	Monday		Tuesday		Wednesday		Thursday
	AM	PM	AM	PM	AM	PM	AM
Caprice 1 & 4	Refractory Metals & Alloys; A Symposium on Research, Development & Applications 1	Refractory Metals & Alloys; A Symposium on Research, Development & Applications II	Refractory Metals & Alloys; A Symposium on Research, Development & Applications III	Refractory Metals & Alloys ; A Symposium on Research, Development & Applications IV	Computational Materials Science at the Microstructural Scale 1	Computational Materials Science at the Microstructural Scale II	Computational Materials Science at the Microstructural Scale III
Caprice 2 & 3		GA 6 - Advances in Steel Technology	P/M Current Research and Industrial Practice I	P/M Current Research and Industrial Practice II	P/M Current Research and Industrial Practice III	P/M Current Research and Industrial Practice IV	P/M Current Research and Industrial Practice V
Rosewood	Symposium to Honor Professor Julia Weertman I	Symposium to Honor Professor Julia Weertman II	Symposium to Honor Professor Julia Weertman III	Symposium to Honor Professor Julia Weertman IV	Symposium to Honor Professor Julia Weertman V	Symposium to Honor Professor Julia Weertman VI	
Rookwood	Design in Materials Education	GA 7 - Environmental and Chemical Degradation of Materials	Processing and Properties of Structural Nanomaterials I	Processing and Properties of Structural Nanomaterials II	Processing and Properties of Structural Nanomaterials III	Processing and Properties of Structural Nanomaterials IV	Processing and Properties of Structural Nanomaterials V
Salon B & C	Powder Processing of Light Metal Alloys I	Powder Processing of Light Metal Alloys II	Pb Free and Pb Bearing Solders I	Pb Free and Pb Bearing Solders II	GA 8 - Microstructure and Mechanical Properties I	GA 9 - Microstructure and Mechanical Properties II	
Salon D & E	Dynamic Behavior of Composites I	Dynamic Behavior of Composites II	New Opportunities in MMC Research and Applications I	New Opportunities in MMC Research and Applications II	GA 1 - High Temperature Behavior I	GA 2 - High Temperature Behavior II	
Salon F & G	Defects in Solidification Processing I	Defects in Solidification Processing II	Advances in Casting High Temperature Alloys	GA 3 - Advances in Processing and Heat Treatment I	GA 4 - Advances in Processing and Heat Treatment II	GA 5 - Advances in Processing and Heat Treatment III	
Salon H & I	Modeling the Performance of Structural Materials I Young Leaders Tutorial Lecture	Modeling the Performance of Structural Materials II	Hydrogen Effect on Materials Behavior I	Hydrogen Effect on Materials Behavior II	Hydrogen Effect on Materials Behavior III	Hydrogen Effect on Materials Behavior IV	
Salon M			Beryllium and Beryllium Alloys I	Beryllium and Beryllium Alloys II	Beryllium and Beryllium Alloys III	Beryllium and Beryllium Alloys IV	

Daily Personal Schedule

Monday, November 1, 1999

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
10:30 am				
11:00 am				
11:30 am				
12:00 pm				
12:30 pm				
1:00 pm				
1:30 pm				
2:00 pm				
2:30 pm				
3:00 pm				
3:30 pm				
4:00 pm				
4:30 pm				
5:00 pm				
5:30 pm				
6:00 pm				
6:30 pm				

Defects in Solidification Processing: Defects in Solidification Processing I

Sponsored by: Materials Processing and Manufacturing Division, Solidification Committee
Program Organizers: Linda L. Rishel, Carnegie Mellon University, Materials Science & Engineering Department, Pittsburgh, PA 15213 USA; Ralph Napolitano, Iowa State University, Ames, IA 50010 USA; Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

Monday AM
November 1, 1999

Room: Salon F&G
Location: Omni Netherland Plaza Hotel

Session Chairs: Linda L. Rishel, Carnegie Mellon University, Mats. Sci. and Eng. Dept., Pittsburgh, PA 15213 USA; Mark Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Prediction of Freckles and Spurious Grains in Directional Solidification of Single-Crystal Ni-base Superalloys: *Christoph Beckermann*[1]; [1]University of Iowa, Mech. Eng., 2412 SC, Iowa City, IA 52242 USA

An overview is provided of recent progress in modeling freckle defects and spurious grains in directional solidification of single-crystal Ni-base superalloys. The model is based on two-dimensional flow, heat transfer, and species transport calculations coupled with a subroutine for calculating the multi-component thermodynamic equilibrium during solidification of Ni-base superalloys. In addition, a model is developed for the transport and remelting of dendrite fragments. Results are presented for freckle formation as a function of the angle of the thermal gradient with respect to gravity. Conditions are established for dendrite fragments to lead to spurious grains or even to a columnar-to-equiaxed transition. The predictions for the initiation of freckles are compared to experiments.

9:10 AM Invited
Carbon Additions and Their Influence on Grain Defect Formation in Nickel-Base Single Crystals: Sammy Tin[1]; *Tresa Pollock*[1]; Wendy Murphy[2]; [1]Carnegie Mellon University, Mats. Sci. and Eng. Dept., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]General Electric Aircraft Engines, One Neumann Way, Cincinnati, OH 45215 USA

Solidification of large single crystals of multicomponent nickel-base superalloys is challenging due to the potential for formation of a variety of grain defects. While it is well known that thermosolutal convective instabilities contribute to the breakdown of single crystal solidification in these materials, the relationship between alloy chemistry and the series of processes that ultimately result in these defects is still not well understood. Recent solidification experiments on a series of single crystal superalloys with varying levels of refractory alloy element additions have shown an unusually strong beneficial effect of carbon on the incidence of grain defects that develop during solidification. These additions of carbon, up to 0.1wt%, result in the formation of Ta-rich MC type carbides that precipitate near the liquidus temperature. Details of these experiments, along with a discussion of possible mechanisms for the beneficial effect of carbon will be discussed.

9:45 AM Invited
Orientation Control and the Stability of Spurious Grains During Single Crystal Processing of Nickel-Base Superalloys: N. D'Souza[1]; M. G. Ardakani[1]; B. A. Shollock[1]; *Malcolm McLean*[1]; [1]Imperial College of Science, Technology and Medicine, Dept. of Mats., Prince Consort Rd., London SW7 2BP UK

The grain morphologies and crystallographic textures produced during directional solidification of the Ni-base superalloys, CMSX 4 and CM186LC, have been determined for a range of solidification conditions that mimic those occurring in large industrial castings. Although the compositions and physical constants of the two alloys are similar, the solidification responses are quite different leading to diffuse and sharp <001> textures respectively. The curvature of the solid/liquid interface has been determined for each solidification condition and the final axial texture attained during directional solidification is shown to be dependent on competitive grain growth that is influenced by the local curvature of the liquidus isotherms and by the alloy melting range. Spurious grains have been deliberately induced during single crystal processing of CMSX4 and the orientations of those that continue to grow have been related to both the orientation of the single crystal matrix and of the macroscopic solidification direction. The extended grain selection criterion, taking into account the local slope of the solid-liquid interface, satisfactorily accounts for the stability of randomly nucleated edge defects. The implications of the study to single crystal processing of large castings is considered.

10:20 AM Break

10:35 AM
The Effect of Primary Axis Misorientation and Low Angle Boundaries on the Mechanical Properties of CMSX-10: *Yakin Patel*[1]; Paul Browning[1]; Allen R. Price[2]; Tammy M. Simpson[2]; [1]Solar Turbines Inc., 2200 Pacific Hwy., P.O. Box 85376, MZ R1, San Diego, CA 92122 USA; [2]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49461-1895 USA

The U. S. Department of Energy's Advanced Turbine Systems (ATS) program supports the development of high efficiency gas turbines for industrial and power generation applications. As part of this program, Solar Turbines is considering the 3rd generation single crystal alloy CMSX-10 for turbine blade application in its ATS engine, the Mercury-50. To maximize casting yield for this component, while ensuring critical mechanical property requirements, the tolerance of CMSX-10 to typical casting defects was investigated. Single crystal specimens with primary axis misorientation between 0 and 30 degrees and bicrystal specimens with low angle boundaries (LABs) between 0 and 14 degrees of misorientation were used. The effects of primary axis misorientation and LABs on the mechanical properties were evaluated. Primary axis misorientation of greater than 15 degrees reduced the tensile and high-cycle fatigue properties. Similarly, the stress rupture and creep rupture properties were affected modestly for the primary axis misorientation of greater than 20 degrees. LABs greater than 10 degrees of misorientation reduced the stress rupture life. Also, LABs greater than 12 degrees of misorientation reduced the tensile and high-cycle fatigue properties.

11:00 AM Keynote

Melt Cleanliness-Advances in Liquid Steel Processing: *Alan W. Cramb*[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The last ten years have been revolutionary in liquid steel processing as industrial processing techniques have been implemented that have allowed chemistry control to ppm levels. In addition to solute chemistry control, there has been a large focus on inclusion control and engineering as the link between inclusion size, distribution and chemistry and mechanical properties has been more clearly understood. This talk will discuss the most recent advances in liquid steel processing from a fundamental point of view and indicate the current philosophies of inclusion engineering that are becoming more prevalent within the steel industry.

Design in Materials Education: Reinvigorating the Materials Design Competition

Sponsored by: Materials Processing and Manufacturing Division, Education Committee
Program Organizers: Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA; Zi-kui Liu, Pennsylvania State University, University Park, PA USA

Monday AM
November 1, 1999
Room: Rookwood
Location: Omni Netherland Plaza Hotel

Session Chairs: Mark Palmer, Virginia Commonwealth University, Richmond VA 23284-3015 USA; Zi Kui Liu, Pennsylvania State University, University Park, PA USA

8:30 AM Opening Remarks-

Re-establishing the Student Design Competition

The MPMD will review the status of the completion and the purpose of this session.

8:40 AM Keynote Address

9:10 AM
Reinvigorating the Student Design Competition:

The MPMD Council would like to re-establish the Materials Design Competition and take the necessary steps to ensure maximum participation by students and universities. The purpose of this session is to find out what is required of students taking the senior-level design courses. There will be a series of presentations by faculty from different institutions, a panel discussion, and a brief working session where the participants will summarize the findings of the symposium and make recommendations to the MPMD Council.

9:40 AM
A Senior Undergraduate Design Problem in Process and Physical Metallurgy: Carburizing of Steel: *Ravi K. S. Ravichandran*[1]; [1]University of Utah, Dept. of Metallu. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

A design problem on carburizing of steel is given as a part of the capstone design course on Process Synthesis, Design and Economics in the senior year, at the Department of Metallurgical Engineering, University of Utah. The problem is intended to expose the students to the practical issues involved in designing a metallurgical process for the carburization of steel and evaluating its feasibility on the basis of economic factors. The design problem tests on students' understanding of concepts of diffusion, gas reactions, reaction rates, physical metallurgy and heat treatment of steel, testing and quality control. The students are asked to consider different steels for carburizing and are required to select one based on the coast of overall process. The steel is required to satisfy a set of mechanical property specifications. The students are asked to design a complete carburizing shop, including consideration of (i) purchase of carburizing furnace, gases and other equipment, detailed process calculations including atmosphere control and the determination of carburizing time and temperature and (ii) the overall cost of the process performed in-house versus subcontracting to an outside source. In this presentation, the details of the problem, the students' experiences as well as problems in working out this design problem will be presented.

10:10 AM
A Senior Undergraduate Design Problem for Mechanical Metallurgy Courses: Material Selection Design for Gas Turbine Blades: *Ravi K. S. Ravichandran*[1]; [1]University of Utah, Dept. of Metallu. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

A design problem involving material selection and design for compressor blades in gas turbine engines is given as a part of the Mechanical Metallurgy course in the senior year, at the Department of Metallurgical Engineering, University of Utah. The problem is intended to expose the students to the issues related to sorting of mechanical property data and the application of mechanical metallurgy concepts to the resolution of an engineering problem. The design problem involves consideration of density, low and high temperature strength, fatigue, creep deformation and rupture data as well as cost, in material selection for a turbine blade. The students are asked to consider five different materials as candidates and are required to select one based on least weight and the ability to meet the stringent mechanical property requirements. The problem has been designed to evaluate students understanding of concepts on strength, fatigue, creep that is usually done in the form of lectures in the later part of Mechanical Metallurgy courses. In addition, the students are required to dig out the required material data from handbooks, research papers and reports. For the past four years since its introduction in the course, very positive responses from the students, regarding this design problem are usually received from the students. In this presentation, the details of the problem, the students' experiences as well as problems in working out this design problem will be presented.

10:40 AM Panel Discussion
The speakers and representatives from MPMD will answer questions about the goals of the design competition and the Senior Design Course.

11:25 AM Working Session
Interested attendees will prepare a summary of the session for review by the MPMD Council, which will include recommendations for the competition.

Dynamic Behavior of Composites: CMC's & PMC's

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Structural Materials Committee
Program Organizers: Rick Hall, University of Delaware, Department of Mechanical Engineering, Newark, DE 19716 USA; Rusty Gray, Los Alamos National Laboratory, Dynamic Properties, Los Alamos, NM 87545-0001 USA; Ken S. Vecchio, University of California, Department of Ames, San Diego, CA 92093 USA

Monday AM
November 1, 1999
Room: Salon D&E
Location: Omni Netherland Plaza Hotel

Session Chairs: Ian W. Hall, University of Delaware, Dept. of Mech. Eng., Newark, DE 19716 USA; Kenneth S. Vecchio, Mats. Sci. Grp., Dept. of Mech. & Aero. Eng., La Jolla, CA USA

8:30 AM
Damping Behavior of Epoxy/Aluminum Composites: *Elizabeth M. Forsbacka*[1]; Catherine R. Wong[2]; Sreeramamurthy Ankem[3]; [1]NASA/Goddard Space Flight Ctr., Component Tech. and Radiation Effects Brnch., Code 562, Greenbelt, MD 20771 USA; [2]Naval Surface Warfare Ctr., Carderock Div., Code 612, West Bethesda, MD 20817 USA; [3]University of Maryland, Dept. of Mats. & Nuclear Eng., Bldg. 090, College Park, MD 20742-2115 USA

The aim of this investigation was to study the effect of aluminum particle size and volume fraction on the damping behavior of epoxy/aluminum composites. The damping capacity of the composites was measured in a fixed-guided-cantilever set-up. Tests were performed on composites with varying particle volume fractions

and with varying particle sizes. Tests were also performed at two different temperatures-below the glass transition temperature (Tg) and above Tg-in order to create a low modulus and a high modulus material within the same sample. Based on the properties of the constituent phases, upper and lower bound loss factors were calculated for various composites and compared with the experimental results. These results were also compared with previous experimental results and a two-dimensional finite element analysis (FEM) model. In general, the damping of a given composite increased with an increase in aluminum particle size, which is in agreement with the FEM results. Details of the investigation will be presented.

9:00 AM

Mechanical Behavior of Estane-Glass Composites as a Function of Temperature and Strain Rate: *George T. Gray*[1]; Carl M. Cady[1]; William R. Blumenthal[1]; [1]Los Alamos National Laboratory, MST-8, MailStop G755, Bldg. 1698, Los Alamos, NM 87545 USA

The high-rate mechanical properties of polymeric composites are of interest for the development of physically-based constitutive materials models for use in predictive finite-element simulations of collateral damage in propellants and energetics. The compressive stress-strain responses of three estane-glass composites containing 35, 50, and 65 vol.% glass spheres have been measured as a function of strain rate, 10^{-3} to 6000 s^{-1}, and temperature,-55 to +55°C. High-strain-rate tests were performed on a specially-designed split-Hopkinson-pressure bar (SHPB) facility designed at LANL utilizing Ti-6Al-4V and/or Mg loading bars. Lower impedance pressure bars are used instead of the more traditional maraging-steel bars to achieve higher signal to noise levels and therefore higher precision data on the relatively low-strength plastic composites. The critical importance of assuring uniform stress state in SHPB samples is graphically demonstrated by comparing/contrasting stress values obtained using a 1-wave versus a 2-wave analysis. Work performed under the auspices of the U.S. Department of Energy.

9:30 AM

High Strain Rate Testing of a Gr/Epoxy Model Composite: Mustafa Guden[1]; *Ian W. Hall*[2]; [1]Izmir Institute of Technology, Cankaya, Izmir, Turkey; [2]University of Delaware, Dept. of Mech. Eng., Newark, DE 19716-3104 USA

In order to understand better the fracture occurring modes in composites subjected to high strain rate testing, and to further investigate previous reports of strain rate sensitivity in such samples, unidirectional graphite epoxy composite plates 12mm thick were fabricated and cylindrical samples prepared for testing in the longitudinal and both transverse directions. Tests were conducted on the Split Hopkinson Pressure Bar with and without pulse shapers at strain rates up to 2000/s. Also, in order to determine the typical fracture modes in each case, material was recovered from fractured samples immediately after passage of the first compressive wave. This was achieved by using heavy spacing collars to restrict total displacement of the bars. Results indicate no strain rate sensitivity of the failure strength or failure strain in any orientation within the high strain rate range investigated. Typical failure modes are described.

10:00 AM Break

10:10 AM

Quasi-Static and Dynamic Mechanical Response of Mollusk Shells: *Marc A. Meyers*[1]; K. S. Vecchio[1]; Rainer Menig[2]; Marc H. Meyers[3]; [1]UCSD, Dept. of AMES, La Jolla, CA 92093 USA; [2]University of Karlsruhe, Institute for Mats. Rsrch. fur Werkstoffkunde 1, Karlsruhe Germany; [3]UCSD, Dept. of Biology, La Jolla, CA 92093 USA

Quasi-static and dynamic compression and three-point bending tests have been carried out on Strombus Gigas (conch) and Haliotis Rufescens (abalone) shells. The mechanical response of the conch and abalone shells is correlated with their respective microstructures and damage mechanisms. The mechanical response is found to vary significantly from specimen to specimen and requires the application of Weibull statistics in order to be quantitatively evaluated. Both the conch and abalone shells exhibited orientation dependence of strength as well as significant strain-rate sensitivity; the failure strength at loading rates between 10 x 103 and 25 x 103 GPa/s was approximately 50% higher than the quasi-static strength. The abalone compressive strength when loaded perpendicular to the shell surface was approximately 50% higher than parallel to the shell surface. The compressive strength of abalone is 1.5 3 times the tensile strength (as determined from flexural tests), in contrast with monolithic ceramics, for which the compressive strength is typically an order of magnitude greater than the tensile strength. Quasi-static compressive failure in both shells occurred gradually, in a mode sometimes described as “graceful failure”. The shear strength of the organic/ceramic interfaces of Haliotis Rufescens (abalone) was determined to be approximately 30 MPa by means of a shear test. Considerable inelastic deformation of the organic layers (up to a shear strain of 0.4) preceded failure. Crack deflection, delocalization of damage, plastic microbuckling (kinking), and viscoplastic deformation of the organic layers are the most important mechanisms contributing to the unique mechanical properties of these shells. Research supported by US Army Research Office MURI Program.

10:40 AM

Dynamic Fracture Toughness of Woven Composites: *J. D. McGee*[1]; Sia Nemat-Nasser[1]; [1]University of California, San Diego, Dept. of Applied Mech. and Eng. Sci., Ctr. of Excell. for Adv. Mats., 9500 Gilman Dr., La Jolla, CA 92093 USA

Woven laminate composites are being considered as structural backing material for composite hybrid armors. Impact induced delamination of laminate composite is undesirable in terms of maintenance and structural integrity of the armored vehicle. On the other hand, delamination provides a mechanism to absorb kinetic energy of an impact event. The impact induced delamination occurs dynamically and is a complex interaction of stress waves in the material and structural deflection. In order to better understand the relationship between energy required to create the surface of the delamination and associated impact damage, a new technique is developed that makes use of a tension Hopkinson bar to evaluate dynamic fracture toughness. Dynamic fracture toughness is compared with values obtained from double cantilevered beam tests and impact tests.

11:10 AM

Influence of Microstructure on Static and Dynamic Fracture Toughness of Reactively-Processed B4C/SiC Ceramic-Matrix Composites: *Jerry C. LaSalvia*[2]; Kenneth S. Vecchio[1]; Jeffrey J. Swab[2]; Parimal J. Patel[2]; Gary A. Gilde[2]; [1]University of California at San Diego, Mats. Sci. Grp., Dept. of AMES, 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]U. S. Army Research Laboratory, Weapons and Matls. Rsrch. Directorate, Aberdeen, MD 21005-5069 USA

Over the years, a number of researchers have attempted to improve the performance of B4C through the formation of composites (e.g. B4C/Al). The formation of ceramic-matrix composites has lead to a class of structural materials with increased damage tolerance (e.g. R-curve behavior). While it is largely believed that composites do offer a means for improving ballistic performance through increasing damage tolerance, little has been done to systematically investigate the influence of microstructural parameters on relevant mechanical properties (i.e. indicators for performance). At typical muzzle velocities for the above threats (Ä 850 m/s), the initial loss in penetration resistance of a ceramic is due to the initiation of microcracks both beneath the point-of-impact (mixed mode, shear dominated) and at the back surface of the tile (mode I, tensile). As a result, short-crack fracture toughness (static and dynamic) is considered to be an important indicator for performance. As part of an on-going opaque armor ceramics improve-

ment effort at the U.S. Army Research Laboratory, B4C/SiC ceramic-matrix composites were produced by reactively hot-pressing B4Si, B6Si, and carbon powder mixtures. The morphology of the resulting phases are strongly affected by the source of carbon used (carbon black versus graphite). In either case, both phases appear to be continuous and interpenetrating. The influence of processing parameters on the resulting microstructure, phases, and mechanical properties will be presented. Microstructure and phases were characterized by scanning electron microscopy and X-ray diffraction, respectively. Mechanical properties such as hardness, four-point bend strength, and fracture toughness were also determined. Dynamic fracture toughness were determined using a modified Hopkinson bar apparatus. Commercially available armor-grade B4C was used as a baseline for comparison of microstructures, phases, and mechanical properties.

Modeling the Performance of Structural Materials: Deformation, Aging, and Environmental Effects

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712-1063 USA; Donald Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

Monday AM — Room: Salon H&I
November 1, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: Eric M. Taleff, The University of Texas at Austin, Texas Materials Institute, Austin, TX 78712-1063 USA

8:30 AM
Cogging Process Modeling of TIMETAL-17: *Vasisht Venkatesh*[1]; Stephen P. Fox[1]; [1]TIMET, Henderson Tech. Labs., 8000 W. Lake Mead Dr., Henderson, NV 89015 USA

The development of accurate modeling capabilities for the primary conversion process of Titanium alloys such as TIMETAL-17 (Ti-5Al-2Sn-2Zr-4Cr-4Mo), a forging alloy for gas turbine engines, continues to be an important area of research. A 4.4 x 4.4 x 6.9 bar of TIMETAL-17 at 1600°F, was forged incrementally along its length, in four passes using an instrumented 500 ton press. Process parameters, e.g., press loads, stroke and velocity, and metal (workpiece) temperature, measured by two embedded thermocouples, were continuously logged into a PC for model validation. The cogging process was then modeled utilizing DEFORM-3D, a three dimensional finite-element analysis software code. Flow stress data, from prior isothermal upset forging, and thermal conductivity and heat capacity data from heating/cooling experiments were used to create a thermo-mechanical database within DEFORM-3D. Model predictions of the final bar geometry, press loads and bar temperatures showed good agreement with the corresponding experimental measurements. References [1] S. P. Fox and D. F. Neal, The Role of Computer Modeling in the Development of Large Scale Primary Forging of Titanium Alloys, Titanium 95: Science and Technology, P. A. Blenkinsop, W. J. Evans and H. M. Flower, eds., The Institute of Metals, London, UK, 1996, pp.628-635. [2] G. Dumas, Y. M. Saunier, F. Poirier and E. Wey, Control of Large Diameter Ti-6-4 Billet Microstructure through Modeling of the Forging Process, Titanium-95: Science and Technology, P. A. Blenkinsop, W. J. Evans and H. M. Flower, eds., The Institute of Metals, London, UK, 1996, pp.651-658. [3] P. Dadras and J. F. Thomas, Jr., Characterization and Modeling for Forging Deformation of Ti-6Al-2Sn-4Zr-2Mo-0.1Si, Metall. Trans., 12A, November, 1981, 1867-1876. [4] D. Zhao, C. Cheng, R. Anbajagane, H. Dong and F. S. Suarez, Three-Dimensional Computer Simulation of Alloy 718, Superalloys 718, 625, 706 and Various Derivatives, E. A. Loria, eds., 1997, pp. 163-172. [5] J. P. Domblesky and R. Shivpuri, Development and validation of a finite-element model for multiple-pass radial forging, Journal of Materials Proc. Tech., 55, 1995, pp. 432-441.

8:50 AM
Modeling of Strain Hardening of Titanium under Different Strain States; The Role of Mechanical Twinning: *Ayman A. Hussien*[1]; Surya Kalidindi[1]; [1]Drexel University, Mats. Eng. Dept., 32nd and Market St., Philadelphia, PA 19104 USA

A finite element simulation is being carried out to simulate the stress strain response using a modified Taylor model to include both slipping and twinning. The models are designed to simulate the experimental results currently available for simple compression, palne-strain compression, and simple shear tests on annealed, polycrystalline samples of high purity titanium. The strain hardening rates were numerically computed, normalized with shear modulus (G) and plotted against ($s-s_o/G$ (where s_o being the initial yield strength of the material) and e. These plots for titanium is found to be almost identical to those of 70/30 brass and other low stacking fault energy fcc metals that undergo mechanical twinning and the results showed a similar reduction of strain hardening in shear as compared to that in compression, previously found for the fcc metals. Microscopy revealed deformation twins but ones significantly different from the fcc metals-the twins were thicker but more widely spaced.

9:10 AM
The Creep of Solid Solution Tungsten-Tantalum Alloys: *Jian Hu*[1]; Hong Gao[2]; Ralph Zee[1]; [1]Auburn University, Mats. Rsrch. and Edu. Ctr., 202 Ross Hall, Auburn, AL 36849 USA; [2]Applied Materials Inc., Physical Vapor Deposition, Austin, TX 78724 USA

W-5%Ta single crystals about 10 mm in diameter and 50 mm in length have been grown at Auburn University from arc cast polycrystalline rods using an electron beam zone melting method. Compression specimens of typical geometry of about 4.6 mm in diameter and 4.6 in length were prepared using EDM machining and followed by electrical polishing to obtain satisfied surface condition. The single crystal and polycrystalline specimens were then tested at elevated temperatures between 1273K and 1773K in high purity Ar atmosphere, with constant crosshead speeds giving initial strain rates between 10^{-5} and 10^{-3} s^{-1}. The creep behavior of W-5%Ta has been predicted based on the obtained stress-strain curves and a predictive model developed earlier by investigators at Auburn University for solid solution refractory alloys. It is shown that Ta addition strengthens tungsten and the improvement in the creep strength of tungsten-tantalum alloys is predicted.

9:30 AM
Modeling of Solute Drag during Creep of Al-Mg-X Alloys: Sean J. Yoon[1]; *Eric M. Taleff*[1]; [1]The University of Texas, Texas Mats. Instit., Austin, TX 78712-1063 USA

Al alloys containing approximately 3 wt pct and higher concentrations of Mg can exhibit solute-drag creep at elevated temperatures and slow strain rates. Solute-drag creep occurs because of drag on dislocation glide motion from the interaction of dissolved Mg solute atoms with dislocations. The interactions which give rise to solute drag in binary alloys have been explained by many investigators with micro-mechanics theories using linear elasticity. Recent investigations have seen slight changes in solute-drag creep of Al-Mg alloys containing ternary alloying additions, such as Mn, Fe, Zn, and Cu, in comparison with binary Al-Mg alloys. The present investigation attempts to model the solute-drag mechanism in order to better understand the behavior of such ternary alloys.

9:50 AM Break

10:00 AM
A Process Model for the Age-Hardening Response of a Type 319 Cast Aluminum Alloy: *Carla A. Cloutier*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Dept. of Mats. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Sci. Rsrch. Lab., Dearborn, MI USA

The use of cast 319 aluminum in automotive applications, especially in engine blocks and cylinder heads, has increased dramatically in the last decade. Competitive pressures have necessitated the development of an optimized heat treatment process for this alloy. Based on an approach suggested by Shercliff and Ashby, a process model was developed to predict the changes in yield strength that result from isothermal aging. Aging curves were developed for cast 319 aluminum with three different levels of copper: 3.0, 3.5 and 4.0%. Aging temperatures ranged from 150-290°C. The components of the model will be reviewed with emphasis on variations due to volume fraction of precipitate and solidification rates. The results show that the aging process of cast 319 aluminum can be reasonably predicted by such and age hardening model.

10:20 AM
Modeling Residual Stresses in 319 during Heat Treatment: *Matt L. Newman*[1]; Jonathan A. Dantzig[1]; Huseyin Sehitoglu[1]; [1]University of Illinois, Mech. and Industrial Eng., 140 Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

Desired weight savings and increased performance have led to the replacement of steel by aluminum alloys in many engine applications. Most aluminum alloys must undergo a heat treatment procedure after casting to attain the desired properties. This heat treatment usually involves quenching from the high temperature solid solution, followed by a controlled aging treatment at an intermediate temperature. The rapid, spatially varying thermal deformations experienced during the quench can induce large-scale plastic deformation and distortion in the part, as well as significant residual stresses. We wish to mitigate these effects by improved process design, using computational modeling. The model development is done in conjunction with an extensive experimental testing program to provide a fundamental understanding of the physical processes. Emphasis is placed on model accuracy, simplicity, and potential application to commercial finite-element codes. Constitutive models are implemented in a semi-analytical model of a beam-quenching problem to predict deformation, and optimization is used to refine model parameters.

10:40 AM
Aging Characteristics of Ti-44Al-xNb Alloys: *Carlos Hernandez*[1]; Shailendra K. Varma[1]; Rabindra Mahapatra[2]; Erica Corral[1]; [1]The University of Texas at El Paso, Dept. of Metallu. and Mats. Eng., El Paso, TX 79968-0520 USA; [2]Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20670-1908 USA

Aging characteristics of four alloys from Ti-Al-Nb system have been investigated to determine the microconstituents. Ti-44Al, Ti-44Al-4Nb, Ti-44Al-8Nb and Ti-44Al-12Nb alloys have been heat treated at 1200°C for 4 hours and quenched in ice water. The samples were then aged at 850, 900, 950 and 1000°C for 4 hours and again quenched in ice water. The higher hardness values are associated with higher amounts of g phase in the microstructure. At lower aging temperatures, presence of Widmanstatten a2 has been observed while a typical duplex structure has been found at higher aging temperatures.

11:00 AM
Cyclic and Static Oxidation Characteristics of Ti-44Al-xNb Alloys: *Alvaro Chan*[1]; Shailendra K. Varma[1]; Rabindra Mahapatra[2]; [1]The University of Texas at El Paso, Dept. of Metallu. and Mats. Eng., El Paso, TX 79968-0520 USA; [2]Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20670-1908 USA

Cyclic and static oxidation characteristics of Ti-44Al-xNb alloys have been determined in a range of temperature from 850 to 1000°C. Ti-44Al, Ti-44Al-4Nb, Ti-44Al-8Nb, and Ti-44Al-12Nb alloys have been subjected to cyclic oxidation at the indicated temperatures where one cycle consists of 55 minutes of heating followed by 5 minutes of air cooling. The study was conducted for a period of one week. The oxidation curves were plotted from the data of weight gain as a function of number of cycles. The static oxidation consists of heating the sample without interruption. These oxidation curves for polycrystalline materials were compared with the curves earlier reported for single crystals of T-44Al-11Nb alloy in order to determine the effect of Nb on the oxidation behavior of alloys from this ternary system. Microstructure relationship with the oxidation behavior has been explored by TEM.

Powder Processing of Light Metal Alloys: Light Metal Alloy Processing

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Composite Materials Committee, Powder Materials Committee
Program Organizers: Joseph W. Newkirk, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-0340 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; James C. Foley, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Marvin G. McKimpson, Michigan Technology University, Institute of Materials Processing, Houghton, MI 49931-1295 USA; James W. Sears, Lockheed Martin, KAPL, Inc., D2, 114, Schenectedy, NY 12301 USA

Monday AM — Room: Salon B&C
November 1, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: Marvin McKimpson, Michigan Technology University, Houghton, MI USA

8:30 AM Invited
Accelerated Maturation of P/M Light Materials: *Hamish Fraser*[1]; [1]Ohio State University, Mats. Sci., Columbus, OH 43210 USA
Abstract Text Not Available

9:00 AM
Simplified Aluminum Powder Metallurgy Processing for Structural Applications: *Iver E. Anderson*[1]; James C. Foley[1]; [1]Ames Laboratory, Metallu. and Ceramics Pgm., 126 Metals Dev., Ames, IA 50011 USA

The need for lighter and more fuel-efficient vehicles has spurred research in aluminum powder metallurgy (P/M) processing. Replacing steel components with Al alloys can provide a 50-70% weight savings and reduced horsepower requirements for drive train parts. Although there are Al P/M alloys like 201AB and 601AB for structural components, mass production of P/M parts from more advanced alloys has been limited. Processing of aerospace Al alloys requires complicated hot working steps that are cost prohibitive. Many of the costly processing steps are aimed at overcoming the effects of the tenacious Al oxide surface film typical of commercial Al powder. Simplified P/M technologies may be developed if less oxidized Al powders are used. An alternative "clean" Al atomization technique will be described along with detailed comparisons of this powder to commercial Al powder, including explosivity measurements and solid state sintering observations. Support is from the DOE-BES under contract No. W-7405-Eng-82.

9:20 AM
Microstructural Characterisation of Direct Laser Fabricated TiAl: *D. Srivastava*[1]; I. T. H. Chang[1]; M. H. Loretto[1]; [1]The University of Birmingham, IRC in Mats. for High Performance Apps., Birmingham B15 2TT UK

Titanium aluminide alloys of near equiatomic composition, are important candidate materials for their potential applications in the aerospace and automotive industry because of their superior mechanical properties at high temperature. However, these alloys, are difficult to process by conventional processing route because of their low ductility and toughness at ambient temperature. Near net shape technologies such as direct laser fabrication are receiving significant interest as a possible fabrication method of components directly from powder. In the present work, near net shape samples of Ti48Al2Mn2Nb alloy have been fabricated by direct laser fabrication using gas-atomised powders as a feedstock material. The evolution of microstructure in the laser treated samples has been examined using optical, scanning (SEM) and transmission electron microscopy (TEM) and compared with the conventionally processed materials. It has been observed that the microstructure is very sensitive to the processing parameters of the direct laser fabrication process. The microstrucure of the laser treated samples has been found to be heterogeneous in nature and extremely fine in comparison to the conventionally processed material. Heat treatments for a number of conditions have been carried out to examine the stability of the microstructure. It has been observed that the microstructure of the samples heat treated at 1273K for more than 24 hours has coarsened. The TEM examination has shown that the microstructures of laser treated samples are considerably modified after heat treatment. The mechanical properties of the direct laser fabricated samples are presented for both immediately after laser fabrication and after a range of heat treatments.

9:40 AM
The Development of In-Situ Sintering Characterization of Aluminum Powder Metallurgy Parts: *David K. Rehbein*[1]; James C. Foley[1]; [1]Ames Laboratory, Metallu. and Cera. Pgm., 211A Metals Dev., Ames, IA 50011 USA

In-situ characterizations of green state part density and sintering state have long been desired in the powder metal community. Recent advances in non-contact electromagnetic acoustic transducer (EMAT) technology have enabled in-situ evaluations of the acoustic attenuation and velocity as sintering proceeds. Pure aluminum (99.999%) and aluminum alloy (4.4% Cu-0.8% Si) powders were prepared with high pressure gas atomization (HPGA). The powders were pressed in a uniaxial die and examined with acoustic waves for changes in velocity and attenuation during sintering. The changes in acoustic properties were correlated with sample microstructures and mechanical properties. Evolution of the acoustic echo train during sintering is shown to provide information on the state of sintering as well as having the potential for detection of interior flaws. This work is funded by a laboratory directed research and development grant.

