect the amount of damage and for "in-situ" monitoring of the damage during tensile tests. After the tensile testing, ultrasonic C-scans and IR thermography were performed on fractured CMC samples for quantitative analyses of the progressive damage. The NDE results have been used to understand the failure mechanisms of CMCs.

9:30 AM
NDE Methods to Detect the Degradation of Fracture Toughness Properties of Nickel-Base Alloys: *R. R. Seeley*[1]; D. E. McCabe[2]; W. A. Simpson[2]; R. B. Dinwiddle[2]; F. M. Haggag[3]; [1]Haynes International, Inc., 1020 W. Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA; [2]Oak Ridge National Laboratory, 1 Bethel Rd., Oak Ridge, TN 37831 USA; [3]Advanced Technology Corporation, 661 Emory Valley Rd., Ste. A, Oak Ridge, TN 37830 USA

Fracture toughness is an important material property for fracture-critical and structural components. Fracture toughness and fracture mechanics analysis can support material selection, component design and nondestructive examination for critical components. The fracture toughness and ductility properties of nickel-base alloys and other structural materials can be degraded during operation at moderately elevated temperatures. This degradation may lead to failure of the component. Work is in progress to determine the feasibility of certain NDE techniques to detect the degradation of fracture toughness properties of HASTELLOY® X and INCOLOY® 800H. The room temperature fracture toughness properties of these materials in thermally degraded conditions have been measured. These materials were then examined by several NDE methods to determine if there is a correlation of NDE signals and fracture toughness. The NDE methods include nonlinear acoustic properties and thermographic properties and the automated ball indentation test. The results of the NDE examinations and possible correlations with fracture toughness will be discussed. HASTELLOY is a registered trademark of Haynes International, Inc. INCOLOY is a registered trademark of Special Metals formerly INCO Alloys.

9:50 AM Break

10:10 AM
High-Temperature Cyclic Fatigue-Crack Growth and Fracture Properties in Ti3SiC2 Ceramics: Kiroshi Shirato[1]; *Da Chen*[1]; Robert O. Ritchie[1]; Michel W. Barsoum[2]; T. El-Raghy[2]; [1]University of California at Berkeley, Matls. Sci. and Eng. Depts., 1 Cyclotron Rd., LBNL MS 62-203, Berkeley, CA 94720 USA; [2]Drexel University, Dept. of Matls. Eng., 32nd and Chestnut St., Philadelphia, PA 19104 USA

Monolithic Ti_3SiC_2 ceramics, processed with reactive hot-pressing techniques, are found to exhibit remarkably high resistance-curve (R-curve) fracture toughness values, K_c ~ 9.5-16 MPa√m, and fatigue-crack growth thresholds, ΔK_{th} ~6.5-9 MPa√m, at ambient temperatures. In this presentation, the corresponding cyclic fatigue and fracture toughness behavior at temperatures up to 1200°C are examined with the objective of defining the high-temperature mechanisms controlling crack growth. Comparisons are made of two monolithic Ti_3SiC_2 ceramics with fine- (~ 3-10 μm) and coarse-grained (~ 50-200 μm) microstructures. Results indicate that although the toughness and ΔK_{th} thresholds are not substantially changed at 1100°C, there is a sharp decrease in ΔK_{th} at 1200°C, above the ductile/brittle transition temperature, where significant creep deformation is first apparent. Of the two microstructures, the coarse-grained Ti_3SiC_2 exhibited substantially higher toughness and cyclic-crack growth resistance at both ambient and elevated temperatures. This resulted from an enhanced effect of grain bridging in the crack wake and a correspondingly more tortuous crack path in the coarser grained microstructure. These effect are discussed in terms of the balance between intrinsic damage mechanisms ahead the crack tip and extrinsic crack-tip shielding processes, which are active behind the tip.

10:30 AM
Low-Cycle Fatigue of ULTIMET Alloy: *Liang Jiang*[1]; Peter K. Liaw[1]; Charlie R. Brooks[1]; Jeremy Harper Strader[1]; D. L. Klarstrom[1]; [1]University of Tennessee, Matls. Sci. and Eng. Dept., 434 Dougherty Eng. Bldg., Knoxville, TN 37996 USA

The cyclic deformation behavior of a wrought cobalt-based alloy, ULTIMET alloy, has been investigated 25, 600, and 900°C. A constant mechanical strain rate of 3.0¥10-3s-1 were used with fully reversed strain ranges between 0.4 to 2.5%. The crack initiation and propagation modes, deformation substructure, and precipitation were characterized through scanning electron microscopy and transmission electron microscopy. Dynamic strain aging (DSA) was observed at 600 and 900°C. The DSA phenomenon made the alloy exhibit a marked cyclic hardening behavior and inverse temperature dependence of the maximum cyclic stress developed at the half fatigue life, accompanied by the formation of high-density stacking faults and dislocation pinning by precipitates. The correlation between the marcroscopic cyclic deformation and fatigue life with the various microstructural phenomena is illustrated.

10:50 AM
Influence of Hold Time on Low-Cycle Fatigue Behavior of Cobalt-Base Superalloy Haynes 188: *Lijia Chen*[1]; Peter K. Liaw[1]; Yuehui He[1]; Michael L. Benson[1]; James W. Blust[2]; Paul F. Browning[2]; Rodger R. Seeley[3]; Dwaine L. Klarstrom[3]; [1]The University of Tennessee at Knoxville, Dept. of Matls. Sci. and Eng., 434 Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy. MZ R-1, P.O. Box 85376, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

The fully-reversed total strain-controlled low-cycle fatigue tests with hold times ranging from 2 minutes to 1 hour at the maximum tensile strain per cycle, as well as stress relaxation tests were conducted at 816 and 927°C in laboratory air on a cobalt-base superalloy, Haynes 188. The chosen total strain range was 1.0%. The influence of hold times on low-cycle fatigue behavior of the alloy was investigated. It was noted that the alloy exhibited the initial cyclic hardening followed by the cyclic softening at 816°C. However at 927°C, the alloy showed either cyclic stability or cyclic softening, which is closely related to the duration of the hold time. It was also observed that the low-cycle fatigue life of the alloy considerably decreased due to the introduction of strain hold times. Generally, a longer hold time would result in a greater reduction in the fatigue life. In addition, the fracture surfaces of the fatigued specimens were observed using scanning electron microscopy to determine the crack initiation and propagation modes. Acknowledgments: This work is supported by the Solar Turbines Inc., Haynes International, Inc., the University of Tennessee, the U. S. Department of Energy's Advanced Turbine Systems Program, the National Science Foundation, under Grant No. DMI-9724476, and the Combined Research-Curriculum Development Program, under EEC-9527527, with Dr. D. Durham and Ms. M. Poats as contract monitors, respectively.

11:10 AM
Simulation of Grain Growth in a Near-Eutectic Solder Alloy: *Michael William Woodmansee*[1]; [1]Georgia Tech, Mech. Eng. Dept., 801 Ferst Dr., MRDC 3338 P.O. Box 1130, Atlanta, GA 30332-0405 USA

Microstructural evolution due to aging of solder alloys determines their long-term reliability as electrical, mechanical and thermal interconnects in electronics packages. The ability to accurately determine the reliability of existing electronic components as well as to predict the performance of proposed designs depends upon the development of reliable material models. A kinetic Monte Carlo simulation was used to simulate microstructural evolution in solder-class materials. The grain growth model simulated many of the microstructural features observed experimentally in 63Sn-37Pb, a popular near-eutectic solder alloy. The model was validated by comparing simulation results to new experimental data on coarsening of Sn-Pb solder. The subgrain growth exponent of two-phase solder was found to be much lower than that of normal grain growth in the experimental system and in the simulations. It was found that the phase composition of solder is important in determining grain growth behavior.