10:00 AM Break

10:10 AM Invited
P/M Titanium and Titanium Aluminide Technology Utilizing Gas Atomized Powder: *John H. Moll*[1]; [1]Crucible Materials Corporation, Crucible Rsrch., 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA

Gas atomization is being routinely used to produce high quality powders of titanium and titanium aluminide alloys. This paper describes the process and recent improvements aimed at increasing yield and improving cleanliness. A summary of the microstructural and mechanical property characteristics of P/M alloys will be presented and compared with conventionally processed material. Applications for gas atomized powder will be reviewed. These include composites, direct metal deposition via laser processing, titanium aluminide sheet for aerospace applications, chemical standards and sputtering targets.

10:40 AM
Quenching Studies of Al P/M Alloys Using the Jominy End Quench: *Joseph W. Newkirk*[1]; D. Scott MacKenzie[2]; Wei Yuan[1]; [1]University of Missouri-Rolla, Dept. of Met. Eng., Rolla, MO 65401 USA; [2]The Boeing Company, Failure Analysis & NDT, P.O. Box 516, MC S102-2152, St. Louis, MO 63166-0516 USA

Aluminum P/M alloys are becoming increasingly more important in transportation applications, such as automobiles. Most of these alloys are heat treatable in order to get high strength to weight ratios. Quenching of Al P/M alloys depends on section size and porosity level, as well as composition. In order to study the heat treatment of Al P/M alloys, the Jominy End Quench has been adapted to aluminum samples. Methods for studying the quenching of smaller P/M samples and the resulting properties that can be measured, such as hardness, conductivity, microstructure, etc. will be presented. The use of these techniques for more rapid process and alloy development will be discussed. Examples on commercial P/M alloys will be shown.

11:00 AM
Microstructural Characteristics of Spray Deposited Commercial Bearing Alloys: *O. P. Pandey*[1]; [1]Thapar Institute of Engineering & Technology, School of Basic & Appld. Sci., P.O. Box 32, Patiala 147004 India

Two commercial grade aluminum based immiscible bearing alloys were spray-deposited using a convergent-divergent type nozzle. The processing parameters for spray-deposition were adjusted in such a way that most of the droplets arrived on the deposition substrate in either the liquid or the semiliquid state. The microstructural features of spray-formed and as cast alloys are compared. In spray-formed alloys equiaxed grains are observed. The cell boundaries and intercellular regions were observed to be decorated with sub-micron sized particles, whereas in normal castings the second phase was observed to be segregated along grain boundaries. The morphology and distribution of second phases were observed to have similarity with those in over-spread and atomized powders produced under similar processing conditions. The microstructural features observed with variation in spray conditions are discussed in detail.

11:20 AM
Fabrication of TiAl Alloy by Blended Elemental Powders Semisolid Forming: *Cui'e Wen*[1]; Kazuo Yasue[1]; Yasuo Yamada[1]; [1]National Industrial Research Institute of Nagoya, Mats. Proc. Dept., 1-1 Hirate-cho Kita-ku, Nagoya 462-8510 Japan

Semisolid forming from blended elemental powders are investigated on Ti-Al alloy. The effects of the forming pressure, temperature, pressure holding time on the density of the green compacts are discussed. The forming method is outlined as follows: the mixture of the blended elemental powders are filled in a metal mold, and heated to approximately the melting point of aluminum. Thereafter, the forming pressure is loaded and held for the prescribed time. The pore-free green compacts are obtained under almost all the forming conditions. However, pores are observed after alloying heat treatment. The volume fraction of the pores depends on the microstructures of the green compacts. Experiments have also been carried out on the effects of the alloying heat treatment conditions on the mechanical properties of the compacts.

11:40 AM
Application of the Blow Casting Process on Titanium Alloy: *Kazuo Yasue*[1]; Cui'e Wen[1]; Koji Shimojima[1]; [1]National Industrial Research Institute of Nagoya, Mats. Processing Dept., Hirate-cho Kita-ku, Nagoya 462-8510 Japan

Because of the extremely high activity at the melting state, titanium alloys tend to react with various oxides during the casting process. Therefore, the crucibles and the materials of casting molds are strictly limited. Furthermore, the low specific heat and the low flowing performance in the melting state give rise to a kind of metal materials which is very difficult to cast. Accordingly, the method by using metal powders as casting mold materials has been investigated in the present paper. The relationships between the casting mold and the composition, the granularity, and the cooling-rate of the metal powders, as well as the reaction performance between the casting mold and the titanium-melt are discussed. Meanwhile, a new technique of blow casting process as loading gas-pressure during the process of melt-pouring has been developed in order to

improve the flowing performance. The relationship between the gas-pressure and the fulfilling degree of the melt is studied in detail.

Refractory Metals & Alloys—A Symposium on Research, Development & Applications I: V, Hf, Re, Rh and Their Alloys

Sponsored by: Structural Materials Division, Refractory Metals Committee, Corrosion and Environmental Effects Committee
Program Organizers: Mehmet Uz, Lafayette College, Chemical Engineering, Easton, PA 18042-1775 USA; Ken Natesan, Argonne National Laboratory, ET/212, Argonne, IL 60439-4838 USA

Monday AM
November 1, 1999
Room: Caprice 1&4
Location: Omni Netherland Plaza Hotel

Session Chairs: Ken Natesan, Argonne National Laboratory, Energy Tech. Div., Argonne, IL 60439 USA; Claude B. Reed, Argonne National Laboratory, Tech. Devel., Argonne, IL USA

8:30 AM Invited
Application of Vanadium Alloys in Fusion Systems: *Dale Smith*[1]; Ken Natesan[1]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

Vanadium alloys have been identified as a leading candidate low-activation structural material for fusion first-wall blanket applications. Candidate vanadium alloys exhibit favorable safety and environmental characteristics, good fabricability, high temperature and heat load capability, good compatibility with liquid metals and resistance to irradiation damage. The focus of the vanadium alloy development program has been on the vanadium-chromium-titanium (0-15% Cr, 1-20% Ti) alloy system. Investigations include effects of minor alloy elements such as Si, Al, and Y and substitution of iron for chromium in the ternary alloy. In recent years, significant progress has been made in the development of vanadium alloys in the areas of production/fabrication of the alloys, thermomechanical treatments (TMT) for the alloys, microstructural characterization as a function of TMT and exposure time and temperature, baseline mechanical properties of the alloys, irradiation effects on the structure/property changes in the materials, and corrosion/compatibility issues pertinent to fusion systems. The present paper will address the materials requirements for a fusion system and discuss the available information on several aspects of V-base alloys which meet those requirements. Work supported by Office of Fusion Energy, U.S. Department of Energy, under Contract W-31-109-Eng-38.

9:05 AM Invited
Reduction of Impurity Levels of Vanadium and Its Alloys for Fusion Applications: *T. Muroga*[1]; T. Nagasaka[1]; [1]National Institute of Fusion Science, Toki 509-5292 Japan

One of the key issues in the development of low activation vanadium alloys for the use in fusion reactors is the establishment of industrial basis toward large scale production. It is particularly important to develop the methodology to reduce interstitial impurities such as carbon, nitrogen and oxygen for the purpose of acquiring good workability, weldability and post-irradiation mechanical properties. It is also crucial to reduce the levels of some elements which will produce long half-lived radioactivity after fusion neutron irradiation. This paper reviews status of the collaboration of NIFS (National Institute for Fusion Science, Japan) with Japanese industries for the production of high-purity large ingot of a V-Cr-Ti alloy. The collaboration activities are summarized as follows. 1. Reduction of nitrogen level in the vanadium ingot by slight modification of the present industrial processes. 2. Investigation of the possible reduction of molybdenum level. 3. Development of the alloying methods minimizing the impurity pick-up. 4. Characterization of the vanadium and its alloys of reduced impurity revels. The goal of this collaboration is to produce a high purity V-4Cr-4Ti ingot of about 200 kg. The resulting ingot will be used for fabrication tests (sheeting, piping etc.), welding tests and various unirradiation and irradiation tests, which will be carried out by Japanese universities.

9:40 AM
Comparison of Oxidation Behavior of V-(4-5) Cr-(4-5) Ti Alloys in High and Low Oxygen Pressure Environments: *Ken Natesan*[1]; Mehmet Uz[2]; [1]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Lafayette College, Chem. Eng., Easton, PA 18042 USA

A study was conducted to compare the behavior of V-4 wt.% Cr-4 wt.% Ti and V-5 wt.% Cr-5 wt.% Ti alloys, which are potential candidates for first-wall and various other components of fusion reactors. The samples were in the form of 1-mm-thick coupons cut from a cold-rolled sheet. All samples were annealed for 1 h at 1050°C in ~10-7 torr vacuum prior to exposure in various oxygen environments. Oxidation experiments were conducted with a thermograv-imetric apparatus at 300-650°C in environments with pO2 in the range of 760-160 torr. A vacuum chamber equipped with a gas-flow regulator was used for experiments at 500-700°C in environments containing 5x10-6 0.1 torr oxygen. Weight-change data, measured as a function of time, was used to model the oxidation kinetics. Also, the cross-sectional area across the thickness of each sample was examined by both optical and scanning electron microscopy to establish the scale composition and oxygen distribution in the interior of the specimen. These results will be presented with emphasis on comparison of oxidation kinetics and microstructures of the two alloys as affected by oxygen pressure and temperature of the exposure environment. Work supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

10:00 AM
Pulsed Laser Welding of V-4Cr-4Ti Alloys: *Zhiyue Xu*[1]; Ken Natesan[2]; Claude B. Reed[1]; [1]Argonne National Laboratory, Tech. Dev. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

V-Cr-Ti alloys are among the leading candidate materials for the construction of the first wall and other structural materials applications in a fusion power reactor. Laser welding is considered an attractive process in the construction of a fusion reactor due to its high penetrating power and potential flexibility. A 1.6 kW pulsed Nd:YAG laser with fiber optic beam delivery was used to carry out bead-on-plate welding investigations on 4 mm thick V-4 wt%Cr-4 wt%Ti material. A systematic study was conducted to determine the effects of processing parameters on weld penetration, bead shape, and porosity. An empirical relationship between specific energy (P/DV) and the weld penetration (Z) was obtained. Deep penetration and porosity-free welds were achieved under an optimal combination of processing parameters including beam travel speed, pulse parameters, and shielding gas arrangement. The key for porosity-free welds was found to be the stabilization of the keyhole and providing an escape path for the gas trapped in the weld during laser welding. Microstructure and microhardness profiles of the welds will be presented and discussed.

10:20 AM Break

10:35 AM Invited
Mechanical Properties of Single Phase Rh-25Ti and Two Phase Rh-15Ti Alloys: *Shankar M.L. Sastry*[1]; Rabindra Nath Mahapatra[2]; [1]Washington University, Mech. Eng., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]Naval Air Systems Command, Mats. Lab., Patuxent River, MD 20670 USA

Rh-Ti is an attractive alloy system for high temperature applications. Rh-25 Ti is a single phase ordered intermetallic compound having a Cu3Au type crystal structure and therefore is an attractive candidate for developing "ductile" intermetallics.

Furthermore,two phase gamma/gamma' type microstructures found in nickel-based superalloys can also be produced in Rh-Ti alloys. In the present investigation a single phase Rh-Ti alloy of the composition Rh-25 at.% Ti and a two-phase Rh-Ti alloy of the composition Rh-15 at.% Ti were prepared by arc melting in vacuum. The arc-melted buttons were isothermally forged at 1100°C, and the mechanical properties at 25°C and 1000-1200°C were evaluated by microhardness measurements, compression testing, and 3-point bend testing. Unlike many other high temperature intermetallics, the Rh-Ti alloys do exhibit room temperature ductility and the high temperature strength and creep resistance are superior in Rh-Ti alloys than in some of the other high temperature intermetallics.

11:10 AM
Electroformed Rhenium Products: *Alexander Smirnov*[1]; [1]Engelhard-CLAL, L.P., 700 Blair Rd., Carteret, NJ 07008 USA

Electroforming complex rhenium shapes from molten salts is a relatively new fabrication method. The basis of the technology is the electrodeposition of a compact rhenium layer onto a mandrel of the required form. Tight tolerance can be achieved especially the concentricity of tubes, rocket nozzles or crucibles. The quality and the mechanical properties of the electroformed rhenium depend on the conditions of electroyses and the concentration of the oxygen-containing impurities in the electrolyte. The parameters studied include the type molten salts, temperature of the electrolyte, voltage, current density, etc. Tensile test results for electroformed deposits of rhenium will be discussed.

11:30 AM
Room Temperature Deformability of HfV2+Ta C15 Laves Phase Based Alloys: *Won-Yong Kim*[1]; David E. Luzzi[1]; *David P. Pope*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. and Eng., LRSM Bldg., 3231 Walnut St., Philadelphia, PA 19104 USA

Prior work has shown that the C15 (cubic) Laves phase HfV2 is made ductile by the addition of Nb, which promotes mechanical twinning at room temperature. It is important to determine whether this ductility is unique to this alloy system, or could be a general result in Laves phase alloys. In this study, Ta is chosen as the ternary alloying element in HfV2 due to its similar atomic size and chemistry with Nb. The phase diagram of the Hf-V-Ta alloy system is first investigated at 1373K using X-ray diffraction, optical microscopy and scanning electron microscopy equipped with wavelength-dispersive X-ray fluorescence spectroscopy. It is found that the C15 HfV2 Laves phase equilibrates directly with the vanadium-rich bcc phase without forming any intermediate phases. With the addition of Ta, the C15 phase field extends along the pseudo-binary line HfV2-TaV2 across the phase diagram with Ta substituting for Hf in HfV2. The lattice parameter of the C15 phase decreases with increasing Ta or V contents. Extensive room temperature ductility can be found in the two phase (Laves/bcc) alloys. For example, in one two-phase alloy with a Laves matrix and approximately equal volumes of the two phases, 2.3% plastic strain is achieved. The microstructures of undeformed and deformed materials are examined by transmission electron microscopy. It is confirmed that mechanical deformation of the Laves phase occurs in this material at room temperature. The deformation mechanisms responsible for this ductility will be discussed.

Symposium to Honor Professor Julia R. Weertman: Dislocations and Cavitation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials, Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Monday AM	Room: Rosewood
November 1, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Hans Weertman, Northwestern University, Evanston, IL 60208 USA; John G. Barker, NIST, Gaithersburg, MD USA

8:30 AM INTRODUCTION

8:35 AM Invited
Cyclic Deformation Dislocation Microstructures: *Michael E. Kassner*[1]; [1]Oregon State University, Mech. Eng., Rogers Hall, Corvallis, OR 97331 USA

Single crystals of aluminum and copper were cyclically deformed, in single slip, to presaturation at 77 and 293 K, respectively. The dislocations substructures were carefully analyzed using conventional BF and DF TEM with particular attention to the dislocation dipole spacing. It was found that, in both metals, the dipole spacing was independent of the location in the heterogeneous substructure, which consisted of dense dipole bundles (or veins), and the relatively low dislocation-density channels. Furthermore, the stress to separate the largest spacing dipoles was nearly equal to the applied stress. The stress necessary to pass dislocations through the dense veins was also about equal to the applied stress. The observations and calculations suggest a uniform state of stress throughout the heterogeneous dislocation substructure, without the presence of significant internal stresses. In-situ reversed deformation experiments were also performed in a HVEM. These results will be discussed in terms of dislocation dynamics and their consistency with the absence of long-range internal stresses.

9:05 AM Invited
Anisotropic Small Angle Neutron Scattering Studies of Alloys and Ceramics: *Andrew J. Allen*[1]; [1]NIST, Mats. Sci. and Eng. Lab., Bldg. 223, Rm. A163, 100 Bureau Dr. Stop 8523, Gaithersburg, MD 20899 USA

Small-angle neutron scattering (SANS) has become widely used to characterize material microstructures, sometimes by exploiting or amplifying anisotropies that are present. In SANS studies of ferritic steels, ferromagnetic domain broadening of the neutron beam must be suppressed by applying a saturation magnetic field, but the SANS non-magnetic and anisotropic magnetic components can then be analyzed independently to test precipitate phase and composition assumptions. In plasma-sprayed ceramic deposits, the Porod scattering anisotropy amplifies that in their void microstructures, and multiple-SANS (MSANS) beam-broadening measurements versus wavelength can be related to the coarse, concentrated void components. Recently, anisotropic MSANS methods have been developed to treat such multi-component systems, and have successfully obtained the mean crack and pore sizes, relative porosities and surface areas, and approximate crack and lamellar pore orientation distributions in plasma-sprayed ceramic coatings.

9:35 AM

Transmission Electron Microscopy Observations of As-Cast and of Compression-Tested Nb-15 at. % Al-10 at. % Ti,-25 at. % Ti and-40 at. Ti Alloys: *C. R. Brooks*[1]; D.-L. Guan[1]; P. K. Liaw[1]; E. A. Kenik[2]; C. T. Liu[2]; [1]University of Tennessee, Mats. Sci. & Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Mets. and Ceramics Div., Oak Ridge, TN 37831 USA

The as-cast structure of Nb-Al-Ti alloys containing 15 at. % Al and 10, 25 and 40 at. % Ti has been charcterized by transmission electron microscopy. The 10Ti alloy contained some d phase (based on Nb3Al) heterogeneously distributed on the matrix grain boundaries. The matrix had the ordered B2 structure. The 25Ti and 40Ti alloys were single phase and had the B2 structure. The electron diffraction patterns of the B2 structure showed diffuse intensities and streaking between fundamental spots. The domain structure was very fine and difficult to resolve. The structure of the as-cast alloys after compression testing to approximately 5% strain at 25, 600 and 900°C was also characterized. The B2 structure was retained in the tested specimens. No additional phases were identified, but the diffuse intensities and streaking were apparent in the diffraction patterns. In the specimens tested at elevated temperatures, the domain structure was easily resolved, with irregular boundaries. The increase in the domain size is due to aging during testing. The dislocation structure in the specimens tested at 25 and 600°C consisted of narrow intersecting bands with dislocation tangles between them. The bands were less defined for specimens tested at 900°C. It was found that the compressive strength increased with increasing Nb content, which is probably due to solid solution strengthening, and not to fine precipitates, such as the w and o phases.

9:50 AM

High Deformation Dislocation Microstructures: *Darcy A. Hughes*[1]; [1]Sandia National Laboratories, Ctr. for Mats. and Eng. Sci., P.O. Box 969, MS 9403, Livermore, CA 94551-0969 USA

Microstructures formed during high deformations have continuously captured both industrial and theoretical interest due to their large contribution to changes in materials properties. With the advancement of more automated experimental techniques and analyses it is possible to quantitatively characterize the different types of dislocation structures as they evolve with increasing deformation. This quantification includes, first, the hierarchical classification of dislocation boundary type and secondly, the determinations of both average values and distributions of boundary spacing and misorientation angle for each type. Results from these studies provide a clear evolutionary pattern that describes the characteristic components comprising a deformation microstructure. In general the evolution with increasing strain is marked by a persistent refinement of the cell block structure in terms of boundary spacing and a concomitant increase in boundary misorientation angle and distribution. Of special interest is the coexistance at all strains of boundaries with low to high misorientation angle. Similitude and scaling hypotheses are utilized to distill these statistical observations and provide a more physical understanding of the evolution and formation of the complex microstructures at large strain, based on observations from low to high strain. The consequences of these observations for the development of texture, stored energy and subsequent recrystallization are briefly discussed. This work was supported by the U.S. DOE under contract no. DE-AC04-94AL85000.

10:05 AM

Misfit Dislocation Development at Highly Mismatched Heterointerfaces: *Eric P. Kvam*[1]; Vidyut Gopal[1]; [1]Purdue University, School of Mats. Eng., W. Lafayette, IN 47907-1289 USA

Initial studies of mismatched epitaxial growths of fcc/fcc metals were used by Matthews, et alia, to predict and explain the introduction of glissile misfit-strain-relieving dislocations. After early confusion, it was found that these also predominantly explained dislocation introduction in strained semiconductor layers. At the higher mismatches available in some epitaxial semiconductor systems, however, basic assumptions of the theory no longer apply. In this study, we describe the behavior of misfit dislocation introduction in a high (> 11%) mismatch epitaxial growth system, InAs on GaP. Initial dislocation introduction occurs during the growth of the very first atomic/molecular layer on the surface, but does not fully accommodate the strain. Later dislocation introduction, to relieve the remaining strain, appears to follow the Matthews description, but with a twist: dislocations are introduced as glissile pairs, rather than individually. These pairs then combine to form energy-efficient locks at the interface. Subsequent annealing induces (nonglissile) motion of the locked dislocations in the interface, making the network spacing more regular. Unlike several recent reports, the later dislocation introduction does not appear to be strongly dependent upon the development of islanding (the transition from two dimensional to three dimensional growth).

10:20 AM BREAK

10:30 AM

Dislocations in Elliptical Holes and Zener-Stroh-Koehler Crack Solutions in Elliptical Coordinates: *Johannes Weertman*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 No. Campus Dr., Evanston, IL 60208-3108 USA

Hirth (Acta mater., v.47, pp. 1-4, 1999) derived stress fields around elliptical holes which contain dislocations in terms of derivatives of complex potential functions. When equilibrium and compatibility equations are expressed in curvilinear coordinates (see my paper in Mori symposium, Mat. Sci. Eng and my fracture book) more direct solutions can be found. The elliptical coordinate system is given by x+iy = a cosh(u+iv) and a is crack half length. The screw dislocation in a cavity solution is szu = 0, svz = s0bG. Here s0 = G/4p (or G/4(1-v)p) and aG = [2/(cosh2u-cos2v]1/2. For a glide edge dislocation the solution is the sum of the stress field szw = rotation pseudo-stress =-2s0baG2 sinhu cosv, suv = s0 ba3G4 cosv coshu sinh2u, suu =-2s0ba3G4 sinv cosh3u, svv =-2s0ba3G4 sinv coshu (cosh2u-cos2v-1), szz = v (suu+svv) and the stress field suv =-2 cosh2u0s0ba3G4 cosv sinhu, suu =-svv = 2 cosh2u0 s0 ba3G4 sinv coshu. Note: u0 = 0 for a crack and u0 = value of u at the cavity wall for the elliptical hole case. A similar solution exists for a climb dislocation. G, b, v have their usual meanings.

10:45 AM

Analysis of Anomalous Slip in Ta Single Crystals Using Optical, Atomic Force, and Orientation Imaging Microscopies: *James S. Stölken*[1]; Wayne E. King[1]; Adam J. Schwartz[1]; Mehdi Balooch[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci. Directorate, L-356, 7000 East Ave., Livermore, CA 94550 USA

Ta single crystals oriented along a single-slip orientation were deformed in compression at 300K and 77K. With the load axis of the sample fixed at [-4 8 19], the vertical faces of the specimen was selected to facilitate the slip trace analysis. The transverse specimen plane is chosen to maximize the projection of the [111] Burgers vector direction onto the plane normal. This is accomplished by choosing the longitudinal plane such that it contains the [111] direction. This gives the longitudinal plane as (-11 23-12) and the transverse plane on which the slip trace analysis is performed is as (533 257 4). Samples deformed at room temperature exhibited wavy glide whereas samples deformed at 77K exhibited anomalous slip. Orientation imaging microscopy has been used to probe lattice rotations occurring as a result of deformation. Optical and atomic force microscopies have also been applied to map the slip traces appearing on the sample surface. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

11:00 AM
Extrinsic Stacking Faults and Plastic Deformation of Intermetallics: *Man H. Yoo*[1]; [1]Oak Ridge National Laboratory, Metals and Cera. Div., P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

Specific energies of the intrinsic and extrinsic stacking faults in a fcc-based superlattice (SISF and SESF) are comparable, but the mobility of a composite Shockley (D-type Shockley) partial bounding a SESF is lower than that of a Shockley bounding a SISF for the reason given by Weertman and Weertman (1964). Two examples are given to point out the important role of synchro-Shockley partials in plastic deformation of intermetallics of the L1o and C15 structures. In explaining the so-called radiation-induced ductility (RID) of Ti-47%Al alloys, the low mobility of a D-type Shockley partial is one of the important factors for the interstitial Frank loops to remain unfaulted and for the heterogeneous nucleation kinetics of deformation twins. In AB2 Laves phase, such as NbCr2 and Hfv2, the intrinsic brittleness stems from the difficulty of this synchro-shear process, within the triple layer of a quadruple unit, which is the basic process for slip, twinning, and polytypic transformation. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

11:15 AM
Grain Boundary Cavitation during the High Cycle High Temperature Fatigue of Copper: *John G. Barker*[1]; Julia R. Weertman[2]; Jan Skov Pedersen[3]; [1]NIST, NIST Ctr. for Neutron Rsrch., 100 Bureau Dr. Stop 8562, Gaithersburg, MD 20899 USA; [2]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [3]Riso National Laboratory, Dept. of Solid State Physics, DK-4000, Roskilde, Denmark

High Temperature fully reversed fatigue tests on high purity copper samples were made as a function of stress amplitude. A mean cavity size was determined from the total cavity volume fraction (Vv) determined from precision density measurements (PDM) and the total cavity surface area (Sv) determined from small angle neutron scattering (SANS) measurements. Under the range of stress amplitudes tested, the plastic strain rate (de/dt) was found to cover three orders of magnitude. The cavity growth rates correlate well to the measured plastic strain by the relation dVp/dt = Ae^1/2. This surprising result will be discussed as it relates to current theoretical understanding of cavity growth during creep, low-cycle and high-cycle fatigue at elevated temperatures.

11:30 AM
Accelerated Shrinkage of Creep Cavities in Dispersion-Strengthened Aluminum by Thermal Cycling: *Christopher Schuh*[1]; B. Q. Han[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Rm. 2036, Evanston, IL 60208 USA

Although dispersion-strengthened metals exhibit much superior creep resistance than their respective parent metals, they fail at much lower total strains due to the development and subsequent linkage of creep cavities. It is also known that creep rupture times of various materials can be extended by intermediate annealing, with or without external hydrostatic pressure, to close creep cavities. We report here experimental data of creep cavity shrinkage for dispersion-strengthened-cast aluminum with about 23% Al_2O_3 dispersoids, annealed isothermally or subjected to thermal cycling without applied stress. We demonstrate that thermal cycling increases the rate of cavity shrinkage relative to isothermal annealing, allowing for recovery of full theoretical density in a shorter time. We attribute this effect to the presence of internal stresses produced by thermal expansion mismatch between matrix and dispersoids. This mechanism is examined in light of existing densification models, and implications for thermal cycling creep and internal stress superplasticity are discussed.

11:45 AM
On Cavitation Process in Metals under Creep-Fatigue Conditions: *Raj Mohan*[1]; [1]Battelle, Eng. Mech. and Mats., 505 King Ave., Columbus, OH 43201 USA

Failure in metals exposed to creep-fatigue conditions predominantly occurs by nucleation and growth of voids along grain boundaries and by eventual coalescence with neighboring voids, thus resulting in intergranular fracture. Over the past two decades, Weertman and her co-workers (Saguesa and Weertman, 1978; Weertman, 1979; Baker and Weertman, 1990) have made seminal contributions toward the mechanistic understanding of this phenomenon. In this paper, we present both experimental and analytical results related to cavitation processes in metals under high as well as room temperature conditions. Interestingly, room temperature creep occurs in Ti-6Al-4V even under high cycle fatigue conditions when the mean stress is near static yield of the material and at high R ratios. Detailed microscopic evidence the evolution of creep deformation and damage in Ti-6Al-4V during high cycle fatigue loading is presented. It is shown that crystallographic cracking within  grains, which appear to be the precursor of microvoids, occurs during very early stages of deformation. In addition, detailed numerical calculations are performed to understand the role of nonlinear effects of material and geometry, material rate-dependence and loading frequency on damage evolution. We also extend our numerical calculations of examine cavity growth process under balanced strain cycling conditions. The results indicate that void growth can occur, consistent with experimental observations under balanced cycling loading conditions provided nonlinear shape changes are taken into account. In addition, the analyses show that the cavity growth rate is constant under balanced cyclic loading conditions. This observation is in agreement with the experimental findings of Baker and Weertman (1990).

Defects in Solidification Processing: Defects in Solidification Processing II

Sponsored by: Materials Processing and Manufacturing Division, Solidification Committee
Program Organizers: Linda L. Rishel, Carnegie Mellon University, Materials Science & Engineering Department, Pittsburgh, PA 15213 USA; Ralph Napolitano, Iowa State University, Ames, IA 50010 USA; Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

Monday PM	Room: Salon F&G
November 1, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Linda L. Rishel, Carnegie Mellon University, Mats. Eng. and Sci. Dept., Pittsburgh, PA 15213 USA; Mark Palmer, Virginia Commonwealth University, Richmond, VA USA

2:00 PM Opening Remarks

2:05 PM
Hydrogen Evolution During Directional Solidification and Its Effect on Porosity Formation in Aluminum Alloys: *Qingyou Han*[1]; Srinath Viswanathan[1]; [1]Oak Ridge National Laboratory, Met. and Cera. Div., P.O. Box 2008, Oak Ridge, TN 37831-6083 USA

Most models for predicting microporosity formation in aluminum alloy castings use the lever rule for tracking hydrogen concentration in the liquid alloy. However, the hydrogen concentration predicted by the lever rule is typically too low to satisfy the threshold concentration for pore nucleation based on classical nucleation and growth theory. As a result, important features of microporosity such as pore size and pore spacing cannot be treated adequately. In this paper, the hydrogen redistribution and diffusion during direc-

tional solidification of an Al-4.5%Cu alloy is calculated. The calculations indicate that without pore nucleation, the hydrogen concentration ahead of the eutectic front can be two orders of magnitude higher than the maximum hydrogen concentration predicted using the lever rule, allowing classical nucleation and growth theory to be applied to microporosity formation during solidification. The model is used to propose a wave-like distribution of pores in castings.

2:30 PM

Porosity Formation in Directionally Solidified Mar-M247 Castings: Theoretical Model: *Deming Wang*[1]; Harry S. Whitesell[2]; Ruel A. Overfelt[2]; [1]Auburn University, Space Power Instit., 231 Leach Ctr., Auburn, AL 36849-5320 USA; [2]Auburn University, Mech. Eng., 201 Ross Hall, Auburn, AL 36849-5341 USA

Increases in microporosity have often been attributed to large pressure drops through the porous mushy zone of solidifying castings. If the available molten metal head is insufficient to compensate for the pressure drop, then porosity would be expected. The pressure drop is believed to influence both the nucleation event and subsequent growth of pores. However, an increasingly tortuous and low permeability mushy zone will also tend to isolate pores and prevent their further growth, thus limiting the total observed porosity. Since the microstructural length scales vary with solidification velocity and imposed thermal gradients, there may be process conditions where both nucleation and growth of interdendritic pores are enhanced. In this paper, the development of microporosity in the interdendritic channels of a directionally-solidified alloy is modeled using porous media flow mechanics (i.e., Darcy's law) coupled with pore isolation concepts. The simulation results predict that the observed porosity is a complex function of solidification velocity, thermal gradient, metallostatic head and icrostructural length scales. The tortuosity of the interdendritic channels is estimated by comparing the theoretical predictions and the experimentally measured icroporosity. Finally, the paper describes the solidification conditions responsible for the occurrence of microporosity in directionally solidified castings of Mar-M247 alloy.

2:55 PM

Cellular Automata for Prediction of Porosity During Solidification: *Robert C. Atwood*[1]; Peter D. Lee[1]; [1]Imperial College of Science, Technology and Medicine, Materials, Prince Consort Rd., London SW7 2BP UK

A continuous-valued cellular automaton model has been developed for the prediction of solidification microstructures. This model is able to emulate a columnar-to-equiaxed transition for some applied conditions, and has been extended to include the prediction of defect population. The effect of segregation and diffusion of dissolved hydrogen during grain growth is simulated, and the effect of impingement of solid grains upon growing pores is accounted for in this simulation. This cellular automaton technique is unique in that it allows the interaction of the developing microstructure and the pore to be explicitly handled. The size and spatial distributions of the pores is predicted, in tandem with the prediction of the growth of the solid phase grain structure. The results of this model are compared with experimental microstructures and in situ radiographic observations of hydrogen pore growth. The sensitivity of the model is discussed with regards to a range of input parameters, including: the nucleation models for both the solid and gaseous phases; the material properties; and the thermal conditions.

3:20 PM Break

3:35 PM

Application of Computer Modeling in Prediction of Porosity Formation in Aluminum Alloy Castings: *Deliang Zhang*[1]; Ming Lun Lang[1]; Wayne McConnel[2]; [1]The University of Waikato, Dept. of Tech., Private Bag 3105, Hamilton, New Zealand; [2]K. H. McConnel Limited, Hamilton, New Zealand

One of the major functions of computer simulation of alloy solidification during casting is to predict porosity formation. Almost all the commercial software packages boast to possess the ability to make this prediction with high precision. However, this seemly simple function is actually a very complex task. There are several important issues which each software package must adequately address before it can have the capacity of predicting the location and size of pores accurately. One of these issues is how the solidification behaviour of each different alloy can be precisely described in the software. This paper discusses how the ways of handling these issues affect the outcomes of simulations by using a commercial software package, AFS Solidification System 3D, as a basic reference. The focus is the effect of description of solid fraction as a function of temperature for different aluminum alloys such as 401 and A356 alloys on the prediction of shrinkage porosity formation in a commercial shape casting. Several techniques are used in establishing the relationship between the solid fraction and temperature, and they include a simple assumption of linear function, the use of Scheil equation, and direct experimental measurements by using thermal analysis instrument.

4:00 PM

Prediction of Microporosity in Aluminum Alloy Castings: Adrian S. Sabau[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Met. and Cera. Div., Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

Heat and fluid flow analyses are applied to the prediction of microporosity in aluminum alloy castings and correlated with experimental results. The computer model for microposity prediction takes into account alloy composition, alloy microstructure, the initial hydrogen content of the liquid alloy, and the resistance to fluid flow to feed shrinkage. The results will be validated by comparison to experimental results from laboratory and production castings.

4:25 PM

Porosity Formation in Directionally Solidified Mar-M247 Castings: Experimental Characterization: *Harry S. Whitesell*[1]; Ruel (Tony) A. Overfelt[1]; [1]Auburn University, Mats. Rsrch. and Edu. Ctr., 201 Ross Hall, Auburn, AL 36849 USA

The solidification microstructure is critical in determining the amount and distribution of porosity that develops during the freezing of castings. However, microstructural effects are often characterized only by the corresponding permeability or tortuosity of the mushy zone. As the microstructural length scales (primary and secondary dendrite arm spacings) decrease, (1) nucleated pores would be increasingly isolated, and (2) the mushy zone permeability would be expected to decrease. Although the first effect would tend to decrease the observed porosity, porous media flow considerations indicate that the second effect would tend to increase the porosity. To better understand these competitive mechanisms, a series of carefully controlled directional solidification experiments were performed on bars of nickel-base superalloy, Mar-M247. Samples were produced with constant dendrite arm spacings throughout an extended length of each cast bar. The axial thermal gradient and withdrawal velocity imposed on each casting were varied between castings to produce a range of microstructures from equiaxed dendritic to near cellular dendritic. Each casting's microstructural features (PDAS, SDAS, carbides, % porosity) have been measured and correlated with the imposed process parameters and compared to similar data from the literature. The development of porosity in these castings is shown to be dependent upon the process parameters and cast microstructures.