Interfacial Dislocations: Symposium in Honor of J.H. Van der Merwe on the 50TH Anniversary of His Discovery - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structures
Program Organizers: Gary J. Shiflet, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA; William A. Jesser, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA

Thursday AM Room: Missouri
October 12, 2000 Location: Regal Riverfront Hotel

Session Chair: Doris Kuhlmann-Wilsdorf, University of Virginia, Dept. of Matls. Sci. and Eng., Charlottesville, VA 22903-2442 USA

8:30 AM
Misfit Dislocations in Epitaxy: *J. H. van der Merwe*[1]; [1]University of South Africa, Dept. of Phys., Pretoria, South Africa

The paper highlights (a) appropriately the Frank-van der Merwe theory of monolayer epitaxy as based on the Frenkel-Kontorowa model, which applies a truncated Fourier series to represent the periodic interfacial interaction, and how it predicts the properties of epilayers, (i) e.g. misfit strain relief by misfit dislocations (MDs) at a critical misfit, (ii) the extension of the theory to epitaxial multilayers, e.g. the concept of critical thickness, pseudostability, etc. that impact on the fabrication of device components, (iii) the role of optimum Fourier coefficients in attaining and stabilizing epitaxial configurations. (b) It is also shown how optimum Fourier coefficients can be calculated using embedded atom method potentials, how MDs contribute to the interfacial energy, a quantity which plays an important role in growing epilayers of uniform thickness and how the Volterra approach to dislocations lends itself to the description of complicated MD configurations.

9:05 AM
Reciprocal Space Formulation and Prediction of Misfit Accommodation in Rigid and Strained Epitaxial Systems: *Max W.H. Braun*[1]; Jan H. van der Merwe[2]; [1]University of Pretoria,Centre for Sci. Educ., Pretoria 0002 South Africa; [2]University of South Africa, Phys. Dept., P.O. Box 392, Pretoria 0001 South Africa

Geometical properties of heteropepitaxial systems in which orientation, symmetry and lattice parameters differ. An energetically founded epitaxial criterion for matching at a planar interface is derived from a generalization of the Frank-van der Merwe theory and the rigid models introduced by Rice and van der Merwe. Formulated in reciprocal space, the criterion is visualized with a construction that allows for structure factors introduced to account for non-primitive natures of substrate and over-growth surface unit cells and is analogous to the Ewald construction of crystallography. Initial and residual misfits and energy minimizing misfit strains and rotations can be predicted directly from the matching criterion. The spacing, Burgers vectors, orientation and nature (edge, screw or mixed) of misfit dislocations or vernier arrays that can accommodate the misfit are available in forms that conform to crystallographic conventions. The description is general and is applicable to any combination of crystal symmetries or mismatch.

9:40 AM
Misfit Accommodation by Compliant Substrates: *W. A. Jesser*[1]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., Charlottesville, VA 22903-2442 USA

Misfit accommodation by compliant substrates has been investigated for several years, but the mechanism by which it operates has not yet been fully explained. The early model of van der Merwe has been used to design compliant substrates based on elastic accommodation of misfit. In this presentation the model is extended to include misfit accommodation by the generation of misfit dislocations. The implications of heteroepitaxial growth on compliant substrates for the case of large bulk substrates introduces the special problem of how the edges of the layers behave during the misfit accommodation process. A quantitative treatment of the twist-misfit interface using the van der Merwe model will be used to address the accommodation of interfacial misfit by conservative and by non-conservative generation of misfit dislocations. The consequences of this model will be discussed in terms of of the operation of compliant substrates.

10:15 AM Break

10:30 AM
Dislocation Dynamics in Strained Epitaxial Films: *Robert Hull*[1]; John C. Bean[2]; Eric A. Stach[3]; [1]University of Virginia, Dept. of Matls. Sci. and Eng., Thornton Hall, Charlottesville, VA 22903 USA; [2]University of Virginia, Dept. of Elect. Eng., Thornton Hall, Charlottesville, VA 22903 USA; [3]Berkeley Laboratories, Nat. Ctr. for Elect. Micro., MS 72-150, Berkeley, CA 94720 USA

The relaxation of stress in lattice-mismatched epitaxial thin films by misfit dislocations has been studied for several decades, following the pioneering work of van der Merwe and co-workers. In the past decade, in-situ annealing of metastably strained films in the transmission electron microscope has provided broad insight into the kinetics of stress relaxation by misfit dislocations. We show how such measurements may be used to provide the fundamental parameters necessary for predictive simulations of plastic relaxation in these structures. Further, the regimes of stress and length scale inherent to these thin film geometries enables measurement of dislocation propagation in new regimes, providing new insight into the fundamental mechanisms of dislocation motion.