4:50 PM

Segregation in Uranium Alloys: *Deniece R. Korzekwa*[1]; [1]Los Alamos National Laboratory, MST-6, P.O. Box 1663, MS G770, Los Alamos, NM 87544 USA

Segregation in uranium-niobium alloys is a defect that affects not only the casting but can become more pronounced with further processing. This segregation can result in large variations in the

properties of part made from this alloy. The commercial casting code FLOW-3D is being used to simulate macrosegregation during solidification. Experiments with uranium-niobium were performed to validate the code and the results compared to the computer simulations.

Dynamic Behavior of Composites: MMC's

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Structural Materials Committee
Program Organizers: Rick Hall, University of Delaware, Department of Mechanical Engineering, Newark, DE 19716 USA; Rusty Gray, Los Alamos National Laboratory, Dynamic Properties, Los Alamos, NM 87545-0001 USA; Ken S. Vecchio, University of California, Department of Ames, San Diego, CA 92093 USA

Monday PM
November 1, 1999

Room: Salon D&E
Location: Omni Netherland Plaza Hotel

Session Chairs: Ian W. Hall, University of Delaware, Dept. of Mech. Eng., Newark, DE 19716 USA; George T. Gray III, Los Alamos National Laboratory, MST-8, Los Alamos, NM USA

2:00 PM
Strain Rate Effects on the Deformation and Fracture of a Continuous Fiber Reinforced MMC: *Carl M. Cady*[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87544 USA

The mechanical properties of an aluminum matrix composite unidirectionally reinforced with Al2O3 fibers has been measured and characterized in longitudinal and transverse compression. The influence of strain rate and fiber orientation on the deformation and fracture response will be presented. The stress-strain response of this composite was found to vary substantially as a function of loading orientation; the quasi-static yield changing from nominally 200 MPa transverse to the fibers to ~1.7 GPa parallel to the fibers under ideal conditions. The main failure mechanism has been identified to be plastic kinking, although an upper bound seems to be attained when the fibers reach their compressive strength. The experimental results are consistent with a plastic kinking model in strain hardening composites. The failure response of the composite transverse to the fibers, under uniaxial stress (quasi-static and dynamic) displays a protracted but substantial load drop after yield followed by continued degradation in load carrying capacity. Lack of ideal parallel fiber construction leads to systematic buckling failure of the alumina fibers through the sample under uniaxial loading.

2:30 PM
Dynamic Crushing of Aluminum Foams and Composites: *Ian "Rick" W. Hall*[1]; Ozgenc Ebil[2]; [1]University of Delaware, Dept. of Mech. Eng., Spencer Lab., Newark, DE 19716-3104 USA; [2]University of Delaware, Dept. of Mats. Sci. and Eng., Spencer Lab., Newark, DE 19716-3106 USA

Dynamic Crushing of Aluminum Foams and Composites Aluminum foams are already in use in applications where good crashworthiness is required. The advantage of foam is that it can confer a high degree of specific energy absorption and can be prepared in a wide variety of shapes. In particular, it can be used to fill hollow sections, yielding improved rigidity as well as improved energy absorption. The present work investigates the effects of section geometry and foam parameters on the crush resistance of filled and unfilled aluminum tubes. Narrow wall tubes have been filled with aluminum alloy and metal matrix foams and subjected to crushing tests from quasi-static (10e-3/s) to high strain rates (2x10e3/s), the latter using the Split Hopkinson Pressure Bar. The paper considers the additive effects of a) filling the tubes with foam and b) using higher strength metal matrix foam, and also considers the usefulness of the SHPB technique for investigations of such low impedance systems.

3:00 PM
The Effect of the Strain-Rate on the Mechanical Behaviour of Ti-6Al-4V/SiC Composites: *Javier LLorca*[1]; Francisco Gálvez[1]; Carlos González[1]; Pedro Poza[1]; [1]Polytechnic University of Madrid, Dept. of Mats. Sci., E. T. S. de Ingenieros de Caminos, Madrid 28040 Spain

Titanium alloys reinforced with long fibres are potential candidates for aeroengine applications because of their large specific stiffness and strength as well as their creep resistance. Impact of fragments is not unlikely in turbine components and it is thus important to study the mechanical behaviour of these composites at high strain rates. To this end, the tensile properties of a Ti-6Al-4V alloy unidirectionally reinforced with a 35 volume % SiC fibres were measured at different strain rates in the range 10-3 s-1 to 400 s-1 using a servo-hydraulic testing machine and a Hopkinson split bar. Extensometers and strain-gauges were used to measure the specimen strain in order to obtain the tensile stress-strain curve. The broken samples were examined in the longitudinal and transverse directions in a scanning electron microscopy to ascertain the effects of the strain rate on the different failure mechanisms. Finally, the in situ fiber strength was determined from tensile tests of SiC fibers extracted from the composite. It was found that the composite yield strength increased with the strain rate whereas the ductility followed the opposite trend and the ultimate tensile remained constant within the experimental scatter. The fractographic observations indicated that the specimen failure was induced by the rapid damage localization around broken fibers, regardless of the strain rate. The fiber cracks were stopped, however, at the fiber C coating in the slow strain rate tests, while they propagated through this coating up to the C/matrix interface in the tests at high strain rates. Numerical simulations demonstrated that this phenomenon increased the load carried by the neighbour fibers around a broken one and thus was responsible for the reduced composite ductility at high strain rates, whereas the strain rate hardening of the Ti matrix led to the increase in yield strength.

3:30 PM
Viscoplastic Response of an A359/SiCp MMC under High-Strain Compression: *K. T. Ramesh*[1]; Yulong Li[1]; Ernest S.C. Chin[2]; [1]Johns Hopkins University, Mech.Eng. Dept., 200 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Army Research Laboratory, Weapons & Mats. Rsrch. Directorate, Aberdeen Proving Ground, MD 21005 USA

The viscoplastic behavior of an A359/SiCp metal-matrix composite and of the corresponding A359 cast aluminum alloy have been determined over a wide range of strain rates (10-4 to 105 s-1) using several different experimental techniques: a servohydraulic test machine, the compression Kolsky bar and pressure-shear plate impact. Both the A359 matrix alloy and the A359/SiCp composite show rate-dependence of the flow stress in compression, with rate-dependencies that increase with increasing strain rate. The unreinforced A359 alloy shows strain hardening that is essentially independent of the strain rate, similar in most respects to the behavior of wrought aluminum alloys such as 6061. The A359/SiCp composite shows rate-dependence similar to that of the unreinforced alloy, but also shows significantly less strain hardening than does the matrix alloy. This is contrary to the predictions of computational models that assume perfect particle-matrix interfaces and undamaged particles. Microscopic observations show that the reduction in strain hardening is a result of progressive particle fracture during these compressive deformations. Using the experimental data on the unreinforced A359 aluminum alloy as the input data for the matrix behavior, and accounting for the particle fracture using statistical analysis, an analytical model developed recently by the authors is used to estimate the mechanical re-

sponse of the composite over the whole range of strain rates. It is shown that these new predictions are a good agreement with the experimental results.

4:00 PM Break

4:10 PM
The Dynamic Evolution of Interfacial Debonding and its Application in Ceramic Matrix Composites: *Yongjian Sun*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The dynamic evolution of interfacial debond in response to the externally applied load is observed in a transparent ceramic matrix composite for the first time. The fiber-matrix crack interaction discloses interesting relationships among crack opening, crack length, and debond length in composites. With the assistance of fracture mechanics models for composites, these relationships are applied for the determination of the bridging stress in fibers and the fracture resistance curves for the composites. This application of interfacial debond gives a good demonstration of the micromechanics of fiber toughening in composites.

4:40 PM
Dynamic Behavior of Metallic-Intermetallic Laminate Composites: *David Harach*[1]; Kenneth S. Vecchio[1]; George T. Gray[2]; [1]University of California, San Diego, Mats. Sci. Group, Dept. of Mech.& Aero. Eng., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA; [2]Los Alamos National Laboratory, Group MST-8, Structure/Property Rels., MS G755, Los Alamos, NM 87545 USA

Metal-intermetallic laminate (MIL) composites based on Ti-Al3Ti can be designed for structural use to optimize the unique properties and benefits of the constituent components, resulting in materials which have the high strength and stiffness of the intermetallic phase and the high toughness of the metal. There is excellent microstructural control and microstructural variability as layer thickness can be chosen with great precision, and the foil stacking sequence and foil material can be varied within the thickness to yield graded structures and microstructures optimized for specific applications. The mechanical response of these novel MIL composites has been examined as a function of microstructure, loading direction and loading rate. The effect of loading rate was examined utilizing a momentum-trapped compression Hopkinson bar. A new dynamic three-point bend test with momentum trapping (single stress pulse recovery technique) has been developed to characterize the mechanical behavior of these composites under dynamic bending conditions. The force-displacement response in three-point bending, and stress-strain response in dynamic compression differ considerably from quasi-static conditions. In three-point bend tests, the dynamic force response is two to three times that under quasi-static conditions, while dynamic compression results in 100-200 MPa increases in yield stress over quasi-static tests. The volume fraction of phases and the orientation of the layers affect the mechanical behavior at both low and high strain rates. Taylor cylinder impact testing has previously been utilized to probe both the deformation response of metals and alloys in the presence of gradients of stress, strain, and strain-rate and as a means to validate constitutive models. This axi-symmetric integrated test provides a readily conducted experimental method by which to examine the large-strain high-strain-rate mechanical behavior of materials while simultaneously exhibiting a large degree of sensitivity to evaluating the effectiveness and correct "physics" implementation in constitutive models. Taylor tests have been conducted on these MIL composites as a function of orientation to evaluate the deformation and damage evolution response of the composite to a uniaxial stress impact.

5:10 PM
Quasi-Static and Dynamic Mechanical Behavior of TiC-Reinforced Ti-6Al-4V: *A. J. Wagoner*[1]; C. L. Briant[1]; K. S. Kumar[1]; C. Bull[1]; [1]Brown University, Dept. of Eng., 182 Hope St., Providence, RI 02912 USA

Ti-6Al-4V is used in many ballistic applications because of its high specific strength. The addition of ceramic particulate reinforcements further augments the strength-to-weight ratio. The mechanical properties of the TiC/Ti-6Al-4V system have been studied as a function of strain rate and volume fraction of reinforcement. Both hot pressed and forged powder samples of 5, 10, and 20%TiC are compared in compression at strain rates of 0.1-1200/s. Quasi-static tests were conducted on a servo-hydraulic Instron machine and dynamic tests on a Split Hopkinson Bar. Results show a significant increase in room temperature, quasi-static yield stress with volume fraction of TiC. At a strain rate of 1.0/s, the yield stress increases from 1200MPa in the monolithic material to 1675MPa in the 20%TiC samples. The flow stress, at a given volume fraction of reinforcement, also increases with strain rate. Dominant mechanisms controlling the plastic response as a function of strain, strain rate, and volume fraction of reinforcement will be identified with the aid of optical and electron microscopy. Support for the research is provided by the Brown University NSF-sponsored MRSEC.

General Abstract Sessions: Advances in Steel Technology

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Monday PM
November 1, 1999
Room: Caprice 2&3
Location: Omni Netherland Plaza Hotel

Session Chair: George Spanos, Naval Research Laboratory, Code 6324, 4555 Overlook Ave., S.W. Washington, DC 20375-5000

2:00 PM
Interphase Boundary Carbides and Ferrite Growth Ledges in F-C-X Alloys: *Gary J. Shiflet*[1]; [1]University of Virginia, Mats. Sci. and Eng., School of Eng. and Appl. Sci., Thornton Hall, Charlottesville, VA 22903 USA

There has been on ongoing debate concerning the growth mechanism of proeutectoid ferrite and pearlitic ferrite in Fe-C-X alloys. The growth interphase boundaries are described in terms ranging from disordered to partially coherent, invoking either the quasi-ledge or ledge mechanism to advance the boundary. This talk will focus on a comparison between interphase boundary precipitate habit planes and the angle they make with the ferrite/austenite growth interface. Modeling will show that the single carbide variant selected is that most parallel to the ferrite growth interphase boundary plane. It will further be demonstrated that this is a direct consequence of nucleation theory and can best be explained by a semi-coherent interphase boundary and growth ledges. This work is supported by NSF-DMR.

2:20 PM
Correlation Between the Crystallography and 3-D Morphology of Proeutectoid Widmanstatten Cementite Precipitates: *M. A. Mangan*[1]; M. V. Kral[2]; G. Spanos[1]; [1]Naval Research Laboratory, Phys. Metallu. Brnch. Code 6324, 4555 Overlook Ave. SW, Washington, DC 20375-5000 USA; [2]University of Canterbury, Dept. of Mech. Eng., P.O. Box 4800, Christchurch, New Zealand

Electron backscatter diffraction (EBSD) was used in conjunction with deep etching to examine the correlation between the cementite: austenite orientation relationships (ORs) and the three-dimensional (3-D) morphology of cementite precipitates in an Fe-

1.34wt%C-13.1wt%Mn steel. ORs between more than 200 Widmanstatten cementite precipitates and the surrounding austenite matrix were determined to be either the well-known Pitsch OR or the Farooque-Edmonds OR. Scanning electron microscopy of the same specimens after deep etching was used to determine the 3-D morphology of each of the precipitates whose OR was determined. These precipitates could be identified as either plates (aspect ratio of length/width close to 1) or laths (aspect ratio greater than 5). Results show Widmanstatten plates consistently exhibit the Pitsch OR and laths have the Farooque-Edmonds OR, indicating that the precipitate morphologies are dictated by their orientation relationships.

2:40 PM

Effect of Cu on the Hot Workability of 304 Austenitic Stainless Steel: *Jeom Yong Choi*[1]; Won Jin[1]; [1]POSCO, Stainless Steel Rsrch. Grp., Pohang P.O. Box 36, 1 Koedong-dong Nam-ku, Pohang, Kyungbuk 790-785 Korea

The mechanism of embrittlement by Cu in steels has been extensively studied. Generally, the addition of Cu tends to make the slab more sensitive to the surface cracking during hot rolling and continuous casting. In addition, the segregation of residual element and grain boundary precipitation are considered as the important factors influencing the embrittlement during hot rolling. The characteristics of embrittlement discussed above are common in carbon steels which have lower solubility of Cu than stainless steels. At present, the available information regarding ductility loss due to Cu addition in stainless steel is very limited. At the present investigation, an attempt has been made to elucidate the cause of ductility loss in stainless steel containing Cu and to examine the effects of cooling rate in promoting hot cracking. Materials used in the present investigation were austenitic stainless steels containing Cu from 1.5wt% to 5.0wt%. Hot deformation test was conducted on the Gleeble-1500 with cross head speed of 3mm/sec. The test temperature ranged from 1173K to 1523K. In addition, the effect of cooling rate on hot ductility was investigated. Hot ductility was evaluated in terms of reduction of area in fractured tensile specimens. Microstructure and fracture surface were examined by optical microscope and scanning electron microscope(SEM). Also, Auger in-situ fracture analysis was performed with notched specimens which were machined from hot tensile prestrained specimens. Reduction of area (RA) linearly decreased with increasing Cu content at deformation temperatures above 1373K. But, below 1373K, the effect of Cu on RA was drastically changed at about 3.0% Cu. At lower Cu contents, RA decreased with Cu content in the same manner with that observed above 1373K. However, as Cu contents exceeded about 3.0%, RA decreased rapidly with increasing Cu contents. RA decreased with increasing cooling rate in the range of 278K/sec~ 373K/sec. At deformation temperature 1323K, RA of 4.8% Cu containing steel decreased more rapidly compared with that of 3.9% Cu containing steel. Fracture mode changed from ductile to intergranular as cooling rate increased. It would be caused by the segregation of Cu and S at the austenite grain boundary.

3:00 PM

Fabrication and Characterization of Surface-Modified Steels with Higher Resistance to Rolling Contact Fatigue: *Byung-Young Choi*[1]; [1]Chonbuk National University, School of Adv. Mats. Eng., 664-1, 1ka, Dukjin-dong, Chonju 561-756 Korea

Surface-modified Steels were fabricated by vacuum induction melting and double forging followed by induction hardening. Their characterization of microstructure and its effect on rolling contact fatigue were conducted under a maximum hertzian contact stress of 492Kg/mm2 with a rotating speed of 8000 rev/min and a constant supply of lubricant. It was found that repeated plastic deformations during rolling contact fatigue that resulted in microstructural degradation in the subsurface below the raceway of rolling contact fatigued specimens. The average half values breadth of X-ray diffraction pattern and microhardness were measured and discussed in terms of microstructural degradation of the specimens with higher resistance to rolling contact fatigue.

3:20 PM

Effect of Nb, V and Ti Additions on the Yield Strength of Hot Direct Rolled Thin Slab Cast Low Carbon Steel: *J. S. Park*[1]; M. Ajmal[2]; R. Priestner[3]; [1]Inchon Iron and Steel Company Limited, Manchester Matls. Sci. Cntr., Inchon Korea; [2]The Engineering University of Lahore, Lahore, Pakistan; [3]The University of Manchester, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK

Ingots of low carbon steels with additions of Nb, V, and Ti were made in a manner that simulated thin slab casting, and were hot direct rolled. Mn and Si contents were kept constant at 1.4 wt% and 0.25 wt% respectively. C contents were varied from 0.05-0.14 wt% and Nb from 0.02-0.05 wt% in Nb-only steels. V up to 0.034 wt% and Ti at 0.007 wt% were added to 0.07 wt%C, 0.04 wt%Nb steels. Segregation of Nb occurred during solidification, which led to precipitation of eutectic NbCN for C contents in excess of 0.09 wt%, and therefore, to the effective loss of a fraction of the Nb addition. The precipitated Nb fraction approached 0.5 at the higher Nb and C contents. The loss of Nb from solid solution before rolling limited the tensile yield strength of the rolled product. The further additions of V alone and V and Ti together had no effect on the yield strength.

3:40 PM Break

3:50 PM

Hot Ductility of 310S Stainless Steel: *Yun Yong Lee*[1]; Soo Chan Lee[1]; Mats Liljas[2]; [1]POSCO, Stainless Steel Rsrch. Grp., Pohang P.O. Box 36, 1 Koedong-dong Nam-ku, Pohang 790-785 Korea; [2]Avesta-Sheffield, Rsrch. and Dev., Sweden

The effect of sulphur and titanium on the hot ductility of 310S stainless steel has been studied by high temperature tensile test. The hot ductility of 310S was drastically decreased with decreasing temperatures and with increasing sulphur contents. The sulphides present in as-cast state were redissolved within solubility limits under the isothermal reheating conditions, and then reprecipitated at grain boundaries during cooling under the hot rolling conditions. The hot ductility was mainly governed by the reprecipitation of low melting sulphides such as CuFeS2, Cu5FeS4 or Cu2S. The quantity and the composition of reprecipitated sulphides have been changed with the cooling rates and the holding time at deformation temperatures. The deterimental effect of sulphur on the hot ductility of 310S can be minimized by increasing manganese content in steel. The addition of small amount of titanium improved hot ductility at low temperature region by not only restraining grain growth during reheating, but also enhancing recrystallization due to TiN precipitation. The addition of titanium enhanced reprecipitation of low melting sulphides by forming randomly distributed fine TiN precipitates. In spite of enhancing reprecipitation the dilution of low melting sulphides into large MnS in vicinity easily occurred because of random dispersion and so the reduction in ductility was prevented. Without Ti addition, the non-equilibrium supersaturation of reprecipitated sulphur at specific grain boundaries such as recrystallized/non-recrystallized boundaries could be facilitated.

4:10 PM

Nucleation and Growth Process of Sticking Particle in Ferritic Stainless Steel: *Won Jin*[1]; Jeom Yong Choi[1]; [1]POSCO, Stainless Steel Rsrch. Grp., Pohang P.O. Box 36, 1 Koedong-dong Nam-ku, Pohang 790-785 Korea

The sticking phenomenon occurs frequently during the hot rolling of ferritic stainless steels, causing surface defects on the hot strip and scoring on the roll surface. In the present study, the nucleation and growth process of sticking particle was determined by mutual

interaction of roll and rolling material. The sticking simulation was performed under the various conditions of roll surface and thickness of scale layer formed on ferritic stainless steels by using a two disk type hot rolling simulator. Four ferritic stainless steels having various Cr content from 11 to 26% were used as rolling material and the high speed steel roll (HSS roll) was used as roll material. The sticking of the bare metal, exposed by scale breakaway during hot rolling, to the roll surface was strongly dependent on the surface roughness of HSS roll and the oxidation resistance of the ferritic stainless steels. The lower oxidation resistance of steels and the higher surface roughness of HSS roll were more sensitive to sticking occurrence. It was also elucidated that the initial sticking particles were nucleated at the scratches formed on the roll surface and were served as the sticking growth sites. As rolling proceeded, the sticking particles grew by the process that the previous sticking particles provided the sticking growth sites.

4:30 PM
Forged and Direct Quenched Ti-Nb-V High Strength Low Alloy Steels: *M. Hadji*[1]; Y. Sikaddour[1]; M. Temar[1]; S. Zidelmel[1]; [1]University of Blida, Instit. of Mech. Eng., P.O. Box 270, Route de Soumaa, Algeria

The influence of composition and microstructure on the mechanical properties is investigated. Three Ti-Nb-V HSLA steels (0.15%C-1.5%Mn-0.18%Si-0.008%Ti; 0.15%C-1.5%Mn-0.18%Si-0.16%Ti-0.03%Nb-0.06%V; 0.15%C-1.5%Mn-0.18%Si-0.25%Ti-0.09%-Nb-0.06%V) were produced. These steels were melted in an open induction furnace, and cast into ingots. The ingots were hot forged, with 80% reduction from a different hot forging temperature and cooled at several rates, in order to obtain different microstructures. Test results revealed that for a certain amount of Ti, the mechanical properties of forged Ti HSLA steels will be improved significantly by direct quenching. The effects of direct quenching from different forging temperatures on the microstructures and the mechanical properties will be presented.

General Abstract Sessions: Environmental and Chemical Degradation of Materials

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Monday PM, November 1, 1999
Room: Rookwood
Location: Omni Netherland Plaza Hotel

Session Chairs: James C. Earthman, University of California, Dept. of Chem. & Biochem. Eng., Irvine, CA 92697-2575 USA; Chengheng Robert Kao, National Center University, Dept. of Chem. Eng., Chung-Li, Taiwan

2:00 PM
Environmentally Assisted Sustained Load Crack Growth in PM Nickel-Based Superalloys: *Zhifan Frank Huang*[1]; Carl Hiroshi Iwashita[1]; Irene Chou[1]; *Robert P. Wei*[1]; [1]Lehigh University, Sinclair Lab., 325, 7 Asa Dr., Bethlehem, PA 18015 USA

To examine the influence of niobium (Nb) on sustained-load crack growth in oxygen, three powder metallurgy nickel-base superalloys, with nominal compositions similar to IN100, but with 0, 2.5 and 5 wt% of Nb, are used. These alloys are gamma prime strengthened and have comparable volume fractions of gamma prime precipitates. Sustained load crack growth experiments are conducted in high-purity oxygen and argon at temperatures 873, 923 and 973K. The environmental cracking sensitivity and activation energy for the 5% wt% Nb alloy are consistent with those of IN718, and support the previously identified role of Nb in enhancing crack growth. The susceptibility of the Nb-free alloy to oxygen, however, is much greater than expected. The results are discussed in terms of the chemical and microstructural contributions to the mechanism for environmental enhancement of crack growth. This research is supported by the Division of Materials Research, National Science Foundation under Grant No. DMR-9632994.

2:20 PM
Environmentally Enhanced Fatigue Crack Growth in PM Nickel-Based Superalloys: *Zhifan Frank Huang*[1]; Robert P. Wei[1]; [1]Lehigh University, Mech. Eng., 325, 7 Asa Dr., Bethlehem, PA 18015 USA

A study was carried out to examine the mechanical and mechanistic implications of hold time, at maximum load, on fatigue crack growth in two nickel-base powder metallurgy (PM) superalloys. The base composition of the alloys is nearly that of IN-100, with one containing no niobium and the other, 5 wt% niobium. Both alloys are strengthened by gamma prime precipitation. A trapezoidal wave form at a load ratio R = 0.1 was used, with 0.05s up and down ramps and hold-times of 0, 3, 10 and 30s. The 0s hold-time corresponded to a cyclic load frequency of 10 Hz. Crack growth experiments were conducted, under constant DK (stress intensity factor range) control, in high-purity argon and oxygen at 873, 923 and 973K. The crack growth response is discussed in terms of a superposition model that considers the cycle and time dependent contributions, as well as environmental contributions, and in terms of the microstructural variables. This research is supported by the Division of Materials Research, National Science Foundation under Grant No. DMR-9632994.

2:40 PM
Environmental Exposure Cracking Tests of 305, 410 Stainless Steels and Low Alloy Steel Bolts/Fasteners in Boiling Acidified Chloride Solutions: *Jose R. Oviedo*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Mats. Sci. and Eng., 10900 Euclid Ave. White Bldg., Cleveland, OH 44106 USA

Stress Corrosion Cracking (SCC) susceptibility of 305 austenitic stainless steel, 410 martensitic stainless steel Q&T at 500 and 975°F, and low alloy steel ASTM A-325 bolts/fasteners have been determined in acidified chloride containing solutions. The SCC tests were conducted using wedge/loaded Disk-Shape Compact Tension specimens (ASTM E-399) machined from the bolts/fasteners and immersed in boiling chloride solutions. The solutions were prepared to contain 25% NaCl in addition to S containing species. The pH in some tests was adjusted to pH = 2.0 via phosphoric acid additions in order to make the solution more aggressive. Some of the tests were accelerated via oxygen additions into the solution. Crack growth was monitored in order to determine the Threshold Stress Intensity for SCC (KISCC), as well as crack growth rate da/dt vs. K. under the conditions tested. Both fractography and metallography were characterized in order to analyze the crack growth mechanisms.

3:00 PM Break

3:10 PM
Sulfidation of Ti3Al Intermetallics: *Ramana G. Reddy*[1]; [1]University of Alabama, Dept. of Met. & Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

The Ti3Al intermetallics sulfidation under oxidizing conditions was investigated. The experiments were carried out at 950°C in 5%SO2 gas using TGA. The reaction products were analyzed using SEM, and X-ray diffraction. From the experimental results, the reaction rate constants were calculated. The calculated Ti3Al sulfidation rates were higher than the oxidation rates obtained under the similar experimental conditions. The present results were also compared with the other intermetallic systems.

3:30 PM
Steam Explosions of Single Drops of a Molten Ferrosilicon Alloy: *Lloyd S. Nelson*[1]; Paul W. Brooks[1]; Riccardo Bonazza[1]; Michael L. Corradini[1]; Kjetil Hildal[2]; Trond Bergstrom[3]; [1]University of Wisconsin, Dept. of Eng. Physics, Madison, WI 53706-1687 USA; [2]NTNU, Instit. of Metallu., N-7016 Norway; [3]SINTEF Materials Technology, N-7034 Norway

It is economically and environmentally advantageous to use water granulation to produce 10-to 20-mm granules of the 75 wt. % Si ferrosilicon alloy. In this process, drops of the molten alloy are sprayed into water and quenched. Unfortunately, the array of molten metal globules produced during water granulation sometimes becomes the "coarse premixture" that precedes a large and devastating steam explosion. To learn about these steam explosions, we have released single drops of the molten alloy into water. We found that the drops do not explode spontaneously but can be induced to explode by exposing them to pressure transients generated in the water with an encapsulated underwater impactor. We have investigated several parameters important to understanding the large scale explosions: threshold pressures for initiating the drop explosions, the water depths over which the drops can be triggered, pressure-volume energies released by the explosions of the drops and the onset of solidification of unexploded drops.

3:50 PM
Recycling of Metal Matrix Composites: *Yoshinori Nishida*[1]; Hiroaki Arima[2]; [1]National Industrial Research Institute of Nagoya, Composite Mats. Lab., Hirate-cho, Kita-ku, Nagoya 462-8510 Japan; [2]Daido Institute of Technology, Graduate Student, Takiharu, Minami-ku, Nagoya 457-8530 Japan

The amount of the MMC products have been increasing. MMCs, especially light MMCs such as aluminum alloy matrix composites are used to improve the efficiency of machines and to reduce CO2 gas release. Since the amount of the MMC products is small, the recycling and reclamation of MMCs has not been a serious problem. However, the recycling will become an important issue in the near future. In this study, the separation techniques of reinforcements from aluminum were developed. The separation techniques are classified into two routes. The first one is the chemical way, by which the interfacial energy between the reinforcement and the matrix metal is changed by some additives. As the additives, fluxes used for the melt treatments to remove inclusions in the melt are effective. The other one is the mechanical way, by which the interfacial energy necessary for the separation is given mechanically. Typical examples of both ways are introduced on alumina short fiber reinforced aluminum matrix composites and the separation mechanism is discussed.

4:10 PM
Novel Systems for Investigating Corrosion Processes under Simulated Service Water Conditions: *K. C. Trandem*[1]; Kh. M. Ismail[2]; P. J. Arps[3]; J. C. Earthman[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Mats. Sci., Irvine, CA 92697-2575 USA; [2]University of Cairo, Dept. of Chem., Cairo Egypt; [3]University of Nevada, Dept. of Chem. and Metallu. Eng., Reno, NV 89557-0136 USA

Field and laboratory "sidestream" systems have been developed to study both abiotic and microbiologically influenced corrosion under service water conditions that are representative of those in power generation facilities. The primary objectives of the present work are twofold: (1) to characterize the corrosion of different alloys under these conditions and (2) to investigate corrosion inhibition using biological methods. In addition to simulating service water conditions, the present side stream systems are capable of combined testing approaches involving microbiology, electrochemistry, and surface chemistry that are being used to provide insight into complex interactions between biofilms and metal surfaces. Corrosion test methods including electrochemical impedance spectroscopy, electrochemical noise analysis, and polarization techniques are being performed in situ for three alloys under three different flow rates. Scanning electron microscopy and energy dispersive x-ray spectroscopy results are also being obtained with tested specimens to further assess the nature of the corrosion processes.

Modeling the Performance of Structural Materials: Deformation, Damage, and Fracture

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712-1063 USA; Donald Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

Monday PM
November 1, 1999
Room: Salon H&I
Location: Omni Netherland Plaza Hotel

Session Chair: Eric M. Taleff, The University of Texas, Texas Materials Institute, Austin, TX 78712-1063 USA

2:00 PM
Microstructural Modeling of Toughened NiAl Composites: *W. O. Soboyejo*[1]; D. Zeng[2]; R. Wang[2]; M. Li[1]; N. Katsube[2]; [1]Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Applied Mechanics and Aviation, Dept. of Aerospace Eng., 150 Boyd Lab., 155 W. Woodruff Ave., Columbus, OH 43210 USA

This paper presents the results of a combines computational [1,3] and experimental [4,5] study of extrinsically toughened NiAl composites. The crack/microstructure interactions in transformation toughened and ductile layer toughened composites are simulated using finite element models of actual composite microstructures. The models include special hybrid elements that enable individual transforming particles to be represented by single elements. Finite element simulations of resistance-curve behavior in the toughened composites are also shown to be in close agreement with resistance-curves obtained from experiments.

2:20 PM
Influence of Test Temperature on Ductility, Deformation and Fracture Behavior of Aluminum Alloy 2524: *D. Kolar*[1]*;* T. S. Srivatsan[1]; P. Magnusen[2]; [1]University of Akron, Dept. of Mech. Eng., Akron, OH 44325-3903 USA; [2]Alcoa Technical Center, Aluminum Co. of America, 100 Tech. Dr., Alcoa Center, PA 15069 USA

An Al-Cu-Mg alloy (2524) was deformed to failure in the T351 microstructural condition in laboratory air environment at ambient and elevated temperatures. Strength was found to decrease with an increase in test temperature but with a concomitant improvement in ductility for both longitudinal and transverse orientations of the wrought alloy plate. No major change in macroscopic fracture mode was observed with direction of testing. Tensile fracture, on a microscopic scale, revealed features reminiscent of locally ductile and brittle mechanisms. The mechanisms and intrinsic micromechanisms governing tensile ductility and fracture processes will be highlighted and discussed in light of concurrent and mutually interactive influences of intrinsic microstructural effects, matrix deformation characteristics, test temperature and grain boundary failure.

2:40 PM
Behavioral Modeling of Particle Reinforced Metal Matrix Composites: *Yu-Lin Shen*[1]; Nikhilesh Chawla[2]; [1]University of New Mexico, Dept. of Mech. Eng., Albuquerque, NM 87131 USA; [2]Hoeganaes Corporation, Rsrch. and Dev. Div., Cinnaminson, NJ 08057 USA

Metal matrix composites (MMCs) offer several advantages over conventional monolithic alloys, including high strength-to-weight ratios and superior fatigue performance. This talk will describe the results of computational modeling of SiC particle reinforced Al composites, with the purpose of predicting the mechanical performance of the composite. It will be shown here that, with an appropriate construction of the model problem, the numerical modeling is capable of rationalizing a variety of experimental results. The cyclic stress-strain behavior of the Al/SiCp composite was simulated. The effects of reinforcement and matrix strength on stress enhancement in fatigue crack-initiating intermetallic inclusions in these composites will also be presented. Specifically, mechanical shielding of the inclusion particle due to the SiC reinforcement will be addressed. Finally, analysis of the response of the composite to macro/microindentation will be discussed. References: 1. Y.-L. Shen, M. Finot, A. Needleman and S. Suresh, Acta Metall. Mater., (1994) 42 77-97. 2. Y.-L. Shen, M. Finot, A. Needleman and S. Suresh, Acta Metall. Mater., (1995) 43 1701-1722. 3. N. Chawla, C. Andres, J.W. Jones, J.E. Allison, Effect of Reinforcement Volume Fraction and Particle Size on the Fatigue Behavior of SiC Particle Reinforced Al 2080 Matrix Composites, Metall. & Mater. Trans. A., (1998) 29A 2843. 4. N. Chawla, C. Andres, J.W. Jones, and J.E. Allison, Cyclic Stress-Strain Behavior of Discontinuously Reinforced Metal Matrix Composites, Scripta Mater., (1998) 38 1596. 5. N. Chawla, L.C. Davis, C. Andres, J.E. Allison, J.W. Jones, The Interactive Role of Inclusions and SiC Reinforcement on the High Cycle Fatigue Resistance of Particle Reinforced Metal Matrix Composites," Metall. & Mater. Trans. A., (1999) submitted.

3:00 PM

A Self Consistent Model for the Elasto-Plastic Behaviour of Particle-Reinforced Composites Including Damage: *Javier LLorca*[1]; Carlos González[1]; [1]Polytechnic University of Madrid, Dept. of Mats. Sci., E. T. S. de Ingenieros de Caminos, Madrid 28040 Spain

A model is developed to compute the mechanical behaviour of two-phase materials including the effects of damage or phase transformation. The material is represented by an interpenetrating network of randomly distributed spheres, which are assumed to behave as isotropic elasto-plastic solids. The incremental self-consistent method is used to compute the effective response of the material as well as the elastic stress redistribution due to damage or phase transformation. As an example, the model predictions are compared with experimental results for a 2618 Al alloy reinforced with 15 vol. % SiC particles, which presented damage by reinforcement fracture during deformation. The model predictions, in terms of the tensile stress-strain curve, the onset of plastic instability and the fraction of broken particles at failure were in good agreement with the experimental results.

3:20 PM Break

3:30 PM

Damage Accumulation and Predicting Failure of HSLA-100 Steel: *D. Chae*[1]; D. A. Koss[1]; [1]Pennsylvania State University, Dept. of Mats. Sci. & Eng., University Park, PA 16802 USA

Accurate predictions of the tensile failure of the high strength steels under the complex loading conditions requires an understanding of the effect of stress state on the damage accumulation process. This study examines the effect of stress triaxiality on damage accumulation and failure, using HSLA-100 steel as a model material. Tensile tests of axisymmetric circumferentially notched specimens have been performed in order to analyze quantitatively the damage evolution and the failure process. The primary source of damage was large voids (^{3} 10 mm^2 cross section area) which nucleate at low equivalent plastic strains. The initial strain-induced growth of these void occurs in a manner expected from the Rice and Tracey relationship for the growth of isolated voids [J. R. Rice and D. M. Tracey, J. Mech. Phys. Solids, 1969, vol. 17, pp201-217]. However, void growth increases rapidly near the failure strain, apparently as a result of void-void interactions. The damage evolution data thus suggest a transition from "slow" to "rapid" void growth at a critical void area fraction, which decreases with increasing stress triaxiality. Since specimen failure occurs soon after the void growth transition, the critical void volume fraction may be considered a failure criterion. Accordingly, we have predicted the failure strains of HSLA-100 steel as a function of stress state by employing the critical void area fraction criterion and fitting the "slow" void growth data to an equation of the Rice-Tracey form. The predicted and observed failure strain values are in good agreement.

3:50 PM

Prediction of Fracture Toughness Using Artificial Neural Networks: *W. J. Liu*[1]; W. R. Tyson[1]; S. Xu[1]; E. Essadiqi[1]; [1]CANMET, MTL, 568 Booth St., Ottawa K1A 0G1 Canada

Knowledge of fracture toughness of materials in terms of plane-strain toughness (KIC), crack tip opening displacement (CTOD) or J-integral (J) is essential for safe design and operation of structures subject to failure due to crack growth. However, such data are generally expensive and difficult to measure. For this reason, many researchers have attempted to correlate a material's fracture toughness with other more easily measured mechanical and physical properties, such as impact energy, tensile properties, and microstructure. Unfortunately, a general and reliable approach for prediction of fracture toughness is still not realized so far. In the present study, a new computer model, which is capable of predicting J values of a material from a set of conventional properties, has been developed based on the concept of the artificial neural network (ANN). The model has been applied to predict the fracture toughness of a series of linepipe steels. The results of the modelling will be discussed.

4:10 PM

Models to Calculation of the Structural Steels Properties: *Bertold B. Vinokur*[1]; Oleg G. Kasatkin[2]; Stanislav E. Kondratyuk[3]; [1]Technical Science, 3901 Conshohocken Ave., Apt. E-21, Philadelphia, PA 10131 USA; [2]Technical Science, 1/22 Filatova St., Apt. 62, Kiev 252042 Ukraine; [3]Technical Science, 3 Leonida Pervomayskogo St., Apt. 15, Kiev 252023 Ukraine

Long-term dream of most metal-scientific workers was creation of dependence of alloys mechanical properties on the chemical composition and heat treatment. This was fulfilled by using of one of mathematical statistics method namely as method of multiple regression analysis with step-by-step elimination of insignificance regressors. It was collected world literature data for 35 years about the low-carbon structural steels quenched from intercritical a-> g temperature range and above A_{c3} point and subsequent low-temperature tempering. The steels have been alloyed with practically all used alloying elements. The created interpolation models was obtained for ultimate tensile strength limit s_u. Tensile yield strength limit s_y, elongation d, reduction of area Y, and impact toughness by Menage a_1 with the notch radius of 1 mm. The created models allow to obtaining influence of each property of each of alloying element and carbon separately and their combinations after quenching from different temperatures. It was using no absolute heating temperature but temperature that was relatively to the temperature of A_{c3} point. A_{c3} point temperatures defines to concrete steel very easily with the new nonlinear interpolation model determined of mutual influence of all alloying elements and carbon. Numeral

tests of all created models have been shown good correspondence with the literature data and practice. All of models can be using very easily.

Powder Processing of Light Metal Alloys: Light Metal Alloys and Composites by Powder Metallurgy

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Composite Materials Committee, Powder Materials Committee
Program Organizers: Joseph W. Newkirk, University of Missouri-Rolla, Dept. of Metallurgical Engineering, Rolla, MO 65409-0340 USA; Iver Anderson, Iowa State University, Ames Lab, Ames, IA 50011-3020 USA; James C. Foley, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Marvin G. McKimpson, Michigan Technology University, Institute of Materials Processing, Houghton, MI 49931-1295 USA; James W. Sears, Lockheed Martin, KAPL, Inc., D2, 114, Schenectedy, NY 12301 USA

Monday PM
November 1, 1999
Room: Salon B&C
Location: Omni Netherland Plaza Hotel

Session Chair: James Foley, Iowa State University, Ames Laboratory, Ames, IA 50011-3020

2:00 PM Invited
P/M Aluminum Alloys and Processes: *Warren H. Hunt*[1]; [1]Aluminum Consultants Group Inc., 4530 William Penn Hwy., #3900, Murrysville, PA 15668-2002 USA

In the quest for cost-effective performance improvement in structural and non-structural applications, powder metallurgy-processed aluminum-based materials are receiving greater attention. A key to achieving the desired levels of performance and final part cost is a comprehensive understanding of the metallurgical factors that affect properties. This paper provides an overview of the characteristics of aluminum-based materials produced from both blended elemental powders and prealloyed powders by a range of processing routes including press and sinter, hot consolidation, and spray forming. In addition, the ability to further enhance properties in aluminum alloys through additions of ceramic particle reinforcements to form metal matrix composites is discussed.

2:30 PM Invited
The Interactive Role of Inclusions and SiC Reinforcement on the High Cycle Fatigue Resistance of Powder Metallurgy Particle Reinforced Metal Matrix Composites: *Nikhilesh Chawla*[1]; Yu-Lin Shen[2]; J. Wayne Jones[3]; John Allison[4]; [1]Hoeganaes Corporation, Rsrch. and Dev. Div., 1001 Taylors Ln., Cinnaminson, NJ 08077 USA; [2]University of New Mexico, Dept. of Mech. Eng., Albuquerque, NM USA; [3]University of Michigan, Dept. of Mats. Sci. and Eng., 2300 Hayward Dr., Ann Arbor, MI 48105 USA; [4]Ford Motor Company, Ford Rsrch. Lab., Dearborn, MI USA

The effect of intermetallic inclusions on the fatigue crack initiation and growth in powder metallurgy processed and extruded 2080 Al alloy and 2080/SiCp composites was investigated. Using surface replication, it was determined that, in the high cycle fatigue region, life is dominated by the initiation process. It was also determined that the majority of initiation sites were associated with intermetallic inclusions. While 2080/SiC/20p showed a definitive relationship between inclusion size and fatigue life, i.e., a higher inclusion size resulted in lower fatigue life, there was no correlation in 2080/SiC/30p. This was attributed to more of the load being shared by the higher volume fraction of SiC particles, and smaller average inclusion sizes in the latter composite. A conceptual model is proposed which accounts for these observations and to qualitatively show the effect of reinforcement and matrix strength on stress enhancement in near-surface inclusions.

3:00 PM
Hot Pressing the Powder Ti-Al-Si Alloys: *Yuri Michael Lytvynenko*[1]; Leonid Denis Kulak[1]; Svetlana V. Kapustnikova[2]; [1]Institute for Problems of Materials Science, 3 Krzhizhanovsky St., Kyiv 262680 Ukraine; [2]SMAU, 4 Gagarin Ave., Dnipropetrivsk 320636 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 quality as compare with cast ones to improve the level of the mechanical properties 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 minutes and at the pressures of 25 and 33 MPa. The microstructure and mechanical properties at the temperatures of 700, 800, 900 and 1000°C were investigated. A quality of the pressing depends on the pressing temperature more than on pressure and exposure. For each kind of the parts the optimal regime was developed because it is necessary to take into consideration the size and shape of ones. The porosity of the parts made at the optimal regimes is about 1%. The specimens for tests of bending strength were not destroyed at the temperature of 1000°C. The bending angle was about 90 degrees. The microstructure consists of the dispersed globulars of titanium silicides which were evenly distributed in the titanium matrix.

3:20 PM
Grain Growth in Ti-Based MMC Advanced Alloys Processed by Mechanical Alloying and Hot Isostatic Pressing: *Sedat Ozbilen*[1]; Cemil Cetinkaya[1]; [1]Gazi University, Metallu. Educ. Dept., Faculty of Tech. Edu., Ankara, Turkey

Grain growth in Ti-based alloys such as Ti-6Al-4V with or without B additions has been invesitgated. For this purpose, mechanically alloyed (MA) and hot isostatically pressed (HIP) powder materials were annealed at different temperatures for different annealing times. Light microscopy studies of these samples were carried out for grain size determination. Kinetics of grain growth of the b phase matrix without B additions and of an alloy matrix with b phase grains containing B additions were studied. The results were compared with those reported in the literature. Attention was paid to the stability of nano-grain structure and the effect of B additions to this aspect of grain growth study.

3:40 PM Break

3:50 PM Invited
Development of High Strength Aluminum Matrix Composites: *Dan Lawrynowicz*[1]; William C. Harrigan[1]; J. Kajuch[1]; [1]Alyn Corporation, 16761 Hale Ave., Irvine, CA 92606 USA

Metal matrix composites, as we know them today, have evolved significantly during the past 20 years. The primary support for these composites has come from the aerospace industry for airframe and spacecraft structures. More recently the automotive, electronic and recreation industries have been working with these composites. The driving force behind the development of most of the existing composites has been their capability to be designed to provide needed types of material behavior. Discontinuously reinforced metal matrix composites have virtually isotropic properties and lend themselves to metallic design methodologies as well as fit into the cost constraints of today's market. During the past few years, Alyn Corporation has been developing a boron carbide reinforced aluminum composite system that has yield strength near 90 ksi with ultimate strength near 100 ksi. This talk will discuss the

combination of mechanical and physical properties developed for this materials system.

4:20 PM Invited
Low-Cost, Powder-Metallurgy-Based Aluminum/Fly Ash Composites: *Marvin G. McKimpson*[1]; [1]Michigan Technological University, Instit. of Mats. Processing, 1400 Townsend Dr., Houghton, MI 49931 USA

Discontinuously reinforced aluminum metal matrix composites are potentially attractive engineering materials for a wide range of structural components, particularly in the transportation industries. At present, however, widespread application of these emerging materials is limited by the high cost of both component materials and fabrication processes. Fly ash, a waste product of coal combustion, is potentially attractive as an aluminum composite additive because of its low cost and demonstrated ability to improve the stiffness, wear and abrasion resistance of aluminum materials. In this paper, recent work related to press and sinter processing of aluminum-fly ash composites produced using cleaned, sized ash will be reviewed. This includes evaluation of several matrix alloy compositions and production of composites using gas atomized aluminum powders with enhanced sinterability. Processing of composites using several different sizes of fly ash reinforcements and different sintering treatments will be reported. Work sponsored by the U.S. Dept. of Energy under contract DE-FC26-98FT40324.

4:50 PM
Extrusion Simulation of 6092/SiC/17.5p Aluminum Composites: *Marvin G. McKimpson*[1]; Tim Loftin[2]; [1]Michigan Technological University, Instit. of Mats. Processing, 1400 Townsend Dr., Houghton, MI 49931 USA; [2]DWA Aluminum Composites, 21130 Superior St., Chatsworth, CA 91311 USA

Wrought, powder-metallurgy-based aluminum metal matrix composites are potentially attractive for a wide range of applications in the transportation industries. Acceptance of these materials for new applications, however, is hindered by the high cost and long lead times for developing and demonstrating required secondary processing operations. Expanded use of metal forming simulation tools can aid in reducing this hurdle. Accordingly, a program was undertaken to apply DEFORM, a commercial finite element simulation package, to axisymmetric extrusion of 6092/SiC/17.5, a powder based discontinuously reinforced aluminum composite. The work involved: 1) developing appropriate high temperature flow stress data on the composite; 2) extruding several 180 mm dia. billets down to 25 mm rounds on an instrumented commercial press; 3) simulating this extrusion operation using the DEFORM code; and 4) using this simulation to generate preliminary extrusion process windows. The results of this investigation will be reviewed, with particular emphasis on the flow behavior observed in the composite and the relevance of these findings for workability of the material. Work sponsored under USAF contract F33615-98-5212.

Refractory Metals & Alloys: A Symposium on Research, Development & Applications II: Nb and Its Alloys

Sponsored by: Structural Materials Division, Refractory Metals Committee, Corrosion and Environmental Effects Committee
Program Organizers: Mehmet Uz, Lafayette College, Chemical Engineering, Easton, PA 18042-1775 USA; Ken Natesan, Argonne National Laboratory, ET/212, Argonne, IL 60439-4838 USA

Monday PM
November 1, 1999
Room: Caprice 1&4
Location: Omni Netherland Plaza Hotel

Session Chairs: Mehmet Uz, Lafayette College, Chem. Eng. Dept., Easton, PA 18042-1775 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN USA

2:00 PM Invited
Microstructure of Nb-1Zr-C Sheet and Tube as Affected by Thermomechanical Processing and High Temperature Exposure: *Robert H. Titran*[1]; Mehmet Uz[2]; [1](Retired), NASA LeRC, 7146 Windwood Way, Olmsted Falls, OH 44138-1167 USA; [2]Lafayette College, Chem. Eng. Dept., 341 AHE, Easton, PA 18042 USA

Effects of thermomechanical processing on the microstructure of Nb-1wt.%Zr-(0.06-1) wt.%C sheet and tube were studied. Sheet samples from a vacuum-arc melted (VAR) ingot were fabricated by cold rolling bars that were single-, double-or triple-extruded at 1900 K. Samples were first given a two-step heat treatment, (DA), of 1 h\@1755 K+2 h \@ 1475 K. The DA samples were then exposed to 1350 K for times of up to ~34,500 h with and without an applied creep stress. Tube shells were fabricated from a second VAR ingot. Tube shells were extruded 8:1, one at 1900 K and a second at 1550 K. Thin-wall tubes were fabricated from each tube shell by 26 cold drawing operations with two in-process anneals. Tube samples were also given a DA heat treatment prior to any testing. Characterization of microstructure before and after each fabrication and testing step was performed using various metallographic and analytical methods. The results will be presented with the emphasis on the effects of processing on microstructure and its thermomechanical stability.

2:35 PM
The Creep Behavior of Refractory Metal Alloys: *Robert W. Buckman*[1]; [1]Refractory Metals Technology, P.O. Box 10055, Pittsburgh, PA 15236 USA

The creep behavior of niobium, tantalum, and molybdenum base alloys tested under uniaxial load will be compared with creep behavior obtained under biaxial test conditions. The biaxial configuration for creep testing is the most efficient method for generating a large volume of creep data. The alloys for which creep behavior will be discussed include the tantalum alloys Ta-10W, T-111, T-222, and ASTAR 811-C; niobium alloys FS-85, WC-103 and Nb-1Zr and molybdenum alloys M0-TZM and Mo-41-48Re. The test temperature range spans 0.35-.05Tm/. This paper will present and discuss creep-behavior of these materials for test times out to 45,000 hours.

3:10 PM
Oxidation of Refractory Metals in Air and Low-Pressure Oxygen Gas: *James R. DiStefano*[1]; Bruce A. Pint[1]; [1]Oak Ridge National Laboratory, Met. and Cera. Div., Oak Ridge, TN 37831-6157 USA

Despite their good high-temperature strength and corrosion resistance in certain environments, refractory metals and alloys generally have poor oxidation resistance in air above ~300°C. At elevated temperatures a low Po2 vacuum environment is usually required to prevent excessive scaling and internal oxidation (Nb, Ta, V) or oxide volatilization (Mo, W). The oxidation behavior of niobium tantalum, vanadium, and molybdenum alloys such as Nb-lZr, Ta-8W-1 Re-0.7 Hf (ASTAR-811C), V-4Cr-4Ti and Mo-46Re has been determined under a range of Po2 and time/temperature conditions. Oxidation rate equations were determined for Nb-lZr and ASTAR-811C. The mechanical properties of Nb-lZr and V-4Cr-4Ti were determined as a function of oxygen concentration. In general internal oxidation of Nb-lZr at intermediate temperatures results in an increase in strength and decrease in ductility. A loss in ductility is the principal effect of increasing oxygen concentration in V-4Cr-4Ti at temperatures around 500°C. In addition, for this alloy a synergism occurred from contamination by both hydrogen and oxygen that reduced ductility beyond that predicted from contamination of the individual gases. After oxidation, heat-treating both Nb-1Zr and V-4Cr-4Ti at high temperature (~950°C) resulted in higher ductility. Microstructural studies indicated this to be related to precipitation of ZrO2 and TiOx, respectively. Research sponsored by the U.S. Department of Energy, Space and National Security Programs at the Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corporation, under Contract DE-AC05-96OR22464 with the U.S. Department of Energy.

3:45 PM Break

4:00 PM

The Use of Phase Equilibria and Transformations for the Microstructural Control of Sigma and Second Phase Alloys in the Nb-Ti-Al System: *Vijay K. Vasudevan*[1]; Joseph C. Mishurda[2]; [1]University of Cincinnati, Dept. of Matls. Sci. and Eng., ERC 501-D, Cincinnati, OH 45221-0012 USA; [2]University of Cincinnati, Dept. of Matls. Sci. and Eng., ERC 560, Cincinnati, OH 45221-0012 USA

In the search for creep resistant materials, intermetallics such as the sigma phase in the Nb-Ti-Al system have been considered. In order to circumvent the problems with low ductility two approaches may be considered: 1) incorporate a more ductile phase (such as beta) or 2) introduce another intermetallic to alter the fracture path (such as gamma). The phase equilibria and transformations of alloys based on sigma + beta and sigma + gamma in the Nb-Ti-Al system, have been sufficiently established to permit the control of the microstructural evolution to produce desirable microstructural distributions of the phases. An initial examination of the transformations within the alloys of interest was performed by differential thermal analysis (DTA). These transformations were then identified by thermal treatments at selected temperatures and the resulting structures fully characterize by x-ray diffraction (XRD), optical metallography (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron probe micro analysis (EPMA). The equilibrium tie lines were established to permit the prediction of volume fraction of phases present. The mechanisms resulting in the desirable distribution of the microstructural constituents were identified. Finally, the low temperature stability at 700°C was examined and potential phase transformations were identified which may effect the application of the alloys.

4:20 PM

Damage Tolerant Nb-Al-Ti Base Alloys for Intermediate Temperature Applications: *Winston O. Soboyejo*[1]; Mingwei Li[1]; R. Hayes[2]; E. Loria[3]; [1]Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Metals Technology Inc., 19801 Nordhof St., Northridge, CA 91324 USA; [3]1828 Taper Drive, Pittsburgh, PA 15241 USA

In recent years, a new class of damage tolerant Nb-Al-Ti alloys and intermetallics have emerged for potential structural applications in the intermediate-temperature regime between 650 and 750°C. This paper presents a review of the structure and mechanical properties of these predominantly beta or B2 alloys/intemetallics. The alloys are shown to have attractive combinations of moderate density (~ 6.1 to 6.5 g/cm^3), room temperature ductility (~ 5 to 30%), fracture toughness (40-110 MPam$^{1/2}$) and comparable fatigue crack growth resistance to structural alloys such as Ti-6Al-4V, Inconnel 718 and Nb-base alloys. The creep properties of the Nb-Al-Ti base alloys are also compared with those of orthorhomic and gamma-based titanium aluminide alloys in the intermediate-temperature regime. The implications of the results are discussed for potential applications in aerospace and land-based engines.

4:40 PM

Deformation Behavior of Nb-Ti-Al Alloys as a Function of Temperature: *Sundar Amancherla*[1]; Richard J. Grylls[2]; Srikumar Banerjee[3]; Hamish L. Fraser[1]; [1]Ohio State University, 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]GE Aircraft Engines, Cincinnati, OH 45215 USA; [3]Bhabha Atomic Research Center, Mumbai 400 085 India

Alloys based on the composition Nb-40Ti-15Al (at%) are being investigated for possible use in high-temperature corrosive environments. These B2-matrix alloys show an interesting combination of mechanical properties, including high yield strength maintained through 800°C, and room temperature ductility exceeding 30%. On aging this alloy at intermediate temperatures, a yield-point is observed on subsequent room-temperature tensile deformation. Also, discontinuous yielding is observed upon testing at intermediate temperatures. These phenomena have been investigated from both a mechanical and microstructural standpoint, and both areas will be addressed here. On testing at intermediate temperatures, a critical strain for the appearance of serrated yielding and a negative strain-rate sensitivity are observed, consistent with a Portevin-Le-Chatelier effect. The microstructural changes during yielding have been studied using transmission electron microscopy (TEM). Localized slip is prevalent, although this does not impact the ductility. Evidence will be presented which suggests that it is the dynamic precipitation and subsequent dissolution of the w-phase which is responsible for the reported effects. Computational efforts aimed at determining the same have been undertaken, and the computational and the experimental results will be compared and contrasted.

5:00 PM

Ordering in Ternary B2 Alloys: Sundar Amancherla[1]; Rajarshi Banerjee[1]; Srikumar Banerjee[2]; *Hamish L. Fraser*[1]; [1]Ohio State University, 2041 College Rd., Columbus, OH 43210 USA; [2]Bhabha Atomic Research Center, Mumbai 400 085 India

The properties of ordered intermetallic materials, especially their mechanical properties, are often strongly dependent on the type and degree of ordering in such materials. Therefore, a better understanding of the complex ordering schemes in these materials will help in understanding as well as tailoring their properties. One of the most commonly observed ordering schemes in bcc based intermetallic alloys is the B2 structure which divides the bcc lattice into two primitive cubic sublattices. In binary B2 alloys, the long range order (LRO) can be described in terms of a single LRO parameter which makes the description of the ordering state relatively simple. However, in ternary intermetallic alloys, two independent LRO parameters are required in order to describe the complex ordering schemes which can develop. An elegant graphical description of the ordering in ternary B2 alloys is the Ordering Tie Line (OTL) which allows a simple visualization of the ordering schemes while rigorously encompassing the information of the two LRO parameters. This paper will focus on the ordering schemes in ternary B2 alloys based on the Nb-Ti-Al system as well as Fe-Al and Ni-Al systems with ternary additions. The OTLs have been calculated using a model based on an exchange reaction rate approach to predicting equilibrium properties. The model is based on the classical Bragg-Williams theory of order-disorder transformations and uses a nearest-neighbor approximation. Experimentally, the OTLs have been determined by measurements of the apparent sublattice

compositions using atom location by channeling enhanced microanalysis (ALCHEMI). A comparison of the calculated and experimentally determined results will be presented emphasizing on the power of the OTL representation. The difficulties associated with a purely numerical description of the ordering in terms of the sublattice compositions in the B2 structure will also be discussed.

Symposium to Honor Professor Julia R. Weertman: High-Temperature Deformation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Monday PM
November 1, 1999
Room: Rosewood
Location: Omni Netherland Plaza Hotel

Session Chairs: William Nix, Stanford University, Stanford, CA 94305 USA; David Dunand, Northwestern University, Evanston, IL 60208 USA,

2:00 PM
Application of Phase Transformation Theory to Fatigue of Solders: *Morris E. Fine*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Defining phase transformations broadly, initiation and growth of fatigue cracks are phasae transformations. Nucleation theory may be applied to nucleation of fatigue cracks and growth theory to propagation of fatigue cracks. Fatigue of lead-tin solders which have high homologous temperatures even at room temperature will be discussed on this basis.

2:30 PM
Creep Properties of Two Precipitation Strengthened, Tin Based Alloys: *Rodney J. McCabe*[1]; Morris E. Fine[1]; [1]Northwestern University, Mats. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Because of their relatively low melting temperatures, creep plays a prominent role in the deformation of tin based solder alloys at room temperature and above. The creep resistance of these alloys can be improved significantly by precipitation hardening. The creep properties of two precipitation strengthened tin alloys, Sn-8Sb and Sn-2In-2Sb, were examined. In the temperature and stress regimes tested, both alloys exhibit two distinct regions with respect to stress and strain rate. The high stress region is characterized by an activation energy of 95 kJ/mol and stress exponents ranging between 10 and 15. In the low stress region, both alloys exhibit a stress exponents between of 4.5 and 5.6. The low stress activation energy for Sn-8Sb is 48 kJ/mol and that for Sn-2In-2Sb is roughly 68 kJ/mol. This behavior is rationalized in terms of measured microstructural characteristics using existing models of creep in precipitation and dispersion hardened metals.

2:45 PM
Shear Properties of Solder Joints: *Robert A. Gagliano*[1]; Morris E. Fine[1]; Semyon Vaynman[1]; Vladimir Stolkarts[1]; [1]Northwestern University, Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60202 USA

Due to heating and cooling and thermal expansion mismatch of components, shear stresses are imposed on solder interconnects. An apparatus was developed to test solder joints in shear. Using the apparatus, monotonic shear loading experiments were performed on eutectic tin lead (63Sn-37Pb) solder joints to investigate the effect that various parameters, such as joint area, joint thickness, and melt time had on the maximum shear strength of the joints. It was found that the strength of the solder was essentially independent of the joint area (within the range from 19-27 mm2) and thickness (between 80 mm 300 mm) and only marginally dependent on the total melt time of the solder (for times up to ten minutes). Typical shear strains to failure were on the order of 100%. Observations of the fracture surface, via SEM, revealed that for all cases, the failure was ductile in nature and took place within the solder, and not at an interface or within the intermetallic. Actual silicon to alumina C4 devices, with 132, 762, and 1475 interconnects with composition of 95.5 wt%Pb-3.5 wt%Sn, were also tested in shear, and the fracture strengths were found to be close to those obtained in bulk specimens of the same alloy.

3:00 PM
Tensile Creep Properties of the 50Au-50Cu Braze Alloy: *John J. Stephens*[1]; [1]Sandia National Laboratories, Mats. Joining Dept. (1833), MS0367, P.O. Box 5800, Albuquerque, NM 87185-0367 USA

The 50Au-50Cu (wt.%) alloy is a solid-solution strengthened braze alloy used extensively in conventional, hermetic metal/ceramic brazing applications where low vapor pressure is a requirement. The elevated temperature mechanical properties are important for permitting FEA evaluation of residual stresses in metal/ceramic brazes given specific geometries and braze cooldown profiles. For material that was annealed for 2 hr. at 750°C and water quenched, a Garofalo sinh equation was found to adequately characterize the minimum strain rate data over the temperature range 450-850°C. At lower temperatures (250 and 350°C), a conventional power law equation was found to characterize the data. For samples held long periods of time at 375°C (96 hrs.) and slowly cooled to room temperature, a slight strengthening reaction was observed: with the stress necessary to reach the same strain rate increasing by about 15% above the baseline annealed and quenched data. However, the relative sluggishness of this ordering reaction does not appear to pose a problem for braze joints cooled at reasonable rates following brazing. The microstructure of fractured creep samples will also be discussed. This work was supported by the U.S. Dept. of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U. S. Department of Energy.

3:15 PM
Creep Behavior of a 7005 Al Composite Containing 10 vol.% Al2O3 Particulates: *Bing Q. Han*[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Mats. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA

Creep tests were conducted over the temperature range from 573 to 773 K on an Al 7005 matrix composite reinforced with 10 vol.% Al2O3 particulates. The results cover almost six orders of magnitude of strain rate and give high values for the apparent stress exponent and the apparent activation energy. The threshold stresses were calculated and creep behavior was compared with the results of an Al 7005 matrix composite with 20 vol.% Al2O3 particulates. The effect of volume fraction of particulates on the creep behavior was analyzed.

3:30 PM Break

3:50 PM
New in High Temperature Creep of Pure and Optimally Alloyed Single Crystals of Refractory Metals: *Oleksandr Illich Dekhtyar*[1]; [1]Kurdumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Inhomogeneous Alloys, 36 Vernadsky St., Kiev 252142 Ukraine

Creep mechanisms and characteristics of pure and optimally alloyed Mo and W single crystals were studied at high (0.57-0.61 of melting point) temperatures in wide applied stress region by light microscopy, X-ray diffraction and transmission electron microscopy methods. It was found that creep rate is controlled by two main mechanisms in general case. The first is a climb of the individual independent edge dislocations at low stresses. The second is a collective climb of the groups of strongly interacted edge dislocations at high stresses. Influence of different substructure strengthening treatments on the creep properties were investigated. It was found that effective substructure strengthening is possible at stresses where the first mechanism is acting only. The interesting peculiarities were found of creep of the optimally alloyed solid solutions. They are connected with characteristics of distribution of the alloyed atoms in the cores of dislocations.

4:05 PM

Deformation by a Kink Mechanism in High Temperature Materials: *Terence E. Mitchell*[1]; John P. Hirth[1]; Pedro Peralta[2]; [1]Los Alamos National Laboratory, Ctr. for Mats. Sci., Mail Stop K765, Los Alamos, NM 87545 USA; [2]Arizona State University, Mech. and Aerospace Eng., P.O. Box 876106, Tempe, AZ 85254 USA

A new double kink dislocation model has been developed to explain the temperature dependence of the yield stress in materials such as oxides and intermetallics that require high temperatures for plastic flow. The major variation in the free energy of double kink nucleus formation with stress is still the kink-kink activation energy. However, there is also a stress dependence of the pre-exponential factor in the strain rate constitutive equation arising from kink diffusion. Numerical solution of the resulting equations shows that there are temperature regimes where the stress can vary logarithmically either directly with temperature or with inverse temperature. The model explains quantitatively the temperature dependence of the critical resolved shear stress (CRSS) on different slip systems for sapphire and spinel in terms of different activation energies for kink diffusion. The model can be modified to explain compositional softening in spinel by incorporating enhanced kink nucleation at cation vacancies. This changes the pre-exponential term and explains why the CRSS decreases as the inverse of the square of the vacancy concentration, as observed experimentally.

4:20 PM

Effect of Sulfur on the Deformation Mechanisms of Zirconium Alloys at Intermediate Temperatures (25°C-400°C): F. Ferrer[1]; A. Barbu[2]; *Thierry Bretheau*[1]; [1]Ecole Polytechnique, Laboratoire de Mecanique des Solides, Palaiseau 91128 France; [2]Ecole Polytechnique, Laboratoire des Solide, Irradies, France

A small addition of sulfur (about 20 wt ppm) reduces steady state creep rate of several zirconium alloys by 3 at 400°C under a 130MPa applied stress [1]. A so strong effect of a so low sulfur content suggests that sulfur directly acts on the deformation mechanisms. Nevertheless these mechanisms are still not well understood although a lot of studies have been performed [2-5]. The present study aims both at the understanding of the effect of sulfur and at a better knowledge of the controlling mechanisms in 25-400°C temperature range, specially under creep conditions. As very little is known about the mechanisms controlling the deformation of zirconium at intermediate temperature, all potential ones are considered: grain boundary sliding, self-diffusion and dislocation motion (cross-slip, lattice friction). Each one is discussed with regards to experimental results and to the plausible effects of a very low amount of sulfur in order to get the relevant one(s). This will in return enable a better understanding of the effect of sulfur. Grain Boundary Sliding: Fiducial micro-grids, deposited on the sample surface (ZrNb1% alloy), show that, whatever the sulfur content, there is no shearing of the grid lines crossing grain boundaries. It implies that there is no major grain boundary sliding. Moreover, tensile tests under constant strain rate (=6.10-5s-1) at 400°C on pure a zirconium (with 1200wt ppm O), show that the strengthening amount due to sulfur is independent of grain size (50, 200,800 µm). This leads to conclude that sulfur is not acting in the grain boundaries and that Grain Boundary Sliding is not the controlling mechanism in the temperature range 25°C-400°C. Lattice Self-Diffusion: Lattice Self Diffusion is often evoked as being the creep controlling mechanism at intermediate temperature [6]. In zirconium the maximum value for the self-diffusion coefficient reported in literature [7] is DL=10-27m2s-1 at T=400°C. Whatever the details of a model based on lattice self-diffusion, the calculated creep rate is several orders lower than the experimental one. This implies that lattice self-diffusion cannot be the controlling mechanism in this temperature range. Moreover it can be mentioned that the sulfur effect appears at 100°C which is a much too low temperature for a significant bulk diffusion. Then, bulk diffusion is not a relevant controlling mechanism at intermediate temperature and the sulfur effect does not result from a sulfur/vacancy interaction. Dislocation glide A study based both on the characterization of slip lines and on TEM observations shows, independently of sulfur content, that: for T<200°C prismatic slip is mainly activated under the form of long screw <a> dislocations ? for T>200°C prismatic slip remains preponderant (80%) but 1st order pyramidal slip {1101}<1120> is more frequently observed ; slip lines become wavy indicating the occurrence of cross slip. The dislocations are still of <a> type but most of them are bowed in pyramidal planes. Concerning the interaction between dislocations and sulfur atoms, three cases can be considered: 1-sulfur is immobile ; 2-sulfur is mobile and follows dislocations (Dynamic Strain Aging (DSA)) ; 3-sulfur atoms segregate into the dislocation core and modify it. On the basis of a model proposed by Friedel [8], it can be concluded that sulfur concentration is much too low to justify a so strong effect for immobile impurities ; this allows to exclude case 1. At a given temperature sulfur atoms diffuse at a velocity vs. If dislocations are moving with a mean velocity vd>>vs, sulfur cannot follow dislocations and there is no dragging force. If dislocations are moving too low (vd<<vs), the equilibrium is always instantaneously reached around dislocations and no dragging force is exerted. The dragging force then only exists when vd#vs, where a dynamic equilibrium is reached. Figure 1: s2% vs T for two different sulfur contents Tests performed on a zirconium show that sulfur effect reaches a maximum at T=400°C for a 6.10-5s-1 strain rate (Fig.1); then probably vd#vs. At the same temperature but for a much lower strain rate (2.10-8s-1; vd<<vs), the effect is still present with the same magnitude what excludes case 2 (DSA) mechanism. On the contrary, case 3 (segregation) remains perfectly plausible. At 250°C for a high strain rate (5.10-2s-1; vd>>vs) the effect of sulfur tends to disappear. Then it can be concluded that sulfur atoms segregate in dislocation cores and follow them whether dislocation velocity is not too high. They probably modify the core structure what can affect strongly their behavior (Peierls force, cross-slip, core self-diffusion). Complimentary tests and core numerical simulations are in progress to test these hypotheses. REFERENCES[1]. D. Charquet, J. Senevat, J.P. Marcon, J.N.M., 255, 78-82 (1998). [2]. A.J. Ardell and O.D. Sherby, TMS of AIME, 239, 1547 (1967).[3]. E.R. Gilbert, S.A. Duran and A.L. Bement, ASTM STP 458, 210-225 (1969). [4]. W.R Thorpe and I.O Smith, J.N.M., 75, 209-219 (1978). [5]. M. Pahutova and J. Cadek, Mat. Sci. and Eng., 11, 151-162 (1973). [7]. O.D Sherby and J.Weertman, Acta Met, 27, 387-400 (1979). [9]. M.C. Naik and R.P. Agarwala, Acta Met., 15, 1521 (1967). [12]. J. Friedel, "Dislocations", Pergamon Press, Paris, p395 (1964).

4:35 PM

Interpretation of Microstructures in High Temperature Deformation: *Hugh J. McQueen*[1]; [1]Concordia University, Dept. of Mech. Eng., 1455 de Maisonneuve Blvd. W., H-549, Montreal, Quebec H3G 1M8 Canada

In each recent historical period, the microstructures produced by high temperature straining were probed by the current technology, thus giving rise to new models and theories of rate controlling mechanisms. The progress in understanding has not been monotonic since occasionally theories were developed to high levels of sophistication while overlooking aspects of the substructure which

were to become significant. New technologies such as TEM, or SEM-EBSP-OIM have made possible great leaps forward but often leave unresolved problems on a different scale. Experimental observations are presented of substructures in Al (with solute, dynamic precipitates, dispersoids and reinforcing particles), in Mg alloys and in both austenitic and ferritic stainless steels, thus providing a range of crystal structures and stacking fault energies. After the historical analysis, the current view of the hot worked state will be presented with comparison of the conflicting theories. The analysis is centered on dislocation strain and there is only mention of pertinent interactions with grain boundary related deformation.