Microcharacterization and Microtexture - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee
Program Organizers: Dierk Raabe, Max Planck Institut fuer Eisenforschung, Department for Microstructure & Metal Forming, Duesseldorf 40237 Germany; Wei Tong, Yale University, Department of Mechanical Engineering, New Haven, CT 06520-8264 USA; Hasso Weiland, Alcoa Technical Center, Alcoa Center, PA 15069 USA

Thursday AM Room: Meramac
October 12, 2000 Location: Regal Riverfront Hotel

Session Chair: Hasso Weiland, Alcoa Technical Center, Alcoa Center, PA 15069 USA

8:30 AM
The Precision of Disorientations Measured from Automatically Analysed of Kikuchi Patterns: *Niels C. Krieger-Lassen*[1]; Darcy A. Hughes[2]; [1]Risoe National Laboratory, Matls. Rsch. Dept., P.O. Box 49, Roskilde DK-4000 Denmark; [2]Sandia National Laboratories, 2 Ctr. for Matls. and Eng. Sci., P.O. Box 969, MS 9405, Livermore, CA 94551-0969 USA

Crystal orientations measured from Kikuchi patterns in the TEM are frequently used for determining disorientations (also referred to as misorientations) across grain or subgrain boundaries, dislocation walls, or other types of lattice defects. In many studies, a high precision of the disorientation measurements is required, both with regards to the rotation angle and axis. This is particularly so when small disorientations are to be measured, especially for the determination of the rotation axis. This paper presents an experimental investigation of the uncertainty of disorientation measurements made on the basis of automatically analysed Kikuchi patterns. The uncertainty is determined by repeating measurements across the same boundary at a number of different sample position. We describe a procedure for determining the mean and the dispersion of a sample of disorientations and show that the uncertainty of disorientations is actually dependent on the rotation angle. We also show, both from experimental data and from simulations, how the rotation angle and the precision of the measured single orientations determine the uncertainty of the rotation axis.

8:50 AM
TEM-Investigation on Interface Structure and Chemical Stability in Composite of NiAl/Al2O3-Fibre Coated with BN: *W. Hu*[1]; K. Wen[1]; G. Gottstein[1]; [1]Institut fur Metallkunde und Metallphysik, RWTH Aachen, Kopernikusstrasse 14, Aachen 52056 Germany

An investigation was carried out on the composite with NiAl matrix reinforced by Al2O3 fibres. The samples were prepared by diffusion bonding. In order to improve the bond between the components the surface of the fibres was coated with BN. The interface structure and chemical stability between the matrix, fibre and BN coating were studied by means of routine TEM, HRTEM and microanalyses such as XEDS, PEELS and electron diffraction (SAD and CBED). It was found that during the diffusion bonding (1400°C/10MPa/2h) a good chemical stability was displayed between NiAl/Al2O3 and Al2O3/BN. But a diffusion controlled chemical reaction has occurred between the couple of NiAl/BN. A thin layer of reaction product with a thickness of ca. 100nm was formed in the NiAl/BN interface. It was identified as AlB12 by microanalyses. The influence of these BN coating and AlB12 thin reaction layer on the mechanical property of the interface and consequently on the property of the composite will be researched in our further investigating work.

9:10 AM
Grain Boundary Character Dependence of the Sensitization of 304 Stainless Steel: *Alisa J. Henrie*[1]; Ryan J. Larsen[1]; Brent L. Adams[1]; [1]Brigham Young University, 435 CTB, Provo, UT 84602 USA

By knowing the inclination of the boundary plane between two crystal grains the definition of a special boundary can be narrowed; a special boundary being one that has superior material properties. Two approaches are being taken to recover the inclination of the boundary. The first involves measuring the in-plane angle consistent with the coherent plane. The second approach is to take serial sections of about 5-10 µm each and reconstruct a three-dimensional model of the grains and grain boundaries. This will allow the full (two parameter) inclination of the boundary to be determined with some degree of precision. In both approaches the samples evaluated are sensitized 304 stainless steel, etched so that the sensitized grain boundaries can be identified visually. The influence of the boundary plane is then correlated with the sensitization of the grain boundaries.