4:50 PM
Superplastic Behavior of TiAl Based Alloy with Rapidly Hot-deformed Microstructures at Low Temperatures: *Yuehui He*[1]; *Baiyun Huang*[2]; *P. K. Liaw*[1]; [1]University of Tennessee, Mats. Sci. and Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Central South University of Technology, Powder Metallu. Rsrch. Instit., Changsha, Hunan 410083 ROC
Abstract Text Not Available

5:05 PM
Dynamic Grain Growth as a Key to Superplasticity?: John R. Seidensticker[1]; *Merrilea J. Mayo*[2]; [1]National Institute of Standards and Technology, Ceramics Div., Bldg. 223, Rm. A367, Gaithersburg, MD 20899 USA; [2]Pennsylvania State University, Dept. Mats. Sci. & Eng., Rm. 115 Steidle, University Park, PA 16803 USA

Daily Personal Schedule

Tuesday, November 2, 1999

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
10:30 am				
11:00 am				
11:30 am				
12:00 pm				
12:30 pm				
1:00 pm				
1:30 pm				
2:00 pm				
2:30 pm				
3:00 pm				
3:30 pm				
4:00 pm				
4:30 pm				
5:00 pm				
5:30 pm				
6:00 pm				
6:30 pm				

Though superplasticity in metals and ceramics is widely different in many respects—different stress exponents, grain size exponents, strains to failure, etc.—the one rule that both metal and ceramics obey in common, with fair precision, is the dynamic grain growth law. This brings up the intriguing possibility that dynamic grain growth is somehow fundamentally at the root of superplastic deformation. A theoretical basis for such a law is derived. It is shown that the corresponding microstructural mechanism is one in which stress-assisted grain growth leads to geometrically necessary "holes" in the microstructure, thereby providing spaces into which adjoining grains can slide.

Advances in Casting High Temperature Alloys—Materials, Applications & Modeling: Superalloys

Sponsored by: Structural Materials Division, High Temperature Alloys Committee
Program Organizers: Boyd Mueller, Howmet Corporation, Whitehall, MI 49461 USA; John J. Schirra, Pratt & Whitney, East Hartford, CT 06108 USA

Tuesday AM — Room: Salon F&G
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Boyd A. Mueller, Howmet, Electronic Product Technology, Whitehall, MI 49461 USA; Jack Schirra, Pratt & Whitney, Materials, East Hartford, CT 06108 USA

8:30 AM
Improved Directional Solidification via Liquid Metal Cooling: *A. Lohmuller*[1]; M. Hordler[1]; A. Pfennig[1]; R. F. Singer[1]; A. Volek[1]; J. Preuhs[2]; J. Grossmann[2]; W. Esser[3]; [1]Lehrstuhl Werkstoffkunde und Technologie der Metalle, Martensstrasse 5, Erlangen D-91058 Germany; [2]TFB Feingusswerk Bochum, Bessemerstrasse 80, Bochum D-44793 Germany; [3]Siemens KWU, Wiesenstrasse 35, Mulheim a.d. Ruhr D-45473 Germany

Higher demands on turbine blades for industrial gas turbines (IGTs) have increased the importance of directional solidification of SX- and DS-components. The trend towards larger components and complex alloy compositions revealed the limits of the conventional Bridgman-(HRS)-technique. At WTM, a pilot plant (max. 38 kg superalloy melt) has been set up to develop a new technology, the Liquid Metal Cooling (LMC)-process. Higher temperature gradients and increased withdrawal rates can be achieved. This results in finer microstructure, higher productivity and reduced time for heat treatment. First promising results indicate the potential of the process [1]. This paper presents the pilot plant at WTM. Several important modifications of the original vacuum furnace design that ensure a good casting quality are reviewed. LMC-and HRS-technique are compared for a cored IGT-blade (~290 mm in length) both for identical casting conditions and for conditions that are typical for each process. Characteristic microstructural values like primary and secondary dendrite arm spacings or g^1-morphology illustrate the advantages of the LMC-process. Measurements on microsegregation are evaluated. The influence of several process parameters (e.g. withdrawal rate) on the microstructure is given adequate attention. In addition, a parameter study on small test pieces contributes to a better fundamental understanding of the LMC-process and shows its potential and limitations. [1] J. Grossmann, J. Preuhs, W. Esser, R.F. Singer. Investment Casting of High Performance Turbine Blades by Liquid Metal Cooling-A Step Forward Towards Industrial Scale Manufacturing-Int. Symp. on Liquid Metal Proc. and Cast.. Sante Fe, New Mexico. Feb. 1999. Vacuum Metallurgy Division of AVS. 31-40.

8:55 AM
Advances in Casting Large Single Crystal Components for Industrial Gas Turbines: *Allen R. Price*[1]; Boyd A. Mueller[1]; [1]Howmet Research Corporation, Adv. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA

The objective of the Land Based Turbine Casting Initiative is to develop and implement technology specifically for land based turbine size components. The technology development occurred in the areas of: low sulfur alloy production, casting process development and improvement, post-casting process development and improvement, and defect tolerance limit establishment. To accomplish the technology development, and ensure the technology advances are transitioned to the industry in a timely manner, Howmet was teamed with utility and industrial gas turbine OEM's. This team addressed the entire chain to produce land based gas turbine castings including: alloy production, investment casting, and engine builders. The team includes: ABB, ARACOR, General Electric Corporate Research and Development, Howmet Corporation, Pratt & Whitney, Solar, and Siemens Westinghouse. This paper will describe the status of the program in each of the above technology thrust areas. In the area of low sulfur alloy, efforts completed include scale-up of Howmet's low sulfur melt processing to production size master heats and oxidation testing of production and low sulfur heats of N5 and PWA-1484. Samples of both 100% virgin heats and virgin/revert blend heats of CMSX-4, and a production heat of CMSX-10 were also tested. Casting process development efforts include benchmarking of industrial and utility sized components to prioritize the common casting defects. Based on the results of the benchmarking, casting experiments were completed on representative utility-sized components. In addition, the benchmarking and development efforts focused on core, wax, and shell materials to improve dimensional quality. Post-cast process development activities involve examining the heat treat response of representative second and third generation single crystal alloys, the impact of part processing on final quality, and developing an NDI process to inspect internal blade quality. For the defect tolerance limit establishment efforts, test material has been produced which contain typical single crystal defects in selected second and third generation alloys. The defects consist of off-orientation, freckles, low angle boundaries, and recrystallized grains. General results will be reviewed and significant accomplishments will be discussed. This research was sponsored by the US Department of Energy's Office of Fossil Energy (Morgantown Energy Technology Center) and Office of Energy Efficiency and Renewable Energy (Office of Industrial Technology) through Oak Ridge National Laboratory.

9:20 AM
Development of a Freckle Predictor for Use in Casting Simulation: *Christoph Beckermann*[1]; [1]University of Iowa, Mech. Eng., 2412 SC, Iowa City, IA 52242 USA

A freckle predictor has been developed for use in full-scale casting simulations of the directional solidification of single-crystal Ni-base superalloys. The freckle predictor is based on a Rayleigh number criterion that has been established using (i) a small-scale simulation model of the flow and multi-component macrosegregation during solidification and (ii) results from specially designed solidification experiments. The Rayleigh number is evaluated in the full-scale casting simulation using local thermal information, solid fraction profiles, and liquid densities. The liquid densities are obtained from detailed calculations of the multi-component thermodynamic equilibrium and solidification path. Examples are presented of the use of the freckle predictor in the simulation of superalloy castings.

9:45 AM
Investment Casting Applications of Process Modeling: *David Hirvo*[1]; Tyrus Hansen[1]; Terry Simon[1]; Boyd A. Mueller[1]; [1]Howmet Corporation, Elect. Pro. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA

Process Modeling is being employed to reduce the development cycles times and improve the quality of investment cast nickel-base superalloys. The application of Process Modeling is the result of reductions in model construction time and improvements in model accuracy and predictive capability. Process Modeling is able to accurately predict macroshrinkage, thermal profiles, and the extent and shape of the solidification zone, and provide tendencies for microporosity and single crystal grain defects such as spurious grains, freckles, and boundaries. In this presentation we highlight the above with examples to production investment castings. Areas for further research to advance the state of the art will be overviewed.

10:10 AM Break

10:20 AM

Utilization of Electronic Product Definition, Investment Casting and Rapid Prototyping Techniques to Produce Experimental High Temperature Material Components: *Marc R. Sauerhoefer*

The progress of computerized product definition and computer integrated manufacturing has provided opportunities in Manufacturing to compress both lead-time and cost while rapidly responding to new design concepts. Large impact is found during the development and preproduction cycles for investment castings of aerospace hardware. Rapid Prototyping, coupled with investment casting, enables experimental and pre-production volumes of hardware to be fabricated directly from a computer solid model. From low-performance equiax to high-performance single crystal materials, highly complex designs are fabricated concurrent with process development. This has resulted in reduced development costs in generating functional test hardware in end state materials while providing greater design flexibility in a fraction of the time.

10:45 AM

Advances in Solidification of Thin Wall Superalloy Castings: *Laurentiu Nastac*[1]; Juan J. Valencia[1]; Thomas C. Kiesling[1]; Michael L. Tims[1]; Sanjay B. Shendye[2]; Mary Lee Gambone[3]; [1]Concurrent Technologies Corporation, Manufacturing Tech. Directorate, 100 CTC Dr., Johnstown, PA 15904-1935 USA; [2]PCC Structurals Inc., Matls. and Tech., 4600 S.E. Harney Dr., Portland, Oregon 97206-0898 USA; [3]Rolls-Royce Allison, P.O. Box 420, Speed Code W-05, Indianapolis, IN 46206-0420 USA

The goal of this work is to develop and optimize manufacturing methods for production of expansive (250 mm x 2000 mm), thin wall (i.e., around 1 mm) structural castings of nickel-based superalloys. A new, proprietary investment casting process, Thermally Controlled Solidification (TCS), developed by PCC Structurals, Inc. (PCC), has been chosen for casting of the diffuser/combustor casing for the AE1107C engine of the Navy V-22 Osprey helicopter. RS5 nickel-based superalloy has been identified as a promising choice for the casing because its high-temperature mechanical properties are superior to those of common superalloys, including IN718. However, production experience with RS5 alloy is limited. One of the objectives of this work is to analyze and compare the solidification characteristics of RS5 and IN718 alloys. In this regard, a methodological approach for estimation of the solidification parameters of multicomponent alloys was developed. The methodology is based on a deterministic solidification-kinetics approach for multicomponent and pseudo-binary alloy systems. The following solidification-related parameters were estimated for IN718 and RS5 alloys: liquidus and solidus temperatures, temperature-solid fraction relationship, liquidus slopes, distribution coefficients, constitutional undercooling parameters, equiaxed and columnar grain-growth coefficients, as well as primary and secondary dendrite arm spacing coefficients. Computation of these parameters considered dynamic coarsening, microsegregation at the scale of dendrite arm spacing, and diffusion in both liquid and solid phases. A comparison between RS5 and IN718 alloys in terms of solidification morphologies is provided. Calculated and experimental results for primary and secondary dendrite arm spacings as related to solidification conditions in the TCS process will be presented. The effects of withdrawal rate on the solidification structures of RS5 and IN718 will be discussed in detail. Current theoretical calculations have revealed the following: (a) the secondary dendrite arm spacing in cast IN718 alloy is approximately 15% larger than that in cast RS5 alloy, while the primary dendrite arm spacing in cast IN718 alloys is about 15% smaller than that in cast RS5 alloy; (b) the main contributing elements in descending order for the growth of the primary phase based on the constitutional undercooling parameter are (i) for IN718: Nb, Ti, Cr, Mo, and Al and (ii) for RS5: Nb, Ti, Co, Mo, Cr, and Al; (c) the overall constitutional undercooling parameter of RS5 is about twice that of IN718 alloy, meaning that RS5 might be more susceptible than IN718 to the formation of solidification-related defects; and (d) withdrawal rate has a significant effect on the solidification structure of both superalloys. In-house experiments were also performed to determine a window of process and material parameters for obtaining the optimum microstructure (including secondary phases and columnar-to-equiaxed transition) in the TCS processed RS5 and IN718 alloys. The microsegregation profiles of several elements were evaluated by Energy Dispersive X-ray Spectroscopy (EDS) as a function of cooling rate for TCS processed RS5 bar castings. The experimentally measured microsegregation profiles were used for calculating the effective distribution coefficients and for estimating the dendrite arm spacings. By using DTA measurements and techniques for metallographic characterization, it was demonstrated that secondary phases (carbides and Laves phases) are formed in both superalloys. Also, similar solidification paths occur in both alloys. The DTA measurements have also confirmed some of the theoretical calculations of the solidification characteristics of these superalloys. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under contract No. N00140-92-C-BC49 to the U.S. Navy as part of the U.S. Navy Manufacturing Technology Program.

11:10 AM

Computational Simulation of the Directional Solidification of PWA-1484 Alloy: *Deming Wang*[1]; Lichen Li[2]; *Ruel A. Overfelt*[2]; [1]Auburn University, Space Power Insti., 231 Leach Ctr., Auburn, AL 36949-5320 USA; [2]Auburn University, Mech. Eng., 201 Ross Hall, Auburn, AL 36849-5341 USA

Current research at Auburn University on nickel-based superalloys is exploiting the directional solidification (DS) technique to minimize convective effects plus decouple the effects of thermal gradient and solidification velocity on microstructural development. These experimental techniques enable rigorous control of the solidification process that results in highly repeatable microstructures. A baffle is used to isolate the hot and cool zones of the laboratory furnace and to flatten the isotherms in the mushy zone. Computer-aided heat and mass transfer simulation techniques are being used to complement the laboratory experiments and are leading to increased understanding of the process. The contribution of the paper is to provide a simple and efficient numerical calculation method to evaluate heat transfer in a practical laboratory furnace for DS. Finite difference equations derived from the control volume method are used to calculate the transient axisymmetric heat transfer (heat conduction/convection and heat radiation). Two different kinds of finite control volumes are respectively used for heat conduction/convection and heat radiation. A net-radiation method with changeable view factors is used during DS processing as the casting sample moves out of the furnace chamber. The simulation results have been verified by experimental data (in-situ thermocouple data and microstructural features) on the nickel-base superalloy PWA 1484.

11:35 AM

A Study on the Tensile Behavior of Polycrystalline Superalloy IN738LC: *Ercan Balikci*[1]; *Reza A. Mirshams*[2]; A. Raman[1]; [1]Louisiana State University, Mech. Eng. Dept., Baton Rouge, LA 70803 USA; [2]Southern University and A&M College, Mech. Eng. Dept., P.O. Box 9987, Baton Rouge, LA 70813 USA

The tensile properties of superalloy IN738LC, having different precipitate microstructures, are evaluated at room temperature, 650°C, 750°C, and 850°C at two different strain rates. The properties can be presented in two groups based on the comparable closeness of the values obtained those of microstructures C and M, with coarse and medium size precipitates, and those of microstructures F and D, with fine and duplex size (medium + fine) precipitates. Preferred orientations, lattice parameters, and metallography are used to characterize the microstructure and tensile testing to determine the yield strength, tensile strength, and strain hardening coefficients. An anomalous increase in yield strength is observed, which occurs at temperatures about 100°C higher with higher strain rate than with lower strain rate applied. The experimental results show that the yield strength is influenced by preferred orientations and precipitate size, while the tensile strength is effected by the size and morphology of precipitates.

Beryllium and Beryllium Alloys; Nuclear and Structural Applications: General Beryllium Issues

Sponsored by: Jt. ASM-MSCTS/TMS-SMD, Nuclear Materials Committee

Program Organizers: Michael F. Stevens, Los Alamos National Laboratory, MS G755, Los Alamos, NM 87545 USA; David J. Michel, Naval Research Lab., Code 6301, Washington, DC 20375 USA

Tuesday AM — Room: Salon M
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: David Michel, Naval Research Laboratory, Code 6301, Washington, DC 20375 USA

8:30 AM Opening Comments and Welcome: Dave Michel and Mike Stevens

8:40 AM
BeAl Alloys-Past, Present and Future: *Gilbert J. London*[1]; [1]Naval Air Warfare Center, Patuxent River, MD 20670 USA

Be metal has limited ductility principally because it cleaves on its primary slip plane leading to early fracture. The lack of non-basal slip systems doesn't help. BeAl alloys were developed in the 60's as a lower cost and possibly more versatile alternative to 'pure' Beryllium. The first commercially exploited alloy, Lockalloy, a binary 62Be38Al alloy was produced by atomization followed by extrusion and hot rolling. Renewed interest developed in the early 90's during the National Aerospace Plane (NASP) program and has increased to the present. The additional ability to produce BeAl based castings has greatly expanded its potential applications. Production, properties, current and future uses will be discussed.

9:10 AM
The Public Health Risks of Commercially-Used Beryllium: *Henry H. Willis*[1]; H. Keith Florig[1]; Benoit Morel[1]; [1]Carnegie Mellon University, Dept. of Eng. and Public Policy, Pittsburgh, PA 15213-3890 USA

Decades of studies on beryllium workers have shown that inhalation of airborne beryllium particles can permanently impair lung function in some individuals. Further, some animal experiments suggest that beryllium might also induce lung cancer. Beryllium and its compounds, once used primarily in defense and aerospace applications, are now finding application in a number of commercial products including consumer electronics, automobiles, and even golf clubs. As beryllium enters wider commerce, it is prudent to ask what risks might be posed to public health by expanding manufacturing and use, and how those risks compare to risks from substitute materials. We address these questions across the entire product life cycle, considering risks from manufacturing, use, recycle, and disposal of specific products. The task is complicated by uncertainties in the types of future uses, uncertainties in the exposure potential from any given use, and uncertainties in the hazard potential of beryllium at very low exposures. Despite these difficulties, we draw some general conclusions about the applications and life-cycle phases likely to present the greatest individual and population risks.

9:40 AM Break

9:50 AM
Discovery of New Human Lymphocyte Activation (HLA) Alleles Associated with Chronic Beryllium Disease: Zaolin Wang[1]; P. Scott White[1]; Owatha L. Tatum[1]; *Babetta L. Marrone*[1]; [1]Los Alamos National Laboratory, Mail Stop M888, Los Alamos, NM 87545 USA

Beryllium is used in a large number of modern industries because of its unique chemical and physical properties. Inhalation of beryllium dust can cause lung disease. Although acute beryllium disease was reported among the industrialized countries in the 1940's and 1950's, Chronic Beryllium Disease (CBD) is now the major illness caused by beryllium exposure. CBD is a systemic granulomatous lung disorder with a mortality rate of about 30% in CBD patients. The incidence rate of CBD correlates inconsistently with exposure levels. The rate of CBD is highest in certain occupations, for example, machinists. However, CBD has also been found in workers with apparently very low exposure to beryllium, such as in plant secretaries and security guards. This has led to the hypothesis that development of CBD has two risk factors: exposure and individual genetic susceptibility. In 1993, Richeldi et al. (L. Richeldi, R. Sorrentino, C. Saltini, Science 262, 242, 1993) published their findings that CBD was associated with the allelic substitution of a lysine for a glutamic acid in position 69 of the HLA-DPB1 gene. Their results, which was confirmed in our laboratory, indicated that up to 97% of CBD patients had the so-called "Glu69" marker. However, about 30-45% of beryllium-exposed unaffected individuals carried the same marker. Because CBD occurs on average in only 1-6% of exposed workers, the presence of Glu69 did not appear to be the sole genetic factor underlying the disease development. Our aim was to find these unknown factors underlying the individual susceptibility to CBD.

10:20 AM
Overview of the New Beryllium Facility at Los Alamos National Laboratory: *Steve Abeln*[1]; [1]Los Alamos National Laboratory, Mail Stop G-770, Los Alamos, NM 87545 USA

The new facility will be described (place holder).

10:50 AM
The Application of Aluminum Beryllium in Fighter Aircraft Structure: *David Bowden*[1]; R. L. Martin[1]; D. Kaczynski[2]; C. Pokross[2]; G. J. London[3]; [1]The Boeing Company, P.O. Box 516, MC S102-2152, St. Louis, MO 63166-0516 USA; [2]Brush-Wellman Engineered Materials; [3]Naval Air Warfare Center

The use of resin matrix composites in aircraft structure has steadily increased to satisfy the demands for low vehicle weight and increased system performance. Composites are especially applicable in fighter aircraft, where a high percentage of the design is stiffness critical. With the increasing emphasis on system affordability, the availability of a relatively low cost sheet metal having high specific modulus to replace more expensive composites could result in significant life cycle cost savings, especially for composite components in aging aircraft having high repair and replacement rates. In this paper, we will discuss the application of aluminum-beryllium alloy sheet as an affordable replacement for composite skins in fighter aircraft empennage structure. We will review the advantages offered by aluminum-beryllium, discuss the unique design and manufacturing approaches necessary to successfully implement this

new material in aircraft structure, and demonstrate the application of aluminum-beryllium in a prototype component.

Hydrogen Effects on Materials Behavior: Fundamental Effects

Sponsored by: Structural Materials Division; ASM International: Materials, Science, Critical Technology Section; Corrosion and Environmental Effects Committee
Program Organizers: Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Russell H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; A. W. Thompson, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

Tuesday AM
November 2, 1999
Room: Salon H&I
Location: Omni Netherland Plaza Hotel

Session Chairs: Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Michael I. Baskes, Sandia National Laboratories, Livermore, CA 94551-0969 USA

8:30 AM Introduction

8:35 AM
Structure Dependence of Hydrogen Effects at Grain Boundaries in an FCC Solid: *Richard G. Hoagland*[1]; Michael I. Baskes[2]; [1]Washington State University, School of Mech. and Mats. Eng., Pullman, WA 99164-2920 USA; [2]Sandia National Laboratories, Mats. Reliability Dept., MS 9403, Livermore, CA 94551-0969 USA

A grain boundary may be identified by five macroscopic variables, three that define the relative orientation of the two grains and two more that define the orientation of the local boundary plane (the tangent plane), and by two microscopic variables that define the in-plane translation of one grain relative to the other. Such variables define a periodic structure, although the period may be large in some cases. However, these variables, per se, provide no details about the atomic structure of the boundary. Indeed, the atomic structure can vary dramatically over the boundary ranging from locations where the structure is close-packed to regions of very low coordination. Such variations appear even in so-called special boundaries, boundaries that have particularly low energies. Furthermore, in many boundaries multiple configurations can occur. Therefore, interactions between the grain boundary and point defects and dislocations may also vary dramatically over the boundary. In this paper we explore aspects of interactions between various boundary structures and interstitial hydrogen in nickel via Monte Carlo and molecular statics using the Embedded Atom Method. We also examine the effects of boundary structure and hydrogen on dislocation-boundary interactions. This work was supported by the United States Dept. of Energy, Office of Basic Energy Sciences, Division of Materials Science under Grant DE-FG03-98ER45697 and the USDOE under contract #DE-AC04-94AL85000.

8:55 AM
Hydrogen Effects on True Surface Energies: *William W. Gerberich*[1]; [1]University of Minnesota, Chem. Eng. and Mats. Sci., 421 E. Washington, 151 Amundson Hall, Minnepolis, MN 55455 USA

Indentation of high hardness, high modulus superlayers bonded to a ductile interlayer on a ceramic or semiconductor can provide fracture energies of well-bonded or weak interfaces. Macroscopic modeling of the external strain energy driving force takes advantage of laminate theory and existing analysis of thin film indentation mechanics. By evaluating a number of film thicknesses ranging from 40 to 3000 nm, it is shown that a plateau exists for Cu/Ti in W/Cu/Ti/Si02/Si below a film thickness near 100 nm. This plateau value of 2J/m^2 for the true surface energy is thought to be commensurate with the true surface energy of the interface in any plastic energy dissipation. This value is further lowered to 1 J/m^2 by cathodically charging the Cu/Ti interface with hydrogen.

9:15 AM
Modeling of the Hydrogen Absorption and Evolution Reactions on Iron and the Effect of Additives: *Mahmoud Hassan Abd Elhamid*[1]; B. G. Ateya[1]; H. W. Pickering[1]; [1]Pennsylvania State University, Mats. Sci. and Eng., 209 Steidle Bldg., University Park, PA 16802 USA

The hydrogen evolution and absorption reactions on iron have been gaining increasing attention in view of their industrial importance. Hydrogen is considered as a clean source of energy. Unfortunately, its evolution on metals is sometimes accompanied by loss of mechanical properties and resulting crack propagation known as hydrogen embrittlement (HE). A necessary step leading to HE is the hydrogen absorption reaction (HAR). The HAR occurs as a direct result of the presence of adsorbed hydrogen on the metal surface, which is an intermediate step in the hydrogen evolution reaction (HER). Accordingly, understanding of the surface (adsorption), and bulk (absorption) phenomena is needed for better understanding of HE. The IPZ model is being used to analyze the kinetics of the HER and HAR on iron. This model describes the kinetics of both reactions through a set of simple analytical equations that can be solved for the different thermodynamic parameters (hydrogen surface coverage) and kinetic parameters (rate constants) involved in both reactions. This model is also being used to investigate the kinetics of both the HER and HAR in the presence of inhibitors, for example, benzotriazole (BTA) and site blocking elements such as (iodide ions). Sponsor: U.S. Steel Research.

9:35 AM
Correlations Between Irreversible Hydrogen Trapping and Hydrogen Embrittlement for Different Groups of Alloys: *Bruce G. Pound*[1]; [1]Exponent Failure Analysis Associates, Mech. and Mats., 149 Commonwealth Dr., Menlo Park, CA 94025 USA

Hydrogen trapping in different groups of alloys was investigated using a potentiostatic pulse technique. Three groups of alloys were studied: high-strength steels (AerMet 100, two 18Ni maraging steels, and AISI 4340 and H11), precipitation-hardened alloys (A-286, 718, 925, 2Be-Cu, and an 18Ni steel), and alloy K-500 in various heat-treated conditions. Irreversible trapping constants (k) were obtained for these alloys and compared with the susceptibilities to hydrogen embrittlement (HE) determined in other studies. Each group of alloys exhibited a correlation between k and the observed susceptibility to HE. The order of the k values for the steels inversely paralleled their threshold stress intensities for stress corrosion cracking (KISCC), and 1/k for 4340 steel followed the change in KISCC with yield strength. Likewise, the k values for the precipitation-hardened alloys were consistent with data for the reduction of notch strength. In the case of alloy K-500, the value of k differed markedly with the type of heat treatment and it could be correlated with the reduction of strength for annealed-and-aged and direct-aged alloy.

9:55 AM
Accelerated Embrittlement of High Strength Steels by Cyclic Hydrogen Potential: *Michihiko Nagumo*[1]; Hideyuki Uyama[1]; [1]Wasada University, Dept. Mats. Sci. & Eng., Okubo 3-4-1, Shinjuku 169-8555 Japan

The role of hydrogen on the embrittlement of steels has been examined with respect to the interactions with defects induced by plastic deformation. Delayed fracture test under cathodic charging using cyclically varied current density was conducted on high strength steels, simulating use of steels under alternating natural environment. Reduction of the time to fracture by increasing the alternation frequency was clearly observed. The associated hydrogen behavior was then examined by means of hydrogen thermal des-

orption spectroscopy. The defects acting as the trapping sites of hydrogen that desorbs with a peak at about 150 increased by plastic deformation. The nature of the defects were assigned to be point-like defects, presumably vacancy-clusters, by using hydrogen as a tracer of the defects in the samples subjected to annealing at different temperatures after deformation. The cyclic alternation of the applied hydrogen potential was shown to accelerate the stabilization of the point-like defects that takes place progressively during the delayed fracture test. A new mechanism of hydrogen embrittlement of high strength steels is proposed.

10:15 AM Break

10:25 AM
Hydrogen Induced Shear Localization: *Petros Sofronis*[1]; Nikolaos Aravas[2]; [1]University of Illinois, Theoretical and Applied Mech., 216 Talbot Lab., 104 South Wright St., Urbana, IL 61801 USA; [2]University of Thessaly, Mech. and Indus. Eng., Pedion Areos, Volos 38334 Greece

It is well known that hydrogen enhanced localized plasticity (HELP) is a viable mechanism for hydrogen embrittlement supported by experimental observations. The objective of this work is to reveal the role of hydrogen in possibly localizing the macroscopic deformation into bands of intense shear from a solid mechanics point of view. The hydrogen effect on material deformation is modeled through the hydrogen induced volume dilatation and the reduction in the local flow stress upon hydrogen dissolution into the lattice. Interstitial hydrogen is assumed to be in equilibrium with the local stress. The analysis of the plastic deformation and the study of the conditions for localization are carried out in plane strain uniaxial tension. The localization criteria employed are those established by the work of Rice and Rudnicki.

10:45 AM
TEM Study of Hydrogen Effects on the Deformation and Fracture of Ni3Al: *Daniel B. Lillig*[1]; Ian M. Robertson[1]; Howard K. Birnbaum[1]; [1]University of Illinois Urbana-Champaign, Dept. of Mats. Sci. and Eng. and Mats. Rsrch. Lab., 1304 West Green St., Urbana, IL 61801 USA

Polycrystalline Ni3Al alloys, produced by rolling and recrystallizing single crystals, suffer significant reductions in ductility with the addition of hydrogen gas and water vapor to the test environment. In situ straining experiments in an environmental cell transmission electron microscope has been used to study the effect of environmental hydrogen on dislocations. Dislocation velocities increase significantly when hydrogen is available from the test environment. Enhanced dislocation velocities have been linked to localized plasticity, which can result in reduced elongation to failure. The role of hydrogen in enhancing dislocation mobility and in promoting edge character dislocations will be discussed.

11:05 AM
TEM Study of Hydrogen Effects on the Deformation and Fracture of Iron Aluminides: *Daniel B. Lillig*[1]; Ian M. Robertson[1]; Howard K. Birnbaum[1]; [1]University of Illinois Urbana-Champaign, Dept. of Mats. Sci. and Eng. and Mats. Rsrch. Lab., 1304 West Green St., Urbana, IL 61801 USA

Removal of hydrogen gas and water vapor from the test environment has been shown to increase the total elongation and to alter the fracture mode of Fe3Al and FeAl alloys with less then 40% Al. In situ straining experiments in an environmental cell TEM allow for the direct observation of dislocation mobility as the test environment is changed. In particular tests in vacuum, hydrogen, dry helium and helium saturated with water vapor will be compared. Dislocation velocities increase significantly when hydrogen is available from the test environment, especially when the source of hydrogen is water vapor. Enhanced dislocation velocities have been linked to localized plasticity, which can result in reduced elongation to failure. The role of hydrogen in enhancing dislocation mobility and in promoting edge character dislocations will be discussed.

11:25 AM
Environmental Embrittlement of Iron Aluminides: *Wann Chiun Luu*[1]; Jiann Kuo Wu[1]; [1]National Taiwan Ocean University, Instit. of Mats. Eng., No.2, Pei Ning Rd., Keelung 20224 ROC

Environmental embrittlement of a series iron aluminides have been systematically evaluated using electrochemical hydrogen permeation measurement, hydrogen microprint technique and slow strain rate tensile test in this study. The results of hydrogen permeation and microprint technique show that £\-disordered solid solution structure of binary and ternary iron aluminides (Fe-10Al, Fe-18Al and Fe-18Al-5Cr) have higher effective diffusivity and permeability than those B2 ordered structure of binary and ternary iron aluminides (Fe-28Al, Fe-28Al-5Cr and Fe-40Al). The slow strain rate tensile test of six iron aluminides in air, oil coating and hydrogen charged were analyzed and concluded. Three£\-disordered solid solution structure of iron aluminides suffer in hydrogen environment which are quite different embrittlement mechanism from those B2 ordered structure of iron aluminides with serious tensile loss in air.

11:45 AM
Examination of Deformation Microstructures in Hydrogen Charged Ni-Base Commercial Alloys: Ram Bajaj[2]; *David Stephen Gelles*[1]; [1]Battelle Pacific Northwest National Laboratory, P8-15, P.O. Box 999, Richland, WA 99352 USA; [2]Bechtel Bettis Inc., P.O. Box 79, Pittsburgh, PA 15122 USA

In an effort to understand the effects of hydrogen on deformation response of Ni-base alloys, tensile specimens were sectioned and examined by transmission electron microscopy to show slip band propagation across grain boundaries. Alloys included X-750 (in two heats), A625, A690, and A600, as well as EN82 and R127 weldments. Specimens without hydrogen and those with ~60 wppm hydrogen were deformed between 1% and 4% plastic strain at strain rates of 1x10-5 or 1x10-7 s-1. Procedures were developed to show slip band structure on both sides of a grain boundary in stereo. Results demonstrated that the mode of deformation without hydrogen present: slip band formation and propagation across grain boundaries, is not significantly altered by the addition of hydrogen. Nor is the stacking fault energy changed in the simpler alloys, A600 and A690, by the addition of hydrogen. The only clear effect of hydrogen appears to be the development of dislocation “debris” immediately adjacent to carbide precipitates at grain boundaries, where boundaries are intersected by slip bands. Examples of the slip band structure observed across grain boundaries will be demonstrated using anaglyph stereo images.

12:05 PM
Hydrogen Embrittlement of Parent and Friction Stir Welded AL 2195 Alloy: *Gan Lu*[1]; E. I. Meletis[1]; [1]Louisiana State University, Mech. Eng. Dept., 2508 CEBA, Baton Rouge, LA 70803 USA

The friction stir welding (FSW) process is being evaluated for application on the Al 2195 cryogenic tank of the Space Transportation System. In the present study, the hydrogen embrittlement response of parent 2195 and 2195 plates joined by FSW was investigated. The parent alloy was tested in the peak-aged (PA) temper and two overcharged conditions and FSW were applied to PA 2195 plates. Static hydrogen charging experiments were first conducted to determine hydrogen intake of the alloy as a function of the aging temper (microstructure) by using x-ray diffraction. Hydrogen embrittlement was assessed by conducting extension rate experiments under cathodic potentiostatic control in 3.5% NaCl aqueous solution. Alloy and weldment microstructures were characterized by transmission electron microscopy and fractographic studies were conducted by scanning electron microscopy. The hydrogen embrittlement behavior is discussed in view of the present and previous results on Al-Li alloys.

New Opportunities in MMC Research and Applications: Automotive and Aeronautical Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee
Program Organizer: Daniel B. Miracle, Wright Laboratory, Materials Directorate, Building 655, WPAFB, OH 45433 USA

Tuesday AM
November 2, 1999

Room: Salon D&E
Location: Omni Netherland Plaza Hotel

Session Chair: Dr. Bill Harrigan, Alyn Corporation, Irvine, CA 92606 USA

8:30 AM Introductory Comments
Dan Miracle, AF Research Laboratory, Materials and Manufacturing Directorate, Wright -Patterson AFB, OH USA

8:40 AM Invited
Need for Discontinuous Reinforced Aluminum in Advanced Aero Structures: *Chris Huskamp*[1]; [1]Boeing, Phantom Works, P.O. Box 516, MS S102-2417, St. Louis, MO 63166 USA

With shrinking defense appropriations, air-frame companies are challenged with the need to produce maximum performance aircraft at a lower cost. With this in mind, structural composites and titanium have been targeted as high-cost materials for which replacements are sought. Discontinuous Reinforced Aluminum (DRA) products are potential replacements for both materials with only a slight degradation in performance balanced by a substantial cost avoidance. However, there is still a need for work that would support a more widespread implementation of DRA's into aero structures. This paper will cover current candidates and avenues for DRA implementation as well as potential applications if key material properties are optimized.