9:30 AM
Orientation Imaging Microscopy Investigation of Bainite-Austenite Structures using a Field Emission Gun Microscope: *Bert Verlinden*[1]; Bocher Philippe[1]; Girault Etienne[1]; Aernoudt Etienne[1]; [1]Katholieke Universiteit Leuven, Dept. of Metall. and Matls. Eng., De Croylaan Nr. 2, Heverlee B-3001 Belgium

Orientation imaging microscopy (OIM) experiments were performed on a bainitic medium carbon steel with large amounts of retained austenite. The main purpose of the present work is to measure–without recalculation–the texture prior to the transformation and to investigate the variant selection during phase transformation. Such results can be obtained only with large amount of retained austenite and using a high-resolution electron backscatter diffraction technique. Consequently, a medium carbon steel was selected and rolling in the austenitic region was performed prior to a bainitic holding treatment in order to stabilize the austenitic phase. With such an experimental procedure, specimens with large amount of retained austenite (up to 25%) and with detectable austenite grain size (a few µm) were obtained. Moreover, field emission gun microscopy (FEG) was used in order to detect the deformed and untransformed austenite phase. The orientation of the bainite variants and the orientation of the former austenite grain could be compared without any assumption on the type of relationships between austenite and ferrite phases. Moreover, the variants can be displayed directly in the reference frame of that former austenite grain. It was found that the texture of the austenite (at high temperature) becomes sharper with increasing hot rolling reduction. After transformation to bainite both a sharper morphological and crystallographic bainite texture was observed with increased pre-deformation during rolling. The bainite was found to obey the Wassermann relationship rather than the Bain or the Kurjumov-Sachs relationship.

9:50 AM Break

10:10 AM
Orientation Imaging Microscopy in Research on Texture in High Temperature Oxidation: *Bae-Kyun Kim*[1]; J. A. Szpunar[1]; [1]McGill University, Dept. of Metall. Eng., Montreal H3A 2B2 Canada

It is well demonstrated that electron backscattered diffraction (EBSD) is a highly promising tool for studying local textures and microstructure. In the present work, the EBSD is applied to examine texture transformation during high temperature oxidation and the process of oxidation is investigated by using this technique. High temperature oxidation tests were performed in the tube furnace up to 950°C with continuous heating under atmospheric pressure. The specimens used for this study were commercial low carbon steel. As a result, the microstructure of iron oxide has been obtained using an orientation imaging microscopic (OIM) mapping of the cross-sectional area of oxidized steel. The process of oxidation was explained by using data obtained from OIM maps. In the study, field emission SEM (FE-SEM) and orientation imaging microscopy (OIM) were used. The relationship between the oxide and steel texture can be analyzed and the results obtained from different phases of the iron oxide texture. Kikuchi patterns of each phase were identified.

10:30 AM
Texture Analysis of Fine Wire with EBSD: *Jae-hyung Cho*[1]; J. T. Moon[2]; Jin Lee[2]; K. H. Oh[1]; [1]Seoul National University, Sch. of Matls. Sci. and Eng., College of Eng., Seoul 151-742 ROK; [2]MK Electron Co., LTD., Research and development, 316-2, Kumeu-Ri, Pogok-Myun, Yongin-Si, Kyunggi-Do, 449-810 ROK

The cold drawing process using dies leads to fiber texture. In fcc metal {111} major texture component and {100} minor components are developed generally. After drawing process, the fine wire with 30µm diameter was annealed fully. The annealed wire has the increased elongation and the decreased strength and hardness. The fine wire is too small to measure the texture with X-ray diffraction and the measurement of recrystallization texture was carried out with EBSD, which can be used for measuring of local microtexture. The recrystallization texture of fine wire consisted of three layers of fiber texture across the diameter. The surface layer has the {100} minor fiber component. The intermediate layer has the {111} major component and the core part has the {100} minor component. Large die angle can develop the shear stress and inhomogeneous texture across wire diameter. The inhomogeneous deformation results in the formation of triple-layered recrystallization texture during annealing. After the fine wire was annealed additionally above recrystallization temperature, the grain size increased and the texture distribution was not changed.

10:50 AM
Single Reflection Method and its Use in Study of Misoriented Microstructures in Highly Deformed Crystals: *V. Rybin*[1]; [1]Central

Research Institute of Structural Materials, Prometey 49 Shpalernaja, St. Petersburg 193015 Russia

The single reflection method (SRM) has been developed in TEM as an accurate technique for local crystallographic analysis, remaining efficient under such conditions as: i) heterogeneous or misoriented (fragmented) structure of micron and sub-micron scales, ii) heavily distorted crystal lattice, iii) high densities of planar, linear and point defects, including fine precipitates. Under similar conditions we have widely used SRM to identify the crystal lattice type and its parameters; to determine crystallographic indexes of linear and planar defects; to determine inter-phase orientation relationships, as well as local orientations and misorientations on the sub-micron scale. The SRM features (procedure, resolution, accuracy) are overviewed and some results are demonstrated. Namely: distribution of misorientation angles in the Mo monocrystal subjected to hydroextrusion; misoriented mesostructure of the Ti-Ti weld connected zone; irradiation-induced fragmented structure in a dispersion-strengthened Cu alloy; misoriented mesostructure in high purity Cu after ECA-pressing process; and misoriented mesostructure in drawn pearlitic steel.

Powder Metallurgy Alloys and Particulate Materials for Industrial Applications: P/M Processing - IV

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: David E. Alman, US Department of Energy, Albany, OR 97321 USA; Joseph W. Newkirk, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-0340 USA

Thursday AM Room: Clark
October 12, 2000 Location: Regal Riverfront Hotel

Session Chairs: D. J. Branagan, Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID 83415-2218 USA; Richard Castro, Los Alamos National Laboratory, Los Alamos, NM USA

8:30 AM
TiC-Reinforced 304L-Stainless Steel-Matrix Composites via Reactive Sintering: *David E. Alman*[1]; Omer N. Dogan[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321-2198 USA

Composites comprised of a 304L-stainless steel matrix, reinforced with upwards of 60 volume percent (vol%) TiC particles were produced by sintering mixtures of 304L-stainless steel powders, titanium and graphite powders. Cold pressed powder compacts reinforced with 0, 20, 30, 40, 60 or 100 vol% TiC particles were vacuum sintered at 1200°C for 15 minutes. The influence of heating rate (2 or 15°C/min on the composite microstructure was examined. In general, the reactive sintered densities of the composites were between those of the relatively dense monolithic 304L-SS and porous monolithic TiC. Composite sintered at the lower heating rate were more dense than corresponding composites sintered at faster rates. This was attributed to the volatilization of gaseous species in the powder compacts during reactive sintering.

8:50 AM
Double Cemented Carbide Composite: *Xin Deng*[1]; B. R. Patterson[1]; M. C. Koopman[1]; K. K. Chawla[1]; C. Coffin[1]; Z. Fang[2]; G. Lockwood[2]; A. Griffo[2]; [1]University of Alabama at Birmingham, Matls. and Mech. Eng. Depts., Rm. 254 BEC, Birmingham, AL 35294-4461 USA; [2]Smith International, Inc., 16740 Hardy St., Houston, TX 77205-0068 USA

Double Cemented Carbide (DCC) is a dual composite composed of spherical reinforcement granules of conventional cemented carbide (WC+Co) in a metal matrix. DCC provides a flexible means for obtaining unique combinations of properties needed for wear resistant materials that must also have good fracture toughness. Compared with conventional cemented carbide, DCC has higher toughness for similar wear resistance and vice versa. In this study DCC composites were produced with independent variations in granule and matrix properties. Granule properties were varied via Co content in the cemented carbide granules. Metal matrix properties were varied by choice of matrix metal, Co or steel, and through heat treatment of the steel matrix. After hot pressing and heat treatment the combined effects of granule and metal matrix properties on the mechanical properties of DCC were investigated. The effects of microstructural parameters on mechanical properties were also determined. Supported by NSF, DMR-9904352.