9:10 AM Invited
Particulate-Reinforced Ti: *Walter Zimmer*[1]; [1]Dynamet Inc., Boston, MA USA
Abstract Text Not Available

9:40 AM Invited
Automotive Needs for Particulate MMC's:
To Be Determined

10:10 AM Break

10:30 AM
Opportunities in the Use of 3D Continuous Metallic Fibres as MMCs Reinforcement: Catherine Salmon[1]; *Francis Delannay*[1]; [1]Université Catholique de Louvain, PCIM, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium

3D networks of interconnected fibres can be quite conveniently produced by sintering mats of continuous metallic fibres. The presentation will deal with the processing and mechanical properties of some metal-metal composites made by infiltration of networks of Fe-base or Ni-base fibres with a variety of Al-base or Zn-base alloys. Processing challenges will be enlightened. The influence of interfacial reactivity and network interconnectivity on the tensile and thermal expansion behaviours will be discussed. The pertinence of continuous elasto-plastic models will be assessed. Internal stress measurements by neutron diffraction have been used for studying the role of interfaces and matrix ductility on the macroscopic behaviour. The possible niches of application of these composites will be reviewed.

10:50 AM
Solutionizing Effects on Aging Behavior of 7075 Aluminum Alloy Composites: Shailendra K. Varma[1]; Erika Esquivel[1]; [1]The University of Texas at El Paso, Dept. of Metallu. and Mats. Eng., El Paso, TX 79968-0520 USA

Age hardenable metal matrix composites (MMCs) have been one of the leading candidates for structural applications, especially commercial aluminum alloy matrices reinforced with alumina particles. The effect of solutionizing temperature and time on the aging behavior of 7075 aluminum alloy in the monolithic form has been compared with a composite containing 0.1 volume fraction of alumina particles. The time required to achieve peak hardness values for similar solutionizing conditions during aging will be compared with those obtained in the previous work on 6061 and 2014 aluminum alloys reinforced with similar volume fractions of alumina particles. The microstructures in the underaged, peak hardened, and overaged conditions will be discussed in terms of the GP zone and formation of other kinds of precipitates.

11:10 AM
Solutionizing Effects on Corrosive Wear Behavior of 7075 Aluminum Alloys Composite: *Gustavo Vasquez*[1]; Shailendra K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metallu. and Mats. Eng., El Paso, TX 79968-0520 USA

A scratch test has been used to determine the corrosive wear behavior of a 7075 aluminum alloy both in its monolithic form as well as composite reinforced with alumina particles. Effect of solutionizing time and temperature on the corrosive wear has been determined by measuring the transient current generated as a result of producing a bare new surface exposed to the electrolyte by dropping a stylus, containing a Vickers hardness indenter on its tip, on to the surface of rotating cylindrical electrode. Near surface microstructures have been observed by TEM and a correlation between the solutionizing parameters and transient current will be established.

11:30 AM
Hot Deformation and Extrusion Modeling of Particulate Matrix Composites: *Hugh J. McQueen*[1]; E. V. Konopleva[1]; [1]Concordia University, Dept. of Mech.Eng., Montreal, Quebec H3G 1M8 Canada

Particulate metal matrix composites (PMMC) have more likelihood of industrial application than metal matrix fiber composites, which suffer high probability of damage to the fine, high modulus, ceramic fibers during fabrication. Moreover, PMMC, inexpensively fabricated into large billets by liquid metal mixing or powder metallurgy, provide significant improvement in strength, modulus, and resistance to fatigue and wear without sacrificing density. The final mass fabrication of components depends on mechanical forming, notably at high temperatures to minimize cracking and particle-matrix degradation. A hot torsion project on alloys 6061, A356, 7075 and 2618 with 10 to 20% of Al_2O_3 or SiC determined hot strength and ductility, constitutive constants and microstructural evolution. The substructure is very heterogeneous exhibiting regions of (i) subgrains (less recovered than the bulk alloy), (ii) non-cellular dense clouds of dislocations, and (iii) small highly misoriented cells which could be dynamic recrystallization nuclei (however they do not appear to grow). The modeling of extrusion of the various particulate metal matrix composites will be reported and their relative extrudability compared.

Pb Free and Pb Bearing Solders: Session I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Jessica H. B. Hemond, AMP Inc., MS 018-020, Harrisburg, PA 17105-3608 USA; K. N. Subramanian, Michigan State University, Department of Materials Science & Mechanics, East Lansing, MI 48824-1226 USA

Tuesday AM — Room: Salon B&C
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: K. N. Subramanian, Michigan State University, Dept. of Mat. Sci. & Mech., East Lansing, MI 48824-1226 USA

8:30 AM

Properties and Performance of Sn-Ag-Bi Solder for Electronics Applications: *Paul T. Vianco*[1]; Jerome A. Rejent[1]; [1]Sandia National Laboratories, Org. 1833, P.O. Box 5800, MS1411, Albuquerque, NM 87185-1411 USA

The results of a study on the 91.84Sn-3.33Ag-4.83Bi (wt.%) solder for use in electronics applications is presented. The solder exhibited satisfactory solderability performance when compared to Sn-Pb alloys. The shear strength of the Sn-Ag-Bi alloy is nearly twice that of the Sn-Pb eutectic solder. It was observed that Pb contamination of the solder weakened its strength as Bi combined with the Pb tramp element. The solder was evaluated on prototype electronic solder joints (surface mount and through hole). Surface mount solder joints were exposed to 10000 thermal cycles (0°C-100°C). Following 10000 cycles, the Sn-Ag-Bi solder exhibited microstructural damage, but without a loss of solder joint integrity. Fillet lifting of through hole solder joints was observed in prototype units. The implication of these results towards the application of this solder alloy in engineering applications will be discussed. Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Company, for the US Dept. of Energy under Contract DE-AC04-94AL85000.

8:55 AM

The Effect of Pb Contamination from Pretinned Leads/Pads on Solidification Behavior of Pb-Free Solders: K. W. Moon[1]; U. R. Kattner[1]; *William J. Boettinger*[1]; C. A. Handwerker[1]; [1]NIST, Metallu. Div. Mats. Sci. and Eng. Lab., 100 Bureau Dr., Stop 8555, Gaithersburg, MD 20899-8555 USA

The effect of Pb contamination on the solidification path of various Pb-free solders is examined. This situation arises from the possibility that Pb-free solders may be used with leads and/or pads that have been pretinned with Pb-Sn eutectic solder. Estimates of Pb contents as high as 8 wt% are obtained using typical solder volumes and pretinning thicknesses. Using a thermodynamic assessment and computation of solder phase diagrams, Scheil calculations reveal the possible Pb levels that can lead to the formation of low temperature ternary eutectics. For example in the widely considered Sn-Bi solders, a ternary eutectic occurs in the Sn-Bi-Pb system at 100°C. The effects of Pb contamination on several other Pb-free solders will also be examined.

9:15 AM

Observation of Fillet Lifting in Sn-Bi Alloys: *William J. Boettinger*[1]; C. A. Handwerker[1]; B. Newbury[1]; T. Y. Pan[2]; [1]NIST, Metallu. Div. Mats. Sci. & Eng. Lab., 100 Bureau Dr., Stop 8555, Gaithersburg, MD 20899-8555 USA; [2]Ford Motor Company, 2000 Rotunda Dr., Dearborn, MI 48121-2053 USA

The probability of fillet lifiting in through-hole solder joints was determined for a series of seven binary Sn-Bi solders with compositions between 2.5 and 70 wt% Bi. Three printed circuit board thicknesses and two pad sizes were examined using a laboratory scale drag soldering process. Composition has the largest effect on failure with a peak in lift-off probability at ~10wt% Bi. In situ observation as well as microstructural analysis indicates that fillet lifting occurs during solidification. The solidification of these alloys is analyszed using a Scheil analysis and the tendency for fillet lifiting is related to a hot tearing criterion orginally developed for castings.

9:35 AM

Microstructural Characterization of Reflowed and Aged Composite Solders with Cu and Ag Particulate Reinforcement: *Fu Guo*[1]; S. Choi[1]; J. P. Lucas[1]; K. N. Subramanian[1]; T. R. Bieler[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824 USA

Composite solders were prepared by dispersing 5 micron size Cu and Ag particles (10, 15, 20 volume percent) into the eutectic Sn-Ag solder matrix. Two different processing methods were used to prepare the composite solders: (i) blending the powdered particles with solder paste, and (ii) adding the particles to the molten solder at 280 degrees Celsius. The composite solders were characterized by studying the morphology, size and distribution of the reinforcing phase. Microstructural features were studied using optical and scanning electron microscopy, and energy dispersive x-ray analysis. The effect of reflow and isothermal aging on the microstructure of the composite solders were compared with those on corresponding non-composite solders. The effect of mechanical working and subsequent reflow on redistribution of the reinforcing phase was investigated with the composite solder containing copper particulate.

9:55 AM

Liquid Density Gradients during Liquid-Phase Sintering: Their Effect on Solid-Phase Settling in Pb-Sn Alloys: *Yijun Du*[1]; Shu Zu Lu[1]; Thomas H. Courtney[1]; [1]Michigan Technological University, Dept. of Metallu. and Matls. Eng., Houghton, MI 49931 USA

Solid phase settling takes place in isothermally heat-treated interconnected liquid-solid mixtures; the phenonmenon is common to liquid-phase sintered materials, for example. If the solid phase density exceeds that of the liquid, the settling results in an interconnected solid-liquid mixture (with a solid content greater than that of the overall alloy) near the bottom of such a heat-treated material. The top of the sample is a single phase liquid at the heat-treatment temperature. Although gravity is the driving force for this settling, the rate-controlling mechanisms for it have not yet been fully identified. We have investigated settling in Pb-Sn interconnected solid-liquid mixtures in an attempt to delineate these mechanisms. In the course of this work, we have come across an unusual phenomenon. In particular, a concentration gradient in the liquid develops during heat-treatment. The net effect of this gradient is to "pump" Sn upward within the liquid, and to do the opposite for Pb. The resulting "distillation" leads to interesting microstructural development. For example, the total solid Pb content in the material decreases with sintering time. We have characterized (at least semi-quantitatively) the extent of the liquid concentration gradient and its main features as they depend on, for example, sintering temperature and overall alloy composition. These results will be presented, and explained as best possible. This work was supported by the National Aeronautics and Space Administration.

10:15 AM BREAK

10:25 AM

A Comparative Study of Interfacial Reaction Between Ag-Sn, Bi-Sn, Pb-Sn Eutectic Solders and Pd/Ni/Cu Metallization: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Mat. Sci. Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Even though Pb-Sn eutectic solder is widely used in electronic industry, due to various reasons there is a growing interest in the use of Pb-free solders. Furthermore, electronic packaging paradigm requires solders having a heirarchy of melting points. The intermetallic formation at the solder/susbtrate interface, both during pro-

cessing and in service, play a key role in determining the mechanical property and reliability of solder joints. As the solder joint size decreases and the service temperature of the advanced products increases, it is necessary to understand and identify the key factors governing the interfacial reaction kinetics. Metallization schemes using Ni and Pd are becoming increasingly popular in electronic packaging. We will present a systematic study of interfacial reaction between Pb-Sn, Ag-Sn, Bi-Sn eutectic solders and Pd/Ni/Cu metallization scheme. The evolution of microstructures due to both liquid-and solid-state reactions will be presented. The products of interfacial reaction are characterized by SEM, TEM, and AEM. The interfacial microstructures will be discussed in terms of diffusion paths using the calculated isothermal sections of the relevant ternary system.

10:50 AM
Formation and Growth of Intermetallic Layers of Eutectic Sn-Ag Solders and Their Composite Solders on Cu Substrate during Isothermal Long-Term Aging: *Sunglak Choi*[1]; J. P. Lucas[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA

Solders exhibit a good bonding to substrates when the intermetallic layers form between them. The intermetallic interfacial layers grow by solid state diffusion and over sufficient time, occupy significant portion of the solder joint. As a result, the growth of the intermetallic layers weakens the mechanical properties of the solder joint and adversely affects its reliability due to the brittle nature of the intermetallic layer. Thus, consideration must be given to limiting the growth of the intermetallic layers present in the solder joint to improve reliability. Single shear lap joints were made with eutectic Sn-Ag solders, and their composites containing about 20 vol% in-situ FeSn2 and Ni3Sn4 intermetallic phases. Aging experiments were performed with solder joints at five different temperatures of 70, 100, 120, 150, 180°C for 4000 hours. The intermetallic interfacial layers between solders and the Cu substrate were examined using optical microscopy and scanning electron microscopy. The growth kinetics of the intermetallic interfacial layers was characterized. Effects of in-situ intermetallic phases on the growth rate were also characterized and compared with respect to the effects of different intermetallic phases such as Cu6Sn5.

11:10 AM
Effects of Small Additions of Cu on the Static and Cyclic Aging Behavior of Eutectic Sn-Ag Solder: *Jeff D. Sigelko*[1]; S. Choi[1]; J. P. Lucas[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824 USA

To understand the role of small alloying additions on the aging kinetics of eutectic Sn-Ag solder, studies were carried out using static and cyclic thermal aging with solder joints representative of electronic interconnects. These joints were prepared by soldering dog-bone shaped copper strips using commercially available solder pastes. Static aging was carried out at various temperatures and times ranging to 4,000 hours. Investigations were also carried out using bulk solder specimens, and compared with those of solder joints to evaluate the role of geometric constraints imposed by the substrates on aging kinetics. Cyclic aging of solder joints was also carried out to understand the role of stress-assisted diffusion on the aging process.

11:30 AM
Intermetallic Compound Formation between Ni and Eutectic BiSn Solder: C. Chen[1]; C. E. Ho[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Chem. Eng., Chungli, Taiwan

The reaction between Ni and the eutectic BiSn solder was studied. Bismuth is an important element in many electronic solders, while Ni is used in many printed circuit board surface finishes. According to the relevant binary phase diagrams, there are intermetallic compounds in both the Ni-Sn system (Ni3Sn, Ni3Sn2, and Ni3Sn4) and the Ni-Bi system (NiBi and NiBi3). It was found that the only intermetallic compound formed was Ni3Sn4. The other intermetallic compounds was not observed. The reactions of Ni with solid BiSn and liquid BiSn produced very different microstructures. Reaction of Ni with the liquid BiSn produced a thick reaction zone, which is a two-phase mixture of Ni3Sn4 needles dispersed in the BiSn matrix. Reaction of Ni with the solid BiSn produced a smooth layer of Ni3Sn4. The detailed reaction product morphologies and the reaction mechanisms for both types of reactions were discussed.

Processing and Properties of Structural Nanomaterials I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Powder Materials Committee, Alloy Phases Committee, Materials Processing & Manufacturing Division, ASM-MSCTS, Materials & Processing Committee

Program Organizers: Leon L. Shaw, University of Connecticut, Department of Metallurgy & Materials Engineering, Storrs, CT 06269-3136 USA; Lawrence T. Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; Carl C. Koch, North Carolina State University, Department of Materials Science & Engineering, Raleigh, NC 27695 USA

Tuesday AM
November 2, 1999

Room: Rookwood
Location: Omni Netherland Plaza Hotel

Session Chairs: Leon L. Shaw, University of Connecticut, Metall. and Mats. Eng., Storrs, CT 06269 USA; David G. Morris, CENIM, Dept. of Phys. Metall., Madrid 28040 Spain

8:30 AM Invited
Changes of Hardness, Strength, Ductility and Toughness of FeAl and TiAl: *D. G. Morris*[1]; [1]CENIM, CSIC, Dept. of Physical Metallu., Avenida Gregorio del Amo 8, Madrid 28040 Spain

Bulk samples of FeAl and TiAl have been prepared by mechanical milling or alloying to powders with nanometre grain size followed by high pressure hot pressing. Mechanical behaviour has been evaluated in a number of ways-hardness indentation, compression and bend testing, and fracture toughness testing on notched samples. Increased hardening is typically found down to grain sizes of about 30nm. Bend strength and fracture toughness remain essentially constant for FeAl down to a grain size of about 50nm, before falling at finer grain sizes. For TiAl the fracture toughness is found to fall from much larger (micron) grain sizes. Mechanical behaviour is discussed in terms of the intrinsic behaviour of the intermetallic with different grain sizes, and the influence of remnant porosity and internal flaws left from material processing.

9:00 AM Invited
Mechanical Behavior of Nano-TiO2 Filled Polymer Composites: *Linda S. Schadler*[1]; Richard W. Siegel[1]; Chek B. Ng[1]; Rebecca Webber[1]; Peter Hess[1]; Brian C. Benicewicz[1]; [1]Rensselaer Polytechnic Institute, Mats. Sci. and Eng., 110 Eighth St., Troy, NY 12180-3590 USA

The properties of nanoparticle filled polymer systems are unique. For example, the modulus and strain to failure can be increased simultaneously which is unheard of in traditional filled polymers. The unique properties are the result of the polymer chain interacting with the nanoparticle oxide surface, and the excellent dispersion of these nanoparticles. When a polymer chain interacts with a surface, the mobility of the polymer chain is reduced creating a bound polymer layer (BPL). In traditional composites, this BPL is an insignificant volume fraction of the composite. For nanoparticle filled polymers, the BPL volume fraction is of the same order as the particle volume fraction. This leads to two types of reinforce-

ment: load bearing and reduction in matrix chain mobility. This paper will report the results of a study on nano-TiO2 filled epoxy and PMMA. We have found significant improvements in dimensional stability, and glass transition temperature, and simultaneous improvements in ductility, modulus, and scratch resistance.

9:30 AM

Synthesis, Characterization and Comparison of a Consolidated Nanostructured Al-Mg-Li Ternary Alloy Versus an Al-Mg Binary Alloy: *Michael S. Ice*[1]; Enrique J. Lavernia[1]; [1]University of California, Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA

The field of nanocrystalline materials has received much interest in recent years due to their unique properties. By achieving a nanocrystalline microstructure many of the mechanical properties of alloy systems can be significantly improved. In this comparison study, two systems, a spray atomized Al-Mg-Li powder and a binary Al-Mg powder, were mechanically milled under liquid nitrogen (cryomilled), to produce a nanocrystalline microstructure. These powders were degassed and consolidated utilizing hot isostatic pressing and extrusion processes. The grain size of each system was measured by both X-ray diffraction (XRD) and transmission electron microscopy (TEM) following the consolidation and extrusion steps. Density, hardness, ultimate tensile strength, yield strength, and elongation were measured each on the extruded samples. The objective of the present study is to determine the effects of the addition of Li on the mechanical properties of the consolidated Al-Mg alloy. Speculation finds that the extruded samples containing Li will exhibit increased strength and hardness with a decrease in density however the addition of Li may also cause embrittlement, which would decrease the ductility of the material.

9:55 AM

Microstructure/Mechanical Properties Relationship in Ni/Cu Nanolaminates: *Fereshteh Ebrahimi*[1]; Dan Kong[1]; Alirio J. Liscano[1]; [1]University of Florida, Mats. Sci. and Eng., 221 MAE, P.O. Box 116400, Gainesville, FL 32611 USA

Electrodeposition is a viable method for producing laminated metallic structures with nano-size layer thickness values. While coatings (<10 micrometer thick) of these materials can be processed relatively easily, the formation of nodules plagues thicker sections which are desirable for electroforming of structural components. In this study Ni/Cu multilayers with various layer thickness values were produced by electrodeposition. The microstructure of the nanolaminates were characterized using cross-sectional TEM, SEM, and x-ray diffraction techniques. Tensile testing was conducted for evaluating the mechanical properties. The results of this study reveal that the quality of the deposits, the existence of twinning in the microstructure, and continuity and straightness of the layers influence the deformation and fracture of the electrodeposited Ni/Cu nanolaminates significantly.

10:20 AM Break

10:40 AM Invited

Strong Nanocomposite Ceramics with Multiple Functionality: *Koichi Niihara*[1]; [1]Osaka University, ISIR (Sanken), 8-1 Mihogaoka, Ibaraki 567-0047 Japan

Silicon nitride (Si3N4) and silicon carbide (SiC) ceramics have a number of attractive properties such as high strength, excellent toughness, high wear resistance and relatively high thermal shock resistance, but the thermal shock fracture resistance and machinability are still not sufficient for wide applications in components for automobiles, refractory nozzles, tubes, crucibles for various molten metals, etc. The hexagonal BN (h-BN) consisting of layered structure like graphite and has numerous interesting properties such as good high temperature mechanical properties, anisotropic thermal expansion, chemical inertness and high thermal conductivity. However, h-BN ceramics are low fracture strength and YoungÕs modulus, despite of high thermal shock resistance and good machinability. Addition of h-BN, therefore, will be expected to remarkably decrease the fracture strength of Si3N4 with an increase in h-BN content, although thermal shock fracture resistance will be enhanced. Niihara and his colleagues have investigated nanocomposites in which the nano-sized particulates are dispersed within the matrix grains and/or at the grain boundaries, and revealed that the dispersion of nano-sized particulates significantly improves the mechanical properties of oxide and non-oxide ceramic materials even at high temperatures. In early nanocomposites, hard/strong materials were mainly incorporated into the matrix materials as dispersoids. In recent investigations, soft/weak materials like metals were also used as dispersoids, and the enhancement of fracture strength by the addition of even soft/weak dispersoids was observed. However, it has not been reported on the improvement of mechanical properties by nano-sized soft/weak ceramic dispersions such as h-BN. Beside, Si3N4/BN and SiC/BN nanocomposite is expected to show not only higher thermal shock resistance but also good machinability with an increase in h-BN content due to cleavage characteristics of h-BN. In this study, Si3N4/BN and SiC/BN nanocomposites were fabricated through unique chemical processes, and effects of nano-sized BN on microstructure and then thermal and mechanical properties were investigated. From this investigation, it was found that the nano-sized BN dispersion could improve the fracture strength, fracture toughness and high temperature properties of Si3N4 and SiC ceramics. In addition, Si3N4/BN and SiC/BN nanocomposites were found to show strongly improved chemical inertness to molten metals, thermal shock fracture resistance and machinability like metals. Special emphasis will be placed on the understanding of the roles of nano-sized BN dispersions in developing the multi-functional ceramics.

11:10 AM Invited

Nanoceramic Toys: *Merrilea J. Mayo*[1]; Rajendra Nath Basu[1]; [1]Pennsylvania State University, Dept. Mats. Sci. & Eng., Rm. 115 Steidle, University Park, PA 16803 USA

This presentation explores a number of emerging applications of nanocrystalline ceramics, both the frivolous and the functional. The processing method appropriate to each application is discussed, along with the special challenges encountered as the size scale of the particles drops below the submicron regime. Dry press and sinter approaches are used to make bulk superplastic ceramics that can be deformed into exotic shapes, as well as high fracture toughness ceramics (KIC>15 MPam^1/2). Filter pressing is used to fabricate superplastic sheet objects. Electrophoretic deposition (EPD) is used to make large area, microns-thick films that are completely free-standing, as well as adherent electrolyte layers for solid oxide fuel cells. EPD is also used to manufacture patterned ceramic films. Finally, the use of nanocrystalline and submicron ceramics in pressureless diffusion bonding is demonstrated, an operation in which hermetic ceramic-ceramic joints are created using a simple furnace bake.

11:35 AM

Creep Behavior of Nanocrystalline Nickel at 290 and 373K: W. M. Yin[1]; *S. H. Whang*[1]; R. Mirshams[2]; C. H. Xiao[2]; [1]Polytechnic University, Dept. of Mech. Eng., Six Metrotech Ctr., Brooklyn, NY 11201 USA; [2]Southern University and A&M College, Dept. of Mech. Eng., P.O. Box 9987, Baton Rouge, LA 70813 USA

The unparalleled high strength of nanostructured nickel at room temperature is strikingly contrasted with its room temperature creep. It is important to understand the role of creep and its mechanism; and in specific, the nature of plastic deformation in this material. In this paper, we will report some of the results from the investigation on creep behavior of this material at above room temperature. The uniaxial tensile creep behavior of full-dense nanocrystalline nickel with 30nm grains produced by electrodeposition processing has been investigated under constant and stepped load conditions at room temperature and 373K from 500 to 1100 MPa. The experimental results showed a significant creep deformation occurred even at room temperature under initial applied

stress of 600 MPa or higher. The creep resistance was very sensitive to test temperature. For example, the minimum strain rate at 373K was an order of magnitude larger than that at room temperature. The grain size and microstructure of as-received and postcrept specimens have been characterized by conventional TEM techniques regarding grain size and shape. An attempt will be made in order to explain the behavior and discussed about possible creep mechanism based on current findings.

12:00 PM
Tensile and Fracture Behavior in Nickel and Nickel Carbon Doped Nanocrystalline: *Reza A. Mirshams*[1]; Cheng H. Xiao[1]; Sung H. Whang[2]; Wei M. Yin[2]; [1]Southern University and A&M College, Dept. of Mech. Eng., P.O. Box 9987, Baton Rouge, LA 70813 USA; [2]Polytechnic University, Dept. of Mech. Eng., 6 Metrotech Ctr., Brooklyn, NY 11201 USA

The potential engineering applications of nanocrystalline materials need more detailed study on deformation and fracture mechanisms at room and elevated temperature under tensile loading. This paper reports results of a series of experiments were carried out on nickel and carbon doped nanocrystalline nickel with different amount carbon concentrate from 250 to 1000 ppm at room to 373 K. Initial results on pure nickel in nano (30 nm) and conventional grain size indicated a significant effect of grain size on yield and tensile strength. Tensile test experiments in two different strain rates indicated no significant strain rate sensitivity for pure Ni-nano grain size. However, amount of elongation was small and it was not changed considerably. Experiments at 373 K showed the tensile properties of carbon doped nickel in nano scale reduced with addition of carbon from 250 to 1000 ppm. Further examination is under way to determine effects of various grain sizes and texture on tensile behavior and fracture toughness. The results will be discussed in relation to the microstructural characteristics by using TEM and SEM.

P/M: Current Research and Industrial Practices: Powder Making and Processing

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee
Program Organizers: F. D. S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metal Engineering, Rapid City, SD 57701-3995 USA; Enrique V. Barrera, Rice University, Metal Engineering & Materials Science Department, Houston, TX 77251 USA; G. M. Janowski, University of Alabama, Department of Materials & Mechanical Engineering, Birmingham, AL 35294-4461 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM	Room: Caprice 2&3
November 2, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Enrique V. Barrera, Rice University, Mech. Eng. and Mats. Sci., Houston, TX 77005-1892 USA; James W. Sears, Lockheed Martin, Building D2, Schenectady, NY USA

8:30 AM OPENING REMARKS AND INTRODUCTIONS: Fernand Marquis, Primary Organizer and Chair, Powder Materials Committee

8:40 AM Keynote
Advances in Gas Atomization Processing Efficiency and Control: *Iver E. Anderson*[1]; R. L. Terpstra[1]; J. Ting[2]; C. Yu[3]; [1]Iowa State University, Ames Lab., Ames, IA 50011 USA; [2]Crucible Research, Pittsburgh, PA 15205 USA; [3]Delavan, West Des Moines, IA 52065 USA

Many advanced technologies based on particulate materials demand the availability of fine powders. High pressure gas atomization (HPGA), a close coupled discrete jet atomization method, has proved to be one of the most effective methods of producing rapidly solidified fine powders with high yields less than 20 microns. Development of HPGA jets with convergent-divergent (C-D) rocket nozzle designs that have high total kinetic energy was conducted to increase atomization efficiency and uniformity. Efficient use of C-D jets in an atomization nozzle required development of a physical model of the gas dynamics in the atomization zone. An unexpected wide pressure range, advantageous for powder size control. Different aspects of this work were supported by USDOE-BES under contract number W-7405-Eng-82 and the Center for Advanced Technology under DOC contract number ITA81-02.

9:15 AM Invited
Gas Atomization into a Sub-Atmospheric Chamber: S. P. Mates[1]; *F. S. Biancaniello*[1]; S. D. Ridder[1]; P. I. Espina[1]; [1]National Institute Standards and Technology, Indust. Bldg. Rm. 156, Gaithersburg, MD 20899 USA

The physical advantages of atomizing into a sub-atmospheric chamber are discussed. Experimental evidence suggests that the length of the supersonic gas jet produced by a close-coupled nozzle has a strong influence on atomization performance and particle size. Longer supersonic jets can produce a smaller average particle size because they provide sustained lengths of large dynamic pressures necessary to energetically atomize metal droplets. Further, gas dynamic theory shows that reducing the chamber pressure can increase the supersonic length while maintaining a constant gas flow rate, potentially improving atomization conditions without increasing gas consumption. Experiments are performed to assess the benefits of atomizing into a sub-atmospheric chamber.

9:45 AM Invited
Melt Atomization with Energetic Fluids: *K. P. Cooper*[1]; [1]Naval Research Laboratory, Mats. Sci. & Tech., Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375-5343 USA

Very fine (<10 μm) metal powders are used in net shape forming via MIM or hot extrusion; electronic components, such as, fine-pitch interconnects; and as fillers in magnetic recording tapes, conducting tapes, capacitative tapes and EMI shielding. Fine metal powders can be produced by maximizing the energy required to overcome surface tension forces thereby increasing the melt surface area. Several melt atomization processes have been developed to achieve maximum energy transfer conditions. We have developed a two-fluid, rapidly spinning dish process. This two-stage atomizer achieves particle size refinement by the combined effects of impact atomization and spinning dish atomization. In this process, an impactor-generated melt spray is directed into a film of oil on the surface of a rapidly spinning dish. The oil film shears the spray droplets into finer particles. One problem we observed was the loss of a significant fraction of powder as deposits on the chamber walls. We studied the effect of rotational speeds and rotational direction on the mean size and distribution of tin powder. As expected, the finest particle size was obtained with both impactor and dish spun at high speeds and in the counter-rotating mode. Under these conditions, a mean particle size of 7 μm was achieved with tin. The finer size fraction powder particles were spherical, smooth and without satellites, while the coarser particles had a dimpled surface. Two melt breakup mechanisms were identified: One, for small particles, involved a) formation of electron-transparent flakes from shear forces, b) flake edge curling, c) filament formation, d) Rayleigh-Taylor instabilities on filament and e) spheroidization. The other, for large particles, involved a) formation of surface instabilities due to their relative motion with the spinning oil and b) freezing of dimpled melt prior to its breakdown into finer droplets.

10:15 AM Break

10:30 AM Invited
Optimization of an Annular Jet Commercial Gas-Metal Atomizer: G. J. DelCorso[1]; F. S. Biancaniello[1]; *S. D. Ridder*[1]; A. Johnson[1]; P. I. Espina[1]; [1]United States Department of Commerce, Nat. Insti. of Stnds. and Tech., 100 Bureau Dr. Stop 8556, Gaithersburg, MD 20899-8556 USA

The performance of a commercial gas-metal atomizer was studied using a number of previously published research techniques. Initially the flow was visualized via schlieren photography to determine the location of important flow features (e.g., shock waves, expansion fans, shear layers). With this information at hand, an experiment was designed to determine the aspiration performance of two prototype geometries. The aspiration results were confirmed with the help of computational fluid dynamic models, which although not as accurate, yielded more spatial resolution of the phenomena at hand. Using these results, a number of 316 SS production runs were performed, and it was concluded that the recommendations suggested by the fluid experiments produced improvements in the fine powder yield of the considered geometries.

11:00 AM
Benefits of Gas Atomization Processing of Metal Hydrides: *M. L. Anderson*[1]; Jason Ting[2]; Iver E. Anderson[3]; [1]Ames Laboratory, Metallu. and Ceramics, 222 Metals Dev., Ames, IA 50011 USA; [2]Crucible Research, 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA; [3]Ames Laboratory, Metallu. and Ceramics, 126 Metals Dev., Ames, IA 50011 USA

Certain types of intermetallic compounds can reversibly absorb and desorb hydrogen by forming crystalline hydrides. These materials have found uses in nickel-metal hydride batteries and as a mechanism for the safe storage of hydrogen, in devices such as cryocoolers. Traditionally, the processing method used to make powders of these materials is by chill casting and crushing the material. This can lead to some undesirable properties such as inconsistent compositions between particles induced by solidification segregation, angular powders that flow and pack poorly, and unwanted impurities from the crushing process. One process that can alleviate these problems is high pressure gas atomization (HPGA). By atomizing the material, spherical powders with consistent compositions can be created. Any phase segregation present in the particles is easily homogenized with a short (less than one hour) anneal step. Atomization also allows for improved post-powder production processing such as surface treatment, electrode consolidation, and heat exchanger bed filling due to the improved powder flow and apparent density. Support for this study is provided by Materials Science Division of DOE/BES under contract W-7405-Eng-82.

11:30 AM
Mechanical Alloying of Ti-Based MMC Alloys by the Zoz Attritor: *S. Ozbilen*[1]; Cemil Cetinkaya[1]; [1]Gazi Technical University, Metallu. Eng., Teknikokullar, Ankara Turkey

Ti-based advanced class materials systems such as Ti-aluminides, Ti-MMC's and CMC's, and Ti-Al-X alloy powders were prepared by the Zoz attritor under controlled atmospheres and processing times. Processed powders by mechanical alloying were characterised by XRD and SEM investigation. High yield in the MA processed powders were discussed and compared with those processed by other types of attritors and mills.

Refractory Metals & Alloys: A Symposium on Research, Development & Applications III: Mo and Its Alloys

Sponsored by: Structural Materials Division, Refractory Metals Committee, Corrosion and Environmental Effects Committee
Program Organizers: Mehmet Uz, Lafayette College, Chemical Engineering, Easton, PA 18042-1775 USA; Ken Natesan, Argonne National Laboratory, ET/212, Argonne, IL 60439-4838 USA

Tuesday AM — Room: Caprice 1&4
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Ken Natesan, Argonne National Laboratory, Energy Technology/Corrosion, Argonne, IL 60439 USA; P. R. Subramanian, UES Inc., Mats. Res. & Dev., Dayton, OH USA

8:30 AM Invited
Observations and Challenges in Development of Refractory Metal-Silicide Multiphase Alloys for Structural Applications: *P. R. Subramanian*[1]; M. G. Mendiratta[1]; D. M. Dimiduk[2]; T. A. Parthasarathy[1]; [1]UES Inc., Mats. and Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Mats. and Manufact. Directorate, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Studies to-date have shown that Nb-and Mo-base metal-silicide multiphase systems may be viable for service at temperatures above those of nickel-base superalloys in structural applications. Exploratory efforts on these systems have demonstrated the potential for a balance of mechanical properties as well as environmental tolerance. The alloy systems specifically under investigation belong to two classes of materials. One class of materials consists of Nb-Ti-Cr-Si-base alloy systems, where a (Nb,Ti) metal phase is in equilibrium with one or more silicide intermetallics as well as a Cr2Nb-base Laves phase. The other class is composed of Mo-Si-B base systems, where a Mo metal phase in equilibrium with one or more Mo-silicides and a borosilicide phase. This presentation concentrates primarily on research progress in the Nb-base systems with focus on alloying strategies and microstructural development. Highlights of developments in the Mo-base systems are also presented. One of the key challenges in these systems is improving their oxidation behavior under both static and cyclic conditions. The two classes of alloy systems differ significantly in the underlying oxidation mechanisms, and consequently, methods to extend the oxidative lifetime are unique to each class. These issues are discussed with reference to specific examples from each of the alloy classes. Oxidation-protection coatings uniquely tailored to the Nb-and Mo-base systems are also under investigation.