9:10 AM
Synthesis of Nanostructured WC-Co Powders through an Integrated Mechanical and Thermal Activation Process: *R. M. Ren*[1]; Z. G. Yang[1]; Leon L. Shaw[1]; Z. G. Ban[1]; [1]University of Connecticut, Dept. of Metall. and Matls. Sci., 97 N. Eagleville Rd., Storrs, CT 06269 USA

In this study we report a new process that allows for large-scale production of nanostructured metal-carbide composites such as WC-Co cermets. The process is termed as the integrated mechanical and thermal activation (IMTA) process because of the integration of mechanical activation and thermal activation in the process. Using tungsten oxide (WO3), cobalt oxide (CoO) and graphite as the starting material, we have demonstrated that the mechanical activation before carbothermic reduction and carburization reactions greatly enhances the formation of WC. Furthermore, the product from this new process is nanostructured powder rather than coarse-grained counterparts. Comparisons between the IMTA process and the current industrial practice in making WC-Co cermets are conducted. The fundamentals associated with the IMTA process are also discussed.

9:30 AM
Micro- and Nano-Particle Reinforced Composites: *Shankar M. Sastry*[1]; [1]Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA

Composites with micro- and nano-grained matrix and micro- and nano-sized reinforcement were produced by particulate processing and severe plastic deformation (SPD) processing. Rapid solidification and spray deposition processing, Solution phase synethesis and gas phase combustion synthesis routes were used for the synthesis of particulate composites. The particulates/preforms were consolidated by two routes: (1) cold pressing to 70% density followed by vacuum hot pressing to near theoretical density and (ii) direct vacuum hot pressing to near theoretical density. Severe plastic deformation (SPD) by equal channel angular extrusion (ECAE) was used With the objective of improving the inter particle and inter agglomerate bonding. Strength and ductility values, anisotropy of fracture toughness and fatigue crack growth rate, and high temperature creep properties of particulate composites processed by different routes will be discussed.

9:50 AM Break

10:00 AM Invited
Materials Science Issues in Powder-Based Layered Manufacturing: *Kershed P. Cooper*[1]; [1]Naval Research Laboratory, Code 6324, 4555 Overlook Ave. SW, Washington, DC 29375-5343 USA

Layered Manufacturing (LM) refers to processes in which models, prototypes or parts are made layer-by-layer in a relatively short period of time from a three-dimensional CAD/CAM rendering of the object or part. LM techniques now allow for the capability to produce polymeric, metallic, ceramic, biological, and multi-material components. These components could be designed for structural, mechanical, or electronic function or a combination of these functions. Almost all LM methods involve particulate material. The particulate could be laid down as a powder bed with the outline of the object defined by a laser beam or a binder. Examples of processes in this category are 3DP, SLA and SLS. The particulate could be incorporated within filaments that are made to trace a path that would define the object or within laminates that would be carved and stacked to make the object. Examples of processes in this category are FDM and LOMS. The particulate could be fed into a melt pool usually formed by a laser beam that follows a predetermined path. Examples of processes in this category are LENS, DLF and LasForming. For the successful fabrication of the object or part, several materials science issues need to be considered. Some of these, such as powder characteristics, sintering, densification, etc. involve basic P/M concepts. In this paper, materials research opportunities in layered manufacturing will be identified and approaches taken by researchers to investigate these opportunities will be briefly described.

10:25 AM
Forming Composite Solder Joints through Sintering: Robert Sebra[1]; Thaer Almasri[1]; *Mark A. Palmer*[1]; [1]Virginia Commonwealth University, Mech. Eng. Dept., P.O. Box 843015, 601 W. Main St., Richmond, VA 23284-3015 USA

The use of metal particles to form a composite solder joint has been advocated as a means to strengthen the joint without sacrificing electrical continuity. In conventional processing this has proved difficult as the molten solder alloy may separate from the reinforcing particles. Composite solder joints have been formed by sintering solder paste, which alleviates this problem. The mechanical properties of solder joints formed by liquid phase sintering of copper reinforced eutectic Sn-Ag-Cu and Sn-Ag-Bi solder paste will be discussed.