9:05 AM Invited
Phase Stability and Mechanical Properties of Mo(Si,M)2/T(Si,M)2 Two-Phase Alloys (T: V, Cr, Nb and Ta, M: Al, Ge and Ga): *Naohisa Okamoto*[1]; Fu-Gao Wei[1]; Yoshisato Kimura[2]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Mats. Sci. and Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan; [2]Tokyo Institute of Technology, Precision and Intelligence Lab., 4259 Nagatsuta, Midori-ku, Yokohama 226-8503 Japan

Molybdenum disilicide, $MoSi_2$, is drawing much attention as a material for high temperature structural applications because of its high melting point and relatively low density. However, it has two drawbacks; inadequate room temperature toughness and insufficient elevated temperature strength. One approach to solving these problems is the addition of alloying elements. Most of the studies have focussed on the additions of transition metal elements which substitute for Mo atoms. The two-phase combination of $C11_b$ $MoSi_2$ and C40 TSi_2, where T is V, Cr, Nb and Ta, may have a potential to improve mechanical properties through microstruc-

tural control. We have systematically investigated the phase stability and the relationship between microstructure and mechanical properties of the $MoSi_2/TSi_2$ two-phase alloys in the pseudo binary and ternary $MoSi_2$-TSi_2(-TSi_2) systems. Moreover, we have selected Al, Ge and Ga as alloying elements for the Si site substitution of $MoSi_2$ and TSi_2 to understand the basic strategy of the $MoSi_2$-based alloy design.

9:40 AM
Refractory Metal Silicide Composites for Industrial Applications: Joseph W. Newkirk[1]; *Martin Perez*[1]; [1]University of Missouri-Rolla, Dept. of Metallu. Eng., Rolla, MO 65409 USA

Refractory metal silicides with the formula A_3B can be fabricated with the refractory metal as a second phase, which reduces the brittleness of the composite material and improves toughness. The silicides have very attractive properties for industrial applications due to their corrosion, oxidation, and wear resistance. Mo and Nb silicides have received a lot of attention lately, but silicide based on other refractory metals have not. Efforts to develop refractory silicide composites based on V, Cr, and Ta with attractive properties will be discussed. The possibilities of using some of these composites as bulk materials or coatings will be discussed.

10:00 AM
The Effect of Refractory Metal Substitution on the Stability of Mo(ss) + T2 Two-Phase Field in the Mo-B-Si System: *R. Sakidja*[1]; J. Myers[1]; S. Kim[1]; J. H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 Univ. Ave., Madison, WI 53706 USA
Abstract Text Not Available

10:20 AM Break

10:35 AM Invited
Evaluation of Oxide Dispersion Strengthened (ODS) Molybdenum and Molybdenum-Rhenium Alloys: *Andrew J. Mueller*[1]; Robert Bianco[2]; Robert W. Buckman[3]; [1]Bechtel Bettis Inc., P.O. Box 79, West Mifflin, PA 15122 USA; [2]BF Goodrich Aerospace R&D, Westinghouse R&D Ctr., 9921 Brecksville Rd., Brecksville, OH 44141-3289 USA; [3]Refractory Metals Technology, P.O. Box 10055, Pittsburgh, PA 15236-0415 USA

At 1600EC (0.65Tm), the creep-rupture life of molybdenum alloyed with up to 50 weight percent rhenium is increased by approximately an order of magnitude over unalloyed molybdenum. A patented process, developed for producing a fine oxide dispersion in unalloyed molybdenum, results in an increase in creep=rupture life at 0.65Tm of three to five orders of magnitude over unalloyed molybdenum and produce ductile fracture behavior at temperatures below room temperature. A molybdenum-14 wt.%rhenium alloy similarly incorporating an oxide dispersion also exhibits a three to five order increase in creep rupture strength while further lowering the ductile-to-brittle transition temperature significantly below room temperature in both the as-worked and recrystalized conditions. The results of mechanical property testing of both alloys are discussed.

11:10 AM
Solidification Experiments and Thermophysical Properties Measurements on Mo-Re Alloys: *C. Berne*[1]; M. Boiniveau[2]; A. Fontes[3]; A. Pasturel[4]; E. Rolland[1]; I. Tosello[3]; B. Vinet[1]; [1]CEA/CEREM, Département d'Études des Matériaux, 17 rue des Martyrs, Grenoble, Cedex 9 38054 France; [2]CEA, Dépt. de Recherche sur les Matériaux Nucléaires, Centre de Valduc France; [3]CEA/CEREM, Département Procédés et Systèmes Avancés, Centre de Saclay, Cédex 91191 France; [4]CNRS, Lab. de Physique et Modélisation de la Matière Condensée, 25 rue des Martyrs, Grenoble 38042 France

A detailed understanding of phase selection and microstructure formation in undercooled melts is of vital importance for optimising the properties and processing of high performance alloys, especially when rapid solidification is involved during production. Besides, in the context of still growing demand for metallic materials for high temperature use, the refractory metals and alloys come primarily into consideration. In the course of this work, undercooling experiments using a 48-m high drop-tube facility are realized in the Mo-Re system which is homologous to the W-Re system studied recently(2). The coupling of these experiments with ab-initio calculations allows us to identify the candidate (transitory) metastable structures occuring during solidification(3). Microstructure comparisons are also performed by realizing local melting by electron beam on selected compositions. As measurements of thermophysical properties are of a crucial importance for the development of refractory alloys, the submillisecond resistive heating technique(4) (electrical resistivity, volume expension, heat capacity) is applied on commercial Mo-Re compositions. (1) W. Löser, A. Garcia Escorial, B. Vinet: Int. J. Non Equil. Process. 11, 1998, 89. (2) S. Tournier, B. Vinet, A. Pasturel, I. Ansara, P. J. Desré: Phys. Rev. B 57, 1998, 3340. (3) A. Pasturel, B. Vinet: MRS Symposium Proc. Vol 481, 1998, 27. (4) T. Thévenin, L. Arles, M. Boivineau, J. M. Vermeulen: Int. J. of Thermophysics 14, 1993, 441.

11:30 AM
Investigation of Mechanical Properties and Microstructure of Various Molybdenum-Rhenium Alloys: *Todd A. Leonhardt*[1]; Jan-C. Carlen[1]; Martin Buck[1]; Charles R. Brinkman[2]; Weiju Ren[2]; C. O. Stevens[2]; [1]Rhenium Alloys Inc., R & D, 1329 Taylor St., P.O. Box 245, Elyria, OH 44036-0245 USA; [2]Lockheed Martin Energy Research Corporation, Oak Ridge National Lab., Oak Ridge, TN 37831 USA

Powder metallurgy sheets of molybdenum-rhenium alloys with 41, 44.5, 47.5 and 51% rhenium content were produced to examine the mechanical properties at room temperature, 1073K, and 1473K. The effect of rhenium additions to molybdenum near and exceeding the saturation point of rhenium in molybdenum were compared to pure molybdenum. Molybdenum-rhenium alloys show an increased strength over pure molybdenum. Microstructure analyses were performed to correlate mechanical properties with the microstructures of each of the molybdenum-rhenium alloys. This study shows that an optimum rhenium content can be achieved to maximize room temperature properties and elevated temperature performance. Some of the major advantages of the molybdenum-rhenium alloys are high melting points, resistance to thermal shock, and high temperature strength at elevated temperatures. The reduction of the ductile-brittle transition temperature coupled with high temperature strength of the molybdenum-rhenium alloys often make them the best choice for applications such as: heat sinks, heating elements, thermocouple sheathings, reflectors, vacuum furnace com-

ponents, electron tube components, hydrazine thrusters, and other important industrial, defense and aerospace applications.

Symposium to Honor Professor Julia R. Weertman: Physical Metallurgy

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Tuesday AM
November 2, 1999
Room: Rosewood
Location: Omni Netherland Plaza Hotel

Session Chairs: Gregory B. Olson, Northwestern University, Evanston, IL 60208 USA; Stuart R. Stock, Georgia Institute of Technology, Atlanta, GA USA

8:30 AM Invited
Heavily Cold Worked Metals: Structure and Properties: *Niels Hansen*[1]; [1]Riso National Laboratory, Mats. Rsrch. Dept., Roskilde DK-4000 Denmark

Large strain deformation of metals and alloys leads to a microstructural subdivision on a very fine scale. Subdividing features are dislocation boundaries and high angle boundaries and key microstructural parameters are the spacing between such boundaries and their angle of misorientation. These parameters have been characterized by TEM including Kikuchi pattern analysis. Supplementary the macroscopic texture has been determined by neutron diffraction. By the way of examples pure materials have been examined after monotonic deformation and after deformation where the strain direction has been changed. It has also been examined, how an addition of fine particles or relatively coarse fibres affects the microstructure and texture evolution. These experiments show a significant affect of both materials and process parameters which is reflected in the mechanical properties and in the recrystallization behaviour of the cold worked metal.

9:00 AM
Numerical Simulation of Ostwald Ripening in Elastically Stressed Solids: *Katsuyo S. Thornton*[1]; Norio Akaiwa[2]; Peter W. Voorhees[1]; [1]Northwestern University, Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305-0047 Japan

We examine the effects of elastic stress on the spatial distribution and morphology of misfitting particles during coarsening in an elastically homogeneous, anisotropic solid. Our computational model simulates Ostwald ripening in a solid-solid system efficiently and accurately by taking advantage of powerful numerical methods such as the boundary integral method with the fast multipole method. We study large systems consisting of many thousands of particles to produce quantitative, statistically meaningful measures of the temporal evolution of the microstructure. We also give special attention to single-particle and few-particle systems in order to shed light on the fundamental aspects of underlying processes that govern the evolution of the microstructure under elastic stress. The recent results from our numerical and semi-analytical studies are presented.

9:15 AM
Exploring Segregation on an Atomic Scale in Grain-Boundary Phase-Space: *David N. Seidman*[1]; [1]Northwestern University, Mats. Sci. and Eng. Dept., 2225 N. Campus Dr., Mats. and Life Sci. Bldg., Evanston, IL 60208-3108 USA

Monte Carlo techniques (Metropolis algorithm and overlapping distributions MC) have been extensively used to explore systematically the eight-dimensional grain boundary (GB) phase space for both twist and tilt boundaries in single-phase binary f.c.c. alloys—Au-Pt, Ni-Pd, and Ni-Pt; the atomic interactions are described by EAM potentials. We use lattice statics calculations to determine the lowest energy GB structures at 0 K and then MC simulation, at elevated temperatures, to calculate the distribution of solute atoms and the Gibbsian interfacial excess for each GB studied. Accurate segregation free energies for individual sites in GBs are calculated with the overlapping distributions MC technique and segregation entropies are determined and found to be a linear function of the segregation internal energies (0 K) for the same GB sites. The effects of both the five macroscopic and three microscopic degrees of freedom (DOFs) are studied and it is demonstrated that the Gibbsian interfacial excess is a complicated function of both macroscopic and microscopic DOFs. Also a GB's atomic structure determines the partitioning of segregating atoms between the cores of the GB dislocations and in the elastic stress field of the GB dislocations. We also demonstrate that none of the geometric criteria suggested in the literature is capable of prediciting the propensity for GB segregation, much less the magnitude. Experimental results have been obtained on selected binary alloys employing combined transmission electron and atom-probe microscopies, which demonstrate the importance of the macroscopic DOFs in determining the value of the Gibbsian interfacial excess of solute for a specific GB, as well as the concentration profiles normal to the GB plane. Research supported by the National Science Foundation/Division of Materials Research.

9:30 AM
A Model of Interstitial Solution Strengthening of Austenite–Relative Strengthening by Carbon and Nitrogen: *W. S. Owen*[1]; M. Grujicic[2]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., Cambridge, MA 02139 USA; [2]Clemson University, Dept. of Mech. Eng., Ctr. for Adv. Manufacturing, Clemson, SC 29634 USA

A quasi-chemical, pair approximation model which describes both the high temperature athermal critical shear stress and the bcc-like low temperature strengthening of alloy austenites strengthened by either C or N is described. Attention is focused on the critical shear stress to move a dislocation in the athermal range of temperature between 298 K and a temperature above which significant diffusion of metal or interstitial atoms occurs in the time required for the dislocation to sweep the slip plane. Equilibrium local order is defined in terms of the distribution of metal and interstitial atoms over octahedral clusters and is determined by a Monte Carlo method using a Boltzmann acceptance criteria. An extended dislocation bounded by Shockley partials intersecting a cluster removes two metal atoms from each cluster and replaces them by two nearest-neighbor metal atoms, while the atoms on the interstitial sublattice remain undisturbed. The change in the number and configuration of pairs is calculated, thereby allowing an assessment of the change in enthalpy and entropy. The critical shear stress is then a linear function of the change in Gibbs energy. The required pair energies and pair-exchange energies are obtained for metal-metal pairs by comparing the Gibbs energy of mixing calculated using the tetrahedral-octahedral cluster variation, pair approximation model with the corresponding Gibbs energy derived from experimental values recorded in ThermoCalc. The energies of metal-interstitial pairs are obtained from the Gibbs energies of hypothetical fcc binary alloys deduced from CALPHAD assessments. Substitutional solution strengthening is neglected in the subsequent calculation because pair exchange energies of metal-metal pairs are much smaller than those of metal-interstitial pairs. Application of the model is illustrated by calculating the increase in the critical shear stress, at 298 K, with increasing C and N content of Fe-12Mn-(C,N), Fe-40Ni-15Cr-(C.N), and two Fe-Mn-Cr-(C,N) alloys. The results demon-

strate the well-known superior strength of the alloys containing N and are in reasonable agreement with the values deduced from measurements of the initial flow stress of polycrystalline alloys.

9:45 AM

Coarsening of Coherent Particles in Ni-Al Alloys: *Hector A. Calderon*[1]; Jose Jesus Cruz[1]; [1]IPN, Dept. Ciencia De Materiales, Upalm Ed. 9, Apdo. Postal 75-707, Mexico, D.F. 07338 Mexico

The coarsening process of g´ particles in single crystals of the alloy Ni-12 at.% Al has been investigated after a series of single and double aging treatments. Special attention is given to the late stages of coarsening and the influence of the elastic interactions between particles. Single aging treatments at 1133 K (close to the g-g'solvus line) produce changes in particle shape, size and spatial arrangement. Groups of large rectangular particles are formed after relatively short aging times. They can be interpreted as a result of a mechanism of particle migration. The second aging has been performed at 923 K. The smaller particles acquire a size comparable to that of the larger ones after approximately 1200 h of aging. Particle arrangement and evolution is controlled by migration mechanisms. The coarsening kinetics are also a smooth function of the aging time. Structural differences have a definite influence in the formation of the observed particle groups. HREM allows observation of the translation do mains of particles in arrays. Different domains are found between particles forming an array, which gives support to the mechanism of migration for the formation of particle groups.

10:00 AM BREAK

10:20 AM

Analysis of Triple Point Distributions in Polycrystalline Materials: *Wayne E. King*[1]; Mukul Kumar[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci. Directorate, L-356, 7000 East Ave., Livermore, CA 94550 USA

Recently, it has been demonstrated that some material properties can be enhanced by grain boundary engineering, i.e., systematic modifications in the topology of the microstructure through thermomechanical processing. Experimental observations have shown that the microstructural feature likely responsible for improved properties is the triple junction. For example, it has been observed that cracks propagating along interconnected networks of random grain boundaries can be arrested when intersecting a triple junction where the remaining two pathways are special boundaries as described by the CSL model. Therefore, it is of interest to characterize microstructures in terms of the distributions of triple junction types. A simple method to describe a triple point is by the types of grain boundaries intersecting at that junction (special vs. random). The distribution of 0-CSL, 1-CSL, 2-CSL and 3-CSL boundaries in the microstruture can then be plotted as a function of the fraction of special boundaries. Such data has been collected using orientation imaging microscopy for ofe-Cu and Inconel 600 over a range of special fraction of grain boundaries. These results have been compared with theoretical models considering isolated triple points and invoking the sigma product rule. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

10:35 AM

X-Ray Line Broadening Analysis of Coherent M2C Precipitation in Ni-Co Secondary Hardening Steels: Y. Nagataki[2]; J. B. Cohen[1]; *G. B. Olson*[1]; [1]Northwestern University, Dept. Mat. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60015 USA; [2]NKK Corporation, Japan

In support of multiscale modeling of microstructural evolution for computational materials design, X-ray line broadening measurements of dislocation cell size and matrix coherency strain are integrated with SANS measurements of M2C precipitate size and phase fraction during secondary hardening of ultrahigh-strength Ni-Co martensitic steels. In addition to the retarding effect of Co in solution, M2C precipitation further retards dislocation recovery, and the effect is greater with the finer-scale precipitation associated with higher thermodynamic driving force. An increase in microstrain distribution amplitude during early precipitation is attributed to carbide coherency strain. A subsequent decrease occurring at or before peak hardness is interpreted as the onset of coherency loss. This supports similar critical particle sizes of ~3nm diameter for coherency loss and the shear-to-bypass transition in strengthening behavior. Research supported by ARO.

10:50 AM

Microstructure Evolution of Ti-Zr-Ni Phases in Non-Stoichmetric Zr-Ti-Mn-V-Ni Hydride Electrode Alloys: *Xueyan Song*[1]; Rui Vilar[1]; [1]Instituto Superior Tecnico, Depart. de Engenharia de Materiais, Av. Rovisco Pais Lisboa Codex, Lisbon 1049-001 Portugal

The microstructure of non-stoichmetric Zr-Ti-Mn-V-Ni hydride electrode alloys was systematically investigated by Transmission Electron Microscopy. For the investigated alloys of Zr0.95-xTix(MnVNi)2.2 (x=0, 0.1, 0.3, 0.4), the non-Laves phase in Ti-containing alloys were found to be Ti-Zr-Ni phases which were related to TiNi phase with BCC structure. The Ti-Zr-Ni phases in Zr0.95-xTix(MnVNi)2.2 (x=0, 0.1, 0.3, 0.4) alloys were determined to be (TiZr)Ni and (TiZr)2Ni phase. Crystalline nature of (TiZr)Ni and (TiZr)2Ni phase varied with the alloy compositions. As x=0.2, (TiZr)Ni phase with B2 type structure and long range ordered tetragonal (TiZr)2Ni phase, as well as new minor phases P and P precipitated in the (TiZr)Ni phase matrix, coexisted in the alloy. The crystal structure of P phase was identified to be face centered orthogonal structure and P phase was identified to be body centered cubic structure. As x=0.3 and x=0.4, the (TiZr)Ni phase transformed to premartensite R phase and only P phase precipitate in the supersaturated R phase matrix. The lattice correspondence between (TiZr)2Ni phase and (TiZr)Ni phase was determined in Zr0.95-xTix(MnVNi)2.2 (x= 0.4) alloy.

11:05 AM

Mesotexture, Deflection and Closure of Fatigue Cracks in Al-Li 2090 T8E41: *Stuart R. Stock*[1]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., 778 Atlantic Ave., Atlanta, GA 30332-0245 USA

High levels of crack closure, leading to extremely low crack propagation rates, accompany large deflections of fatigue cracks in samples of Al-Li 2090 T8E41. The orientations of the pancake-shaped grains through which a crack cuts and the positions where and the stresses at which crack faces contact must affect subsequent crack extention. Quantification is essential. To this end, results of x-ray microtomography and of x-ray microbeam diffraction of Al-Li 2090 T8E41 are summarized here and reveal a complex, three-dimensional distribution of crack opening as a function of load and a characteristic mesotexture related to large crack deflections, respectively. Large sections of fatigue cracks close at loads above the nominal closure load, and mixed mode crack surfaces appear to be important. Groups of five or more adjacent grains possess nearly identical orientations (i.e. spatial as well as orientational correlation), this type of near-single-crystal mesotexture occupies fully forty percent of the volume and specific orientations of these grain groups produce sharp changes in crack path. Finally, the interplay of mesotexture and physical crack closure in fatigue crack propagation is addressed.

11:20 AM

Effect of Boron on the Surface Chemistry of Single Crystal Ni3(Al, Ti): *Jinliu Wang*[1]; [1]Northwestern University, Mats. Sci. and Eng., MLSB #2036, 2225 North Campus Dr., Evanston, IL 60208 USA

Previous work demonstrated that water dissociates into hydrogen on Ni3(Al,Ti) (100) surfaces. There is clear evidence that this dissociation reaction results in the reduced ductility of many polycrystalline aluminum-based alloys in a moist environment. Ductility measurements further show that boron may affect the production of atomic hydrogen from water vapor dissociation and its

surface mobility. To explore the effect of boron, we first dosed the surface of clean Ni3(Al, Ti) (110) with controlled amounts of boron, using a specially designed low-energy boron ion source, followed by low-temperature exposure to D2O and temperature-programmed desorption. Results indicated that water dissociates into atomic hydrogen on clean Ni3(Al, Ti) (110) boron-free surface. This dissociation reaction is strongly suppressed by boron adsorption at coverage >= 0.25 monolayer. The state of the surface was followed by x-ray photoemission. These studies showed that addition of boron reduces the production of atomic hydrogen. The significance of this observation will be discussed.

11:35 AM
Long Range Correlation of Orientation Measured by the Disorientation Correlation Function in Deformed 6022 Aluminum Alloys: Paul S. Lee[1]; *Anthony D. Rollett*[1]; Brent L. Adams[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes, Pittsburgh, PA 15213-3890 USA

A considerable effort has been expended to understand the development of microstructure in terms of the crystallographic orientation of the material at various structural scales. We have extended this approach by developing a disorientation correlation function (DCF) which is related to the two-point orientation correlation function, in order to investigate spatial correlation of orientation at the mesoscopic scale. We define the disorientation correlation function (DCF), f(r), as the mean value of the disorientation (angle) between all pairs of material points separated by a vector r. A DCF map is constructed by varying the separation vector (r). We have used data obtained by Orientation Imaging Microscopy to construct two dimensional DCF maps. The function is normalized to a perfectly disordered aggregate structure comprising the same grain size and texture. We have investigated the DCF as a function of strain level for samples of alloy 6022 T4 aluminum, deformed in plane strain tension. The results indicate the presence of long-range orientation correlations extending over more than five grain diameters. Substantial changes in the DCF are observed as a function of strain level. At large strains, for example, grains lying along the extension direction are more highly correlated than in any other direction. These observations of long range orientation correlation may have implications for strain localization in the deformation of sheets. This work was primarily supported by the Office of Energy Research, US Department of Energy under grant number DE-FG02-96ER45601.

Beryllium and Beryllium Alloys; Nuclear and Structural Applications: Beryllium-Corrosion and Environmental Issues

Sponsored by: Jt. ASM-MSCTS/TMS-SMD, Nuclear Materials Committee
Program Organizers: Michael F. Stevens, Los Alamos National Laboratory, MS G755, Los Alamos, NM 87545 USA; David J. Michel, Naval Research Laboratory, Code 6301, Washington, DC 20375 USA

Tuesday PM
November 2, 1999
Room: Salon M
Location: Omni Netherland Plaza Hotel

Session Chair: Loren A. Jacobson, Los Alamos National Laboratory, MST-6, Los Alamos, NM 87545 USA

2:00 PM
High Temperature Oxidation of Beryllides and Beryllium Modified Intermetallics: *Robert J. Hanrahan*[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., TA3 MS G770, Los Alamos, NM 87545 USA

Among the beryllides are some of the highest melting temperature intermetallic phases with the lowest densities (on the order of 2-4 gm/cc). Despite the potentially attractive refractory properties of beryllides, only a handful of studies on the oxidation of these compounds have been conducted since the mid 1960's. The problems identified in the oxidation of beryllides, notably intermediate temperature disintegration and high temperature oxide volatility in the presence of water vapor, were based on a very few studies which did not eliminate possible improvements through alloying additions. Today, the beryllides deserve further investigation in order to understand the properties of beryllium modified intermetallics, which (given the limitations of the periodic table) may very well represent the last and best available option for improving both the mechanical and oxidative properties of this class of materials. We will review the oxidation of monolithic beryllides and present recent results on the oxidation of beryllium modified aluminides (NiAl, TiAl) and silicides (Mo5Si3).

2:30 PM
Corrosion of Single Crystal and Polycrystalline Beryllium: *James Eugene Hanafee*[1]; [1]Lawrence Livermore National Laboratory, Chemistry and Mats. Sci., L352, 7000 East Ave., Livermore, CA 94550 USA

Polycrystalline beryllium is anisotropic, and corrosion might occur more readily on some surfaces than others. This may have practical consequences in beryllium applications. In this study, high purity, zone refined, single crystals were prepared with surfaces parallel to the basal plane and the two prism planes. The surfaces were characterized using optical microscopy, SEM and AFM, and then subjected to carefully controlled corrosion conditions. The type, depth, and orientation of the pits on the three cystallographic surfaces was determined after exposure to the corrosion media, and compared. Implications for structural grades of beryllium are discussed.

2:55 PM
Characterization of Beryllium with Radiation Damage: *Harriet Kung*[1]; Robert D. Field[1]; B. Utkelbayev[2]; P. Chakrov[2]; [1]Los Alamos National Laboratory, Mail Stop G755, Los Alamos, NM 87545 USA; [2]Institute of Atomic Energy, Nat. Nuclear Ctr., Almaty, Kazakhstan

The behavior of hydrogen isotopes in the beryllium is one of the important scientific and engineering problems related to the design of fusion reactor. Special interest is focused on obtaining data on the defect structure produced by high dose hydrogen implantation, and on the determination of the state of hydrogen in the material. In the present work, hot pressed beryllium (TGP-56 grade) was irradiated with 650 keV protons at a temperature below 50°C and a beam current of 1 mA/cm2, where the maximal hydrogen concentration was 10000 appm H (1 at.% H-atomic hydrogen). A technique was applied that allows the study of the whole range of the ion damaged zone by transmission electron microscope. High resolution transmission electron microscopy characterization of Be samples has been conducted using JEOL3000F HRTEM equipped with a field emission gun. The results show that near the implanted areas there are small regions with structure different from the parent matrix (h.c.p. Be). Computer generated FFT patterns taken from the lattice images of these regions show a four-fold symmetry, suggesting the presence of a cubic-type structure (e.g. b.c.c.). The lattice parameter of the possible bcc phase is estimated to be ~ 2.8Å, which is slightly larger than the reported value (2.55Å) in the literature. The possibility of stabilizing a metastable form of b.c.c. Be via the implantation process will be discussed.

3:20 PM Break

3:30 PM
The Corrosion of Beryllium and Beryllium Weldments: *Scott Lillard*[1]; Mary Ann Hill[1]; Robert Hanrahan[1]; Darryl Butt[2]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., MST-6, MS G755, Los Alamos, NM 87545 USA; [2]Ceramatec, 2425 S. 900 W., Salt Lake City, UT 84119 USA

The passivity of 98.5% pure beryllium has been studied in ambient temperature aqueous solutions as a function of pH. Below solution pH 2, potentiodynamic polarization curves exhibited active dissolution at all applied anodic potentials. In solution pH 2-12.5 it is shown that beryllium exhibits passive anodic behavior. Oxide growth rate (as measured by electrochemical impedance spectroscopy) was determined to be 6.0 Å/V over the potential range of 0-4V in pH 7 solution. At higher anodization potentials the growth rate appeared to be somewhat lower. In chloride solution, the pitting potential of Be was found to vary logarithmically with chloride concentration. To study the galvanic effects at Be weld zones, both bulk analogs and Al/Si eutectic welds were fabricated. As anticipated from bulk analog experiments and the EMF series, Be dendrites (solidified from the weld melt) preferentially corrode from the weld matrix. That is, the weld is cathodically protected by the bulk Be. Similarly, the behavior of second phase constituents in Be (FeBe12, TiBe12, etc.) are predicted from the results of bulk analogs.

3:55 PM

Comparison of Elevated Temperature Properties For HIP'D Impact Ground Beryllium and HIP'D Gas Atomized Beryllium: *D. E. Dombrowski*[1]; W. J. Haws[1]; P. C. McKeighan[2]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA; [2]Southwest Research Institute, P.O. Drawyer 28510, San Antonio, TX 78228-0510 USA

The elevated temperature (ambient to 648°C) thermomechanical properties of two beryllium grades made by Hot Isostatic Pressing (HIP) are compared: S-65H (made from impact ground powder) and 0-30 (made from gas atomized powder). Successful measurements of elevated temperature smooth and notched fatigue were made for the first time on modem beryllium grades. Valid beryllium Klc fracture toughness results were obtained for the first time at temperatures above room temperature. Elevated temperature creep, tensile, electrical resistivity and thermal conductivity data are also presented. This data addresses needs of fusion energy designers for advanced tokamak reactors such as the International Thermonuclear Experimental Reactor (ITER).

4:20 PM

Examination of the Fatigue Endurance Limit of AlBeMet 162: *M. Svilar*[1]; C. Pokross[2]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA; [2]Brush-Wellman Inc., 14710 Portage River S. Rd., Elmore, OH 43416-9502 USA

AlBeMet 162 (38wt% Al-62 wt% Be) is a high specific stiffness, low density aluminum-beryllium alloy being used in high performance automotive and, satellite and aircraft control surface applications. Fatigue can be an important design property in some of these applications. Rotating beam fatigue measurements are presented at room temperature and 250°C (480°F). An experimental process to improve the fatigue strength of AlBeMet 162 at high performance automotive engine temperatures of about 250°C (480°F) resulted in an increase of room temperature 10 7 cycle fatigue endurance compared to previous data. The results show 10 7 cycle fatigue endurance limits is approximately 70% of the transverse yield stress and 90 % of the longitudinal yield strength. Fractography was used to determine the effect of microstructure on the variability in cycles to failure for specimens tested at similar stresses.

General Abstract Sessions: Advances in Processing and Heat Treatment I

Sponsored by: TMS

Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Tuesday PM — Room: Salon F&G
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: John H. Perepezko, University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., Madison, WI 53706 USA

2:00 PM

An Experimental and Theoretical Study of Alloys Unidirectional Solidified Under the Condition of the Applied Electric Field: *Yuning Jiao*[1]; Susumu Takamori[1]; Goro Arakane[1]; Yoshiaki Ohsawa[1]; Akira Sato[1]; [1]National Research Institute for Metals, Mats. Creation Station, 1-2-1 Sengen, Tsukuba 305-0047 Japan

On the basis of the model of atomic cluster jump theory, a microdynamical model of crystal growth rate was developed under the electric fields. The classical continuous growth mode seems to be only one of the special case. Theoretical analysis showed that the crystal growth rate is dependent on not only the nature of metal as well as the undercooling and so on but also the applied electric fields. A novel model, the effect of an applied electric field on the solute distribution, the constitutional undercooling and the temperature distribution at solid/liquid interface during unidirectional solidification, has been established theoretically. Theoretical analysis shows the electrotransport of solute strongly influences the solute distribution and then changes the constitutional undercooling and the temperature distribution. But all changes are connected with the current direction of applied electric field (positive or negative). A new critical criterion without the constitutional undercooling in front of the so lid/liquid interface is obtained for unidirectional solidification under an applied electric field. A novel experimental method of unidirectional solidification was designed by adding an applied electric field. The experimental results indicated that the influence of electric current density on the morphology and distribution of microstructure of the Cu-85Sn peritectic alloy was very strong. The morphology of the eutectic phase clearly showed the direction of the fluid flow under an applied electric field. With the increasing electric current density, the morphology of the primary phase e was changed from regular plate-like to irregular plate-like and that of the peritectic phase h from dendrite to regular equiaxed grain. An area with total peritectic phase could be obtained. The analysis for the experimental results showed that the fluid flow is a main factor, which affected the morphology and distribution of the primary and peritectic phase under an applied field.

2:20 PM

Structural Identification of a Metastable $CuInSe_2$ Phase Formed by Solid State Diffusion: *Zhenfu Dong*[1]; Joon Sik Park[1];

John H. Perepezko[1]; [1]University of Wisconsin-Madison, Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

During solid state precipitation, it is rare to observe dendritic precipitates. Nevertheless, solid state dendritic precipitation would be expected when some critical structural and diffusion conditions are simultaneously satisfied, such as relatively isotropic interfacial energy and low lattice mismatch between the precipitate and matrix, low diffusivity within the precipitate and widely spaced precipitates. In the present work, upon annealing a Cu_2Se/In_2Se_3 diffusion couple at 550°C and ambient condition, a $CuInSe_2$ (CIS) phase with dendritic morphology was precipitated within a FCC Cu_2Se matrix. According to the literature, the stable structure for the CIS phase at ambient pressure is always a chalcopyrite-type (tetragonal) regardless of the synthesis methods. However, it is difficult for the structural conditions required by dendritic precipitation to be satisfied when a tetragonal structure precipitates within a cubic structure matrix. Therefore, the structure of the dendritic CIS precipitate was examined using electron diffraction combined with the simulation of electron diffraction patterns. A metastable zincblende-type (cubic) rather than the stable chalcopyrite-type structure was uniquely determined for the CIS phase formed during the reactive diffusion. The formation of the dendritic morphology is discussed in terms of the structural similarity and orientation relationship between the precipitate and the matrix, as well as the diffusion characteristics in the Cu_2Se matrix. The support of ONR (N00014-92-J-1554) is gratefully acknowledged.

2:40 PM

Processing and Alloy Chemistry Effects on the Recrystallization Kinetics of Aluminum Alloy AA5052: *Malesela Jones Papo*[1]; Burton R. Patterson[1]; Hasso Weiland[2]; [1]University of Alabama-Birmingham, Dept. of Mats. and Mech. Eng., BEC 240, 1150 10th Ave. S., Birmingham, AL 35205 USA; [2]Alcoa, Alcoa Tech. Ctr., Alcoa Center, PA 15069 USA

The effects of Cr level, preheat temperature, and hot versus cold rolling, on the recrystallization kinetics of AA5052 have been studied using stereological methods. Results suggest that Cr addition retards the recrystallization kinetics in both the hot and cold rolled materials. Increased preheat temperature shows faster transformation kinetics. Nucleation rate measurements indicate greater rates for the 0% Cr addition, especially at lower volume fraction transformed. Materials with 0.2% Cr with different preheat temperatures show no clear difference in nucleation rate. Cahn-Hagel growth rate measurements show highest growth rate for the cold rolled, 0% Cr material. The cold rolled high preheat, 0.2% Cr material shows slower growth rate than its low preheat counterpart, especially at higher volume fractions. All cold rolled materials show constant, then decreasing growth rates with time. The hot rolled alloy shows the lowest, but constant growth rate.

3:00 PM

Transformation from Metallic Glass to the Equilibrium Phases in Al90Fe5Gd5: *K. Matsuda*[1]; G. J. Shiflet[2]; R. E. Hackenberg[2]; S. Ikeno[1]; [1]Toyama University, Dept. of Sys. Eng. for Life Sci. Mats., Toyama 930-8555 Japan; [2]University of Virginia, Mats. Sci. and Eng., Thornton Hall, Charlottesville, VA 22903 USA

The equilibrium phases in annealed Al90Fe5Gd5 amorphous glass were investigated by analytical and high resolution transmission electron microscopy. Phases identified include FCC aluminum, Al3Gd, and a few Al-Fe-Gd ternary intermetallic compounds. In Al90Fe5Gd5 amorphous glass annealed at 773K for 5.4ks, there are FCC aluminum, Al3Gd, and Al20Fe2Gd and Al10Fe2Gd ternary intermetallic compounds. The aluminum grains contain less than 0.04mass% Fe and Gd. Other phases of ternary intermetallic compounds appeared after prolonged annealing for more than 86.4ks. These ternary intermetallic compounds have similar crystal structures and different chemical compositions from each other as established by EDS and X-ray diffraction. Results will be compared to calculated ternary phase diagrams using Thermo-Calc.