10:45 AM
Solder Joints from Low Flux Solder Paste: Jamie Schmidt[1]; Nicole Erdman[1]; *Mark A. Palmer*[1]; [1]Virginia Commonwealth University, Mech. Eng. Dept., 601 W. Main St., P.O. Box 843015, Richmond, VA 23284-3015 USA

Many of the solder alloys considered as alternatives to eutectic lead-tin melt at significantly higher temperatures. Liquid phase enhanced sintering has been shown to be a possible option for forming solder joints from these materials without raising the processing temperature. An added advantage of this is that as the amount of liquid metal is reduced, the required amount of flux can be reduced. The properties of solder joints prepared by liquid phase sintering of solder paste with reduced amounts of flux will be presented and reviewed.

11:05 AM
Computer Modeling of Temperature Prediction for Electroconsolidation®: *F. C. Chang*[1]; R. R. Fessler[2]; B. D. Merkle[3]; J. M. Borton[3]; W. M. Goldberger[3]; [1]Argonne National Laboratory, Energy Tech. Div., ET 308, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Biztek Consulting, Inc., 820 Roslyn Place, Evanston, IL 60201-1724 USA; [3]Superior Graphite Company, 1620 E. Broad St., Apt. 703, Columbus, OH 43203-2072 USA

Electroconsolidation® is a new process for rapid, pressure-assisted densification of powder preforms. Parts to be consolidated are placed within a bed of free-flowing powder and pressure is applied by rams acting on the powder media. The heat for densification is generated internally by electrical resistive heating of the media while it is in direct contact with the preform(s). A model has been developed to predict temperature profiles within the die during operation. Numerical simulation was preformed by coupling a 3-D finite element elctromagnetic fields program (ELEKTRA) and a 3-D finite difference materials process simulator (MaPS) to incorporate the effect of pressure and electrical fields on heat transfer and temperature variation within the die during heating. The methods used in developing the model are presented and temperatures predicated at different positions within the die are compared with actual measurements made during operation. Development of the model minimizes the experiments needed to design and evaluate commercial applications of the process.

11:25 AM
Production of Rapidly Quenched Powders for Hydrogen Absorbing: *Evgeniy A. Shadchin*[1]; Yuri M. Lytvynenko[2]; [1]Institute of Physics, 46 Nauki Ave., Kyiv 03028 Ukraine; [2]Institute for Problems of Materials Science, 3 Krzhizhanovsky St., Kyiv 03142 Ukraine

The compositions of hydroben absorbing alloys are selected so that an optimum of hydrogen dissociation pressure is received. The chemical compositions of such alloys are close to amorphous ones and usually consist of metals which are easily forming hydrides (Ti, Zr etc.) and metals which are not forming the hydrides (Ni, Fe, Co). The impact-centrifugal spraying is one of the perspective methods for production of rapidly quenched powders of a wide range of hydrogen absorbing alloys. The original method dispersing of a melt jet which based on series of successive binary contacting of a dispersed fluid alloy with heat conducting surfaces allows to receive a dispersed amorphous powder with a branched surface at cooling rate up to 10 to the 6th K/s. By such method it is possible to receive the rapidly quenched powders of Ni-Cu, Nd-Fe-B, Nd-Ni, Zr-Co, Zr-Ni systems for different applications.

11:45 AM +
Processing, Microstructure and Properties of a Copper Alloy Coating Fabricated Via Laser Cladding Processing: Dong Shi-Yun[1]; Han Jie-Cai[1]; *Du Shan-Yi*[1]; [1]Harbin Institute of Technology, Ctr. for Comp. Matls., 92 W. St., Nan Gang District, Harbin, Heilongjiang Province 150001 China

Abstract: A copper base powder system, consisting of Cu, Ni, Mo, Co, Fe, Cr and Si, was designed according to metallurgical characterization of binary Cu alloys, such as Cu-Ni, Cu-Mo, Cu-Co and Cu-Fe to get high strength Cu alloy. A 5kW CW CO2 laser was employed in laser cladding the powder into the Cu alloy coating on Al alloy ZL104. The laser cladding processing parameters were studied to get good coating, and the operating diagram of the processing parameter was gained. Microstructure investigations showed that the melt of the Cu alloy separated into two liquids, L1 and L2. And L1 liquid solidified into spherical particles of silicides which had hardness Hv of 800~1000. L2 liquid solidified into supersaturated solid solution (Cu, Ni) as base phase of the coating. The base phase had fine dendritic microstructure. The coating had good wear resistance at both room temperature and elevated temperature.

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