3:20 PM

Strain Induced Melt Activated (SIMA) Processing of ZA-27 Alloys: *Pinar Ercan*[1]; Mahmut Ahsen Savas[1]; Sabri Altintas[1]; [1]Bogazici University, Mech. Eng., Nispetiye Cad., Bebek, Istanbul 80815 Turkey

In this study, strain induced melt activated (SIMA) process is conducted on Zn-27 wt. % Al alloy to obtain a globular microstructure suitable for thixoforming. A die with a punch is manufactured for gravity and squeeze castings, where the specimens obtained are subjected to hot and cold rolling with varying deformations. The globular microstructure is sought for by heat treatment with varying temperatures above the solidus temperature of the alloy. The parameters such as casting procedure, temperature, and time affecting the SIMA process at various stages are investigated. Hardness Test is conducted on cast specimens and metallographic examination is done after every stage of the process. At present, Zn-27 wt. % Al alloy is a suitable alloy for the SIMA process, a globular microsturucture can be obtained, provided that the cast structure, hot and cold deformation ratio, the annealing temperature and also the time are carefully controlled.

Hydrogen Effects on Materials Behavior: Adsorption, Permeation, and Diffusion

Sponsored by: Structural Materials Division; ASM International: Materials, Science, Critical Technology Section; Corrosion and Environmental Effects Committee

Program Organizers: Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Russell H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; A. W. Thompson, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

Tuesday PM — Room: Salon H&I
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Russell H. Jones, Pacific Northwest National Laboratories, Richland, WA 99352 USA; Brian P. Somerday, Sandia National Laboratories, Livermore, CA USA

2:00 PM

Hydrogen Effects on Metallic Glasses: *Dan Eliezer*[1]; Noam Eliaz[1]; [1]Ben-Gurion University of the Negev, Dept. Mats. Eng., P.O. Box 653, Beer-Sheva 84105 Israel

The interaction of hydrogen with metallic glasses has been studied extensively during the last two decades. These studies were motivated by both scientific and technological interest, mainly the potential use of amorphous hydrides in hydrogen storage technology. In this work we summarize our recent progress and review our current understanding of hydrogen interaction with metallic glasses. We will discuss in detail absorption/desorption characteristics, hydrogen effects on the microstructure and thermal stability, and possible mechanisms of hydrogen embrittlement in various systems. The use of advanced experimental techniques, such as thermal desorption spectroscopy (TDS) and laser-induced shock waves measurements, in studying the effects of hydrogen on metallic glasses will be presented. Some of the issues to be discussed in detail include: a) the significant increase of desorption temperatures due to the presence of hydride forming elements; b) possible effects of the high pressures involved in electrochemical charging on fracture characteristics; c) hydrogen effects on the local microstructure of amorphous FeSiB alloy; and d) embrittlement of this alloy by a mechanism of high-pressure bubble formation.

2:20 PM
Diffusion of Hydrogen in Amorphous Metals: *Noam Eliaz*[1]; Dan Eliezer[1]; David Fuks[1]; [1]Ben-Gurion University of the Negev, Dept. Mats. Eng., P.O. Box 653, Beer-Sheva 84105 Israel

The study of hydrogen diffusion in amorphous metals and alloys is of both scientific and technological interest. Both experimental and simulation results have indicated the deviation from Arrhenius law of the diffusion coefficient of hydrogen in glassy metals. This deviation has been explained by the existence of various kinds of jumps or in terms of continuous distributions of activation energies due to different kinds of disorder. In this work, the experimental evidence for the deviation from Arrhenius law is reviewed, and a new model which explains this behavior in terms of the short-range order of the structure and its temperature dependence is suggested. Accounting for the temperature dependence of the structure of amorphous metals leads to pronounced temperature dependence of the activation energy for diffusion processes. This effect reflects itself in the non-Arrhenius behavior of the diffusivity of small-atomic-volume impurities in amorphous metals. To illustrate the applicability of this model, the results on hydrogen diffusion in dilute amorphous Fe-H, Fe-Si-H and Fe-B-H alloys will be presented. The effect of the alloying elements on the activation energy of hydrogen diffusion will be discussed in terms of their electronic structure and mean volume.

2:40 PM
Fracture Behavior of ReNi5 Powders during Hydrogen Charging and Discharging Cycles: *S. B. Biner*[1]; [1]Iowa University, Ames Lab., Metallu. and Ceramics, 208 Metals Dev., Ames, IA 50011 USA

In this study, the evolution of the stress states in $ReNi_5$, where Re denotes the rare earth elements, particles during hydrogen charging and discharging cycles were investigated using coupled diffusion-deformation FEM analyses. The results indicate that large tensile stresses, of the order of 20-30% of the modulus of elasticity, develop in the particles even in the absence of both internal and external crack-like defects. The internal and external cracks behave differently from each other during hydrogen charging and discharging cycles. Therefore, the fracture resistance of the particles containing external cracks will be different from the particles having internal cracks. The disc-shaped particles, in addition to having faster charging-discharging cycles, may offer better resistance to fracture than the spherical particles. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-ENG-82. This research was supported by the Director of Energy Research, Office of Basic Sciences.

3:00 PM
Deuterium Retention and Release in Coated and Bare Aluminum: *Kristin L. Kurz*[1]; Rion A. Causey[1]; Donald F. Cowgill[1]; Bernice E. Mills[1]; [1]Sandia National Labs/8700, 8716/Surface Chem., P. O. Box 969, MS 9161, Livermore, CA 94551-0969 USA

The Accelerator Production of Tritium (APT) project utilizes spallation neutrons incident on thousands of 3He gas filled metal tubes to produce tritium by way of the exothermic 3He(n,p)3H reaction. Depending on the final design for the diameter of the tubes, between 10 to 15 % of the 190 keV tritons and 30 to 45 % of the 600 keV protons produced are directly implanted into the metal tube walls. To minimize tritium permeation into the coolant surrounding the tubes and to minimize tritium inventory, it is desirable to have the implanted tritium migrate back to the inner surface of the tubes and rapidly recombine to be released as T2 and HT. The work presented here is part of the research being performed at Sandia National Laboratories to develop a tube design that enhances this release process. Al 6061-T6 is the baseline material for fabrication. Tritium retention and release characteristics for bare aluminum, Cu coated aluminum, and Ni coated aluminum has been measured as a function of total particle fluence. The samples were implanted with deuterons varying in energy from 15 keV to 200 keV at 100°C up to a fluence of 3x1018 D/cm2. The samples were also implanted with protons varying in energy from 150 keV to 600 keV to duplicate the additional radiation damage and H-loading. The deuterium retention was measured by mass spectrometry during thermal desorption. The thermal desorption spectra were compared to theoretical calculations of diffusion and trapping. The bare aluminum retained approximately 20 % of the implanted deuterium. The Cu and Ni coatings reduce the retention significantly. SEM images of both the cross sections and surfaces were used to correlate material effects with retention. Simple modeling that includes both mobile and fixed deuterium concentrations is consistent with the experimental findings.

3:20 PM
The Potential Role of Hydrogen in Stress Corrosion Cracking of 5083 Aluminum: *Russell Howard Jones*[1]; Donald R. Baer[1]; Michael J. Danielson[1]; Charles F. Windisch[1]; John S. Vetrano[1]; [1]Pacific Northwest National Laboratory, Mats. Dept., P.O. Box 999, MSIN P8-15, Richland, WA 99352 USA

Aluminum alloys are being considered as structural materials for producing lightweight automobiles. Aluminum-magnesium alloys are candidates for components that require moderate strength, formability and weldability. Alloy 5083 is an Al-Mg alloy with 4.5 wt% Mg which meets these goals but alloys with greater than about 3% Mg have shown susceptibility to corrosion and stress corrosion (SCC) following low temperature heat treatments or thermal exposures as low as 90°C. There is also a desire to increase the strength of solid solution strengthened Al-Mg alloys by increasing the Mg concentration; however, the it is not clear whether the role of Mg in the corrosion and SCC susceptibility is associated Mg segregation and/or precipitation of the Al3Mg2 phase at grain boundaries. This knowledge is required to identify methods to reduce the corrosion and SCC susceptibility of these alloys. Segregation to grain boundaries can be reduced by alloy modifications while precipitation at grain boundaries an be reduced by thermal treatment. A thermal treatment to control grain boundary precipitation of the Mg rich Al3Mg2 phase has shown a promising reduction in the corrosion susceptibility of an alloy with 7% Mg. Further development of this approach could lead to high strength Al alloys for automotive applications. The corrosion and stress corrosion cracking (SCC) susceptibility of an Al-Mg alloy has been shown to depend on the precipitation of the Mg rich phase, Al3Mg2, but not the enrichment of elemental Mg at grain boundaries to concentrations up to 3 times that of the alloy. These results were determined by measuring the progress of Mg enrichment at grain boudaries, for increasing thermal treatment times, using Auger Electron Spectroscopy (AES) of grain boundaries exposed by fracture within the spectrometer. The progress of the Al3Mg2 precipitation was followed by Analytical/Transmission Electron Microscopy (AEM), for the same thermal treatment times. The lack of a Mg segregation effect was demonstrated by both X-Ray Photoelectron Spectroscopy (XPS) analysis of Mg implanted Al following in situ electrochemical tests and SCC testing while the dominance of Al3Mg2 precipitation was demonstrated by electrochemical analysis and SCC testing. The hydrogen permeation rate in Al is highly dependent on the nature of the passive film and higher hydrogen permeation rates have been demonstrated at potentials anodic to the open circuit potential. Crack growth tests of A5083 conducted at potentials cathodic and slightly anodic to the open circuit potential have been conducted and demonstrated faster cracking at the slightly anodic potentials with no increase at cathodic potentials. This is consistent with reported hydrogen permeation results for Al. This research was funded by the Division of Materials Sciences of the Offices of Basic Energy Sciences of the U.S. Department of Energy.

3:40 PM Break

3:50 PM
Hydrogen Uptake during Environment Assisted Cracking in Al-Zn-Mg-Cu: *Lisa M. Young*[1]; Richard P. Gangloff[1]; [1]University of Virginia, Mats. Sci. Dept., Materials Science Bldg., School of Eng. and Applied Sci., Charlottesville, VA 22903-2442 USA

The goal of this research was to quantify short-transverse aqueous environment assisted cracking (EAC) kinetics and associated crack tip hydrogen uptake as a function of aging treatment in a precipitation hardened Al-Zn-Mg-Cu alloy, AA 7050. The approach utilized high resolution, in-situ crack length measurements via compliance to quantify Stage II fracture mechanics-based EAC rates in an aqueous chromate/chloride environment. Because crack growth for this alloy is highly sensitive to electrode potential (E), the WOL specimen was maintained at constant E throughout each 1-3 week experiment. Substantial EAC susceptibility was observed in the underaged and peak aged conditions of AA 7050, where the highest crack growth rates (1.2x10-3 and 1.2x10-4 mm/sec, respectively) occurred at the highest applied potential (-445 mVSCE). Underaged and peak aged crack kinetics consistently exhibited a complex incubation and hysteresis response to changes in applied anodic potential. In contrast the overaged condition was highly EAC resistant, but not immune, exhibiting crack growth rates £ 2x10-8 mm/sec or ~10,000 times slower than the peak aged condition at the highest applied potential (-445 mVSCE). Scanning electron microscopy of fracture surfaces and optical microscopy of metallographic cross-sections of an EAC crack showed that slow rate incubation EAC (1x10-6 mm/sec ³ da/dt ³ 1x10-7 mm/sec) and fast rate EAC (da/dt ³ 1x10-5 mm/sec) for all tempers were mainly intergranular between high angle grain boundaries. In contrast resistant overaged material exhibited slow transgranular cracking. Thermal desorption spectroscopy (TDS) and nuclear reaction analysis (NRA) were used to characterize crack wake hydrogen (H) uptake during EAC. Results for both methods revealed enhanced H uptake under applied anodic potential for tempers exhibiting fast cracking. The TDS specimen depth into the crack wake (1 mm) was likely much greater than the H-enriched crack tip process zone size. However, due to the high sensitivity of the technique, TDS results clearly indicated enhanced uptake in fast-cracking EAC regions. Hydrogen analyses were complicated by the possible dependence of H-production and uptake on wake exposure time and pH in the occluded crack environment, but the crack wake H concentration, normalized by the wake exposure time, correlated with aqueous EAC rate. Nuclear reaction analysis, with spatial resolution on the order of 10 nm, was used to investigate the H concentration profile to a depth of 1 mm into the crack wake surfaces and revealed unexpectedly high surface H concentrations (~2000 wppm). These high concentrations confirmed estimates that were calculated based on TDS-measured average concentrations and effective hydrogen diffusivity. These-concentration profiles indicate that the observed crack velocities can be supported by bulk H-diffusion into the crack tip process zone, where EAC is promoted by a hydrogen embrittlement mechanism.

4:10 PM

An Investigation into the Hydrogen Uptake, Diffusion, and Hydrogen Environment Assisted Cracking of an Al-Zn-Mg-(Cu) Alloy: *George A. Young*[1]; John R. Scully[1]; [1]University of Virginia, Ctr. for Electrochem. Sci. and Eng., Dept. of Mats. Sci. and Eng., Charlottesville, VA 22903-2442 USA

There is strong evidence to indicate that hydrogen embrittlement plays a significant, if not controlling, role in the environmentally assisted cracking of 7XXX series aluminum alloys. In order to better understand hydrogen environment assisted cracking (HEAC), crack growth rate tests in the K-independent stage II crack growth regime were conducted on fracture mechanics specimens of an Al-6Zn-2Mg alloy with either 2.14 or 0.06 wt.% alloyed copper in the underaged, peak aged, and overaged tempers. Crack growth rate tests were performed in 90% relative humidity (RH) air between 25 and 90°C to assure hydrogen embrittlement control. Hydrogen uptake in humid air and hydrogen diffusion and trapping were investigated via nuclear reaction analysis (NRA) and thermal desorption spectroscopy (TDS). Lastly, near crack tip hydrogen concentrations and concentration profiles were analyzed via NRA and secondary ion mass spectroscopy (SIMS) using a liquid gallium, focused ion beam sputtering source (FIB/SIMS). Increased degree of aging produced significantly lower stage II crack growth rates in 90% RH air for the copper bearing alloy over the entire temperature range investigated (25-90°C). However, only a slight decrease was observed in the low copper alloy. For identical aging conditions, the low copper variant of the alloy cracked faster than the copper-bearing alloy. The humid air fracture path is predominately along high angle grain boundaries in each case. The crack growth kinetics of each alloy and temper were well described by an Arrhenius-type rate equation. Activation energies for crack growth ranged between ~59 kJ/mol for the low copper alloy in both the peak aged and overaged tempers to ~98 kJ/mol for the copper bearing overaged alloy. Nuclear reaction analysis of HEAC crack wakes indicated that 90°C, 90% RH air produced near crack-tip hydrogen concentrations on the order of 10,000 wt. ppm. These high near-surface hydrogen concentrations were confirmed to be associated with internally dissolved and trapped hydrogen via FIB/SIMS depth profiles. Comparison of this data with experiments on the diffusion and trapping of hydrogen indicates that the observed hydrogen concentration profiles can be supported by either true lattice or trap affected volume diffusion, without the need to invoke fast path diffusion or dislocation transport. The observed stage II crack growth rates could be supported by volume diffusion if the critical distance that hydrogen must diffuse is <10 :m. Hydrogen production and uptake occurred readily on fresh metal surfaces of underaged and overaged AA 7050 exposed to 90°C, 90% RH air but were impeded by the overaged AA 7050 native oxide (formed in 20°C, 22% RH, 48 hrs). Notably, the native oxide of the highly HEAC susceptible underaged AA 7050 did not impede hydrogen production and uptake. Hydrogen diffusion experiments revealed significant differences in the true lattice diffusion coefficient as a function of alloyed copper level and aging treatment. In the copper bearing alloy, increased degree of aging (underaged to overaged) decreased the lattice diffusivity of hydrogen. In the low copper alloy, aging from the peak aged to the overaged temper had no effect on the diffusivity of hydrogen within experimental error. TDS spectra revealed that dislocations and vacancies are hydrogen trap states but the role of precipitates in the alloy is unclear. These results help explain and quantify empirically known trends concerning HEAC resistance and establish new findings. In the copper bearing alloy, overaged tempers are more resistant but not immune to HEAC. Humid air is an aggressive environment for Al-Zn-Mg alloys because water vapor reacts with bare aluminum to produce high surface concentrations of hydrogen that diffuses ahead of the crack tip. Internally dissolved hydrogen then, leads to crack extension via hydrogen embrittlement and the production of more bare aluminum surface. One reason increased degree of aging increases HEAC resistance in copper bearing 7XXX series alloys is that hydrogen diffusion is slower. Overaging has little benefit on low copper alloys because no such decrease in the diffusivity of hydrogen occurs.

4:30 PM

Internal Hydrogen Embrittlement of High-Strength Beta-Titanium Alloys: *Richard P. Gangloff*[1]; Sean P. Hayes[1]; [1]University of Virginia, Dept. of Matls. Sci., SEAS-Thornton Hall, Charlottesville, VA 22903-2442 USA

The objective of this research is to identify the dissolved-hydrogen concentration and microstructure conditions that promote brittle cracking of beta-Ti sheet microstructures, and to elucidate the crack tip damage mechanism. Experimental results show that short-term, high current density electrochemical charging followed by thermal homogenization effectively introduces low concentrations of hydrogen into beta-Ti alloys. A method using direct current electrical potential difference monitoring of crack growth and J-integral fracture mechanics accurately characterizes the effect of dissolved hydrogen on the initiation and stable growth of a precrack in a sheet compact tension specimen. Two solution treated and aged (STA) beta-Ti alloys, Ti-15V-3Mo and Ti-7Mo-5Fe-1.5Al (wt pct), are susceptible to severe hydrogen embrittlement. The threshold stress intensity (K_{TH}) for hydrogen cracking decreases monotonically, and subcritical crack growth rate increases, with increasing predissolved hydrogen concentration in the range from 50 to 1500

weight-ppm. For example, a hydrogen concentration of 400 to 500 ppm reduces K_{TH} to 50% of the as-received fracture toughness for each alloy. Larger hydrogen concentrations (800 ppm) reduce K_{TH} asymptotically to 25% of K_{IC}. Cracking is transgranular for all hydrogen contents examined. While the scale and volume fraction of alpha hinder precise determination, fractographic and metallographic analyses suggest that hydrogen promotes increasingly severe microcracking at alpha/beta interfaces. Consistent with the importance of this interface cracking, the all-beta microstructure is not susceptible to internal hydrogen embrittlement. Hydrogen-enhanced cracking only occurs in beta/alpha microstructures produced by isothermal aging in excess of a critical time. This aging-time dependence appears to be related to a critical amount of alpha precipitates that provides a near-continuous crack path across a beta grain, and perhaps high-local stresses from constraint. Alpha precipitated at beta grain boundaries does not promote hydrogen cracking. This research was supported by the NASA-Langley Research Center (Grant NAG-1-745) with W.D. Brewer and D.L. Dicus as program monitors.

4:50 PM
Environmental Effects in NiTi-Based Alloys: *J. H. Zhu*[1]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Metals & Ceramics Div., P.O. Box 2008, Bldg. 4500S, MS 6115, Oak Ridge, TN 37831-6115 USA

Environmental embrittlement has been identified as one of the major causes for low ductility and poor fracture toughness in a number of ordered intermetallics containing reactive elements, such as Al, Ti, Si, or V, when tested in moisture-or hydrogen-containing environments at ambient temperatures. Environmental effects in equiatomic NiTi alloy has not been studied so far, even though it has ordered B19' or B2 structure and is an important shape memory alloy. In this study, we report the tensile properties in different environments of several NiTi-based alloys with ordered B19' and B2 structures. No environmental embrittlement was observed for both B19' NiTi and B2 NiTi-7Fe alloys tested in moist air and gaseous hydrogen at ambient temperatures. Even though Ti is reactive enough to dissociate water vapor in air to generate atomic hydrogen and Ni can act as catalyst to dissociate H2 into atomic hydrogen, the presence of hydrogen does not cause hydrogen embrittlement for these alloys. On the other hand, a B2 NiTi-10Fe alloy is severely susceptible to environmental degradation. The possible mechanisms responsible for the different environmental embrittlement behaviors of NiTi-based alloys will be discussed.

New Opportunities in MMC Research and Applications: Aerospace and Electronics Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee
Program Organizer: Daniel B. Miracle, Wright Laboratory, Materials Directorate, Building 655, WPAFB, OH 45433 USA

Tuesday PM
November 2, 1999
Room: Salon D&E
Location: Omni Netherland Plaza Hotel

Session Chairs: Dr. Dan Miracle, Air Force Research Laboratory, AFRL/MLLM, Dayton, OH 45433-7817 USA

2:00 PM Invited
MMC's for Electronic Substrates: Dr. *Carl Zweben*[1]; [1]Composites Consultant, 62 Arlington Rd., Devon, PA 19333 USA

Electronic packaging is a rapidly growing industry, with US sales of $9 billion. There are strong drivers to reduce the weight, size and cost of new products while maintaining reliability. In addition, increasing functionality is causing severe thermal management problems. The limitations of conventional packaging materials have led to the continuing development of an increasing number of new composites that demonstrate major improvements, including: higher thermal conductivity (up to 5 times copper), tailorable coefficient of thermal expansion (CTE), minimizing thermal stresses; high stiffness and strength; lower density; weight reductions up to 90%; size reductions up to 60%; and, in some cases, lower cost. Metal matrix composites (MMCs), in particular, are making great inroads. Key packaging MMCs at this time include a number of metals reinforced with fibers and particles. A variety of processes are used to make these materials, including some that are net shape. MMCs are now being used in a growing number of high volume commercial and aerospace production applications at the rates of hundreds of thousands of piece parts annually. Examples include cellular telephones and ground stations, laptop computers, electric vehicles, and aircraft and spacecraft electronic systems. This penetration is particularly significant, considering that the development of MMC packaging technology began less than 20 years ago. Despite the great success of MMCs to date, there are needs and opportunities for new and improved materials and processes. This paper provides an overview of current packaging materials, including traditional materials, MMCs and other composites, the processes by which they are made, and applications. We also look at future directions.

2:30 PM Invited
Space Applications for MMC's: *Suraj Rawal*[1]; [1]Lockheed Martin Astronautics, P.O. Box 179, Mail Stop DC3085, Denver, CO 80201 USA

Both continuous and discontinuous fiber-reinforced metal matrix composites(MMC's) have been designed and developed for defense, aerospace, and commercial applications. These composites include B/Al, Gr/Al, Gr/Mg, Gr/Cu, SiCp/Al, SiC/Al, and SiC/Ti-6Al-4V. Before MMCs are selected for specific applications, five key factors must be considered: structural and thermal performance, weight, space durability, producibility, and cost. Performance has usually been considered more important than cost, but not always. Spacecraft applications for MMC's include the general categories of precision structures, non-precision structures, and thermal management. While MMC's have been included in several material trade studies for different applications, often they have not been down-selected because of the lack of designers' confidence and industry base. For example, the designer can buy traditional materials or polymer matrix composites to a specification, but most often MMC parts are available only on a best effort basis. A system-level perspective of MMCs for space applications is discussed in this presentation.

3:00 PM Invited
Particulate-Reinforced MMC's for High Temperature Applications: *Chris Rhemer*[1]; [1]Pratt and Whitney, West Palm Beach, FL USA
Abstract Text Not Availabe

3:30 PM
Microstructure and Mechanical Properties of In-Situ Processed Ti-TiB Composites Containing High Volume Fractions of TiB Whiskers: Satyam S. Sahay[1]; [1]University of Utah, Dept. of Metallu. Eng., 135 S. 1460 E. Rm. 412, Salt Lake City, UT 84112 USA

A series of in-situ titanium composites with varying volume fractions of titanium monoboride (TiB) whiskers, were made by mixing various proportions of titanium (Ti) and titanium diboride (TiB2) powders followed by hot pressing. The phases present were identified by X-ray diffraction. Microstructural examination revealed three different types of TiB whisker morphologies; (i) long and needle shaped TiB whiskers that are isolated and randomly oriented in the Ti matrix at relatively low volume fractions (0.3), (ii) colonies of refined and densely packed TiB whiskers from intermediate (0.55) to high volume fractions (0.73 and 0.86)

and (iii) coarse and elongated TiB particles with a few needle shaped whiskers at the highest volume fraction (0.92). In all the composites, TiB was found to be the predominant reinforcement. Mechanical properties including elastic modulus, strength as a function of temperature and ductility were evaluated on selected composites. The composite microstructure, the evolution of different morphologies of TiB whiskers and their effect on mechanical behavior are discussed. Supported by Army Research Laboratory through Contract No: DAAL01-96-C-0081.

3:50 PM

Microstructure of and Hardness Variation in Titanium-Titanium Monoboride (Ti-TiB) Functionally Graded Material: *Ravi K.S. Ravichandran*[1]; [1]University of Utah, Dept. of Metallu. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Microstructural characteristics of titanium-titanium monoboride (Ti-TiB) functionally graded material (FGM) in the Ti-B system was investigated. The FGM was synthesized in solid state and it consisted of graded layers with varying volume fractions of Ti and TiB phases in the thickness direction. Microstructure and the crystallography of constituents were investigated by optical microscopy and X-ray diffraction. While the TiB was present as isolated whiskers at low volume fractions, the whiskers were interconnected at high volume fractions. The variation of hardness as a function of distance across the graded layers was determined. Although the hardness variation followed the intended composition profile designed in the processing stage, there was substantial variation across a given layer, suggesting possible inter-diffusion of B between the layers. The microstructural and the hardness data are rationalized in terms of phase diagram information, morphology and properties of the metallic and ceramic phases. Supported by Army Research Laboratory through Contract No: DAAL01-96-C-0081.

4:10 PM

Double Cemented Carbide-An Old Metal Matrix Composite Idea Revisited: *Krishan Kumar Chawla*[1]; Burton R. Patterson[1]; Zak Fang[2]; Mark Charles Koopman[1]; Charles Parkman Coffin[1]; Bhavesh Patel[1]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., BEC Rm. 254, Univ. Station, Birmingham, AL 35294-4461 USA; [2]Smith International, Smith Tool Div., 16740 Hardy St., P.O. Box 60068, Houston, TX 77205 USA

Cemented carbide cutting tool inserts consisting of tungsten carbide (WC) particles distributed in a cobalt (Co) metal matrix have been in commercial use for a long time. They really combine aspects of a brittle ceramic and a ductile metal to produce a composite with properties that neither one of the individual components has. Although such a particulate metal matrix composite undoubtedly has been a commercial success, in this presentation we wish to explore the possibility of extending the microstructural flexibility and the range of properties. This has been accomplished by advancing the concept of a hybrid metal matrix composite consisting of discrete granular composite reinforcements in a metal matrix. We call it double cemented carbide (DCC) composite. It promises to be a general extension of the idea of composites. Some preliminary results on the processing, microstructure, and properties of such composites will be presented.

4:30 PM

Codeformation Processing of Discontinuously-Reinforced Metal/IMC Composites: *Judson S. Marte*[1]; Stephen L. Kampe[2]; Tony Zahrah[3]; [1]General Electric Corporation, Rsrch. and Dev., P.O. Box 8, Bldg K1-MB101, Schenectady, NY 12301 USA; [2]Virginia Polytechnic Institute and State University, Mats. Sci. and Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA; [3]MATSYS Inc., Springfield, VA USA

Aspects of the codeformation processing of multiphase materials were investigated to assess their influence on microstructural development. A series of composites having mechanically-dissimilar components has been produced in which the high temperature flow behavior of the reinforcement could be varied independent of the matrix. This was accomplished by the use of a series of intermetallic matrix composites (IMCs), consisting of titanium aluminide reinforced with titanium diboride particulate, used as discontinuous reinforcement within an otherwise conventional Ti-6Al-4V metallic matrix. Metal/IMC powder blends were extruded at temperatures high enough to achieve commensurately deformed microstructures. The results of the codeformation processing were analyzed in terms of the plastic strain of the IMC relative to that within the matrix. Codeformability was shown to increase with increasing IMC particle size, volume percentage of the IMC, extrusion temperature, homologous temperature, extrusion strain rate, and decreasing TiB2 reinforcement percentage within the IMC. This work was sponsored by MATSYS, Inc. and the Office of Naval Research under contract no.

4:50 PM

Manufacturing, Microstructure, and Properties of Ternary Cu-Based in Situ Composites: *Dierk R. Raabe*[1]; [1]Max-Planck-Institut fuer Eisenforschung, Max-Planck-Str. 1, Duesseldorf 40237 Germany

The paper presents recent developments in the new field of ternary in situ processed Cu-based metal matrix composites. The alloys are designed using minor additions of Ag and a high melting body centered cubic (bcc) transition metal such as Nb or Cr. The Ag occurs in the form of a solid solution in the Cu-matrix, as Cu-Ag eutectic, or as Ag precipitations. The refractory constituents have negligible solubility in the matrix and thus solidify with dendritic morphology. The composites are produced by inductive melting, casting, and subsequent wire drawing or cold rolling. Upon wire drawing the various phases form into fibers the spacing of which continuously drops with increasing strain. The composites are very ductile so that maximum wire strains in excess of h = 10 were attained. Composites with Cr as the bcc metal require an intermediate heat treatment during deformation. The paper presents microstructures and properties of various ternary composites and dicusses their potential for applications in the fields of robotics and high field magnet design.

5:10 PM

Thermodynamic Modeling of the Nb-Ti-Si-Al Quaternary System: *Haiyan H. Liang*[1]; Shuang-Lin Chen[2]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 Univ. Ave., Madison, WI 53706 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA

Phase equilibria in the Nb-Ti-Si-Al quaternary system are thermodynamically modeled using the CALPHAD methodology. The alloying effect on the microstructural constituents of the Nb-Si-based in-situ composites can now be predicted by model calculation. The predicted phase stability, phase transformations and solidification paths for the key subsystems, i.e. Nb-Si-based ternaries, are in accord with experimental data available in the literature. For the quaternary system, the calculated phase equilibria in the Nb-Ti-rich region are believed to be reliable. However, in the absence of experimental data, the calculated phase equilibria should be used with caution. It is anticipated that utilizing thermodynamic model-calculations will speed up the development of advanced composites.

Pb Free and Pb Bearing Solders: Session II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Jessica H. B. Hemond, AMP Inc., MS 018-020, Harrisburg, PA 17105-3608 USA; K. N. Subramanian, Michigan State University, Department of Materials Science & Mechanics, East Lansing, MI 48824-1226 USA

Tuesday PM
November 2, 1999

Room: Salon B&C
Location: Omni Netherland Plaza Hotel

Session Chair: Jessica H. B. Hemond, AMP Inc., Mats. Eng. & Res., Harrisburg, PA 170105-3608 USA

2:00 PM

Design of Near-eutectic Sn-Ag-Cu-X Alloys for Enhanced Solder Joint Performance: *I. E. Anderson*[1]; T. Bloomer[1]; R. L. Terpstra[1]; [1]Iowa State University, Ames Lab., 126 Metals Dev., Ames, IA 50011 USA

The dual demands of harsh operating conditions and extreme reliability on solder joints in electronic systems for applications such as automotive and avionics has driven the search for a replacement for Sn-Pb eutectic solders. Concerns about Pb as a toxic component of discarded electronic assemblies has focused the search to Pb-free solders. A new Pb-free eutectic solder for general applications, Sn-4.7Ag-1.7Cu (wt.%), and related near-eutectic alloys with a lower alloy cost, e.g., Sn-3.8Ag-0.7Cu and Sn-3.5Ag-1.0Cu, have been alloyed with minor transition metal (Co, Ni,Fe) additions to modify the as-solidified morphology and depress the growth rate on annealing of the solder/substrate (Cu) interfacial phases. The alloy design hypothesis, the effects on melting behavior and the results of microstructural analysis will be discussed along implications for enhanced solder joint performance. Support received from USDOE-BES, Materials Science Division under contract no. W-7405-Eng-82.

2:25 PM

Stress Relaxation of Lead-Free Solders: *Susheel Ganeshrao Jadhav*[1]; Dong Seo[1]; K. N. Subramanian[1]; J. P. Lucas[1]; *T. R. Bieler*[1]; [1]Michigan State University, Dept. of Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824 USA

Solders in electronic circuits experience high homologous temperatures under service conditions. Hence, time and temperature dependent creep processes play a significant role in damage accumulation and the resultant mechanical behavior of solder joints. In this investigation stress relaxation studies were conducted at constant strain levels at various temperatures using Sn-Ag solders with (a) small additions of alloying elements such as Cu, and (b) composite solders with intentionally incorporated intermetallic reinforcements. The results of these studies were compared to those of non-composite eutectic Sn-Ag solder to provide a better understanding of thermally-activated processes on the damage accumulation mechanisms.

2:45 PM

Temperature Dependence of Shear Strength and Associated Microstructural Effects in Sn-Based Pb-Free Solders: A Comparative Study: *Tamara E. Bloomer*[1]; Iver E. Anderson[1]; Robert L. Terpstra[1]; James C. Foley[1]; [1]Ames Laboratory, Metallu. and Ceramics, Iowa State Univ., 137 Wilhelm Hall, Ames, IA 50011 USA

The need for high performance Pb-free solders for the electronics industry has intensified recently due to both European Union legislation and a Japanese commercial initiative. To date, few direct comparisons of the relevant mechanical properties and the microstructural stability of current solder options have been reported. In this study, six Sn-based solder baseline alloys were compared to six variations of Ames Laboratory's Sn-4.7Ag-1.7Cu (wt. %) eutectic alloy. Shear strength measurements at room temperature, 100°C and 170°C will be reported and correlated to differences observed in the microstructure, including any aging effects. Shear strength was determined by the asymmetric four-point bend test, a new test that produces a pure shear stress state in butt solder joints. These results should provide a clear ranking of the Pb-free solder alloys from this group that are most suited for high performance applications. Support from USDOE, Office of Science under contract no. W-7405-Eng-82 is gratefully acknowledged.

3:05 PM

Current Status of Pb-Free Solders for Microelectronic Interconnects: *Darrel R. Frear*[1]; [1]Motorola, AISL, MDEL725, 2100 E. Elliot Rd., Tempe, AZ 85284 USA

Elemental Pb has been a primary constituent of low temperature solder alloys for over 3000 years but recent concern over its toxicity have resulted in proposed and, in some cases, enacted legislation to ban Pb in electronic assemblies. The response to this potential ban has been extensive research over the last 10 years on Pb-free solder alloys. This presentation will be a review of the work performed world-wide on Pb-free solders with an emphasis on recent developments. The most common alloy family for Pb-free solders found in the open literature is a variation of the eutectic Sn-3.5Ag with alloying elements of Bi, Sb, In, and Zn but other alloys such as Sn-In, Sn-Sb, Sn-Bi and Sn-Zn have also been studied. This review will include a discussion of the physical (melting, wetting, intermetallic formation, etc.) and mechanical (strength, creep, thermomechanical fatigue, etc.) behavior of promising Pb-free solder alloys based on published results. The behavior of the solders will be related to their microstructure with recommendations of optimal alloy composition as a function of performance requirements.

3:30 PM BREAK

3:40 PM

Creep and Deformation Behavior of Certain Pb and Pb-free Solder Alloys for Interconnects in MCMs: *K. Linga Murty*[1]; [1]North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA

Mechanical properties of some lead and lead-free solder alloys that are considered for application in multichip modules for various interconnects, will be described. A major objective of these studies has been to examine their reliability. Creep and deformation characteristics of lead-free alloys (In, Sn, Sn-Ag and Sn-Sb) were determined at ambient and high (homologous) temperatures from which the stress and temperature variations of the strain-rate are derived. These results will be compared with those corresponding to lead alloys (Pb5Sn and Pb62Sn). The experimental range of stress and temperature levels lead to changes in the operating deformation mechanisms which need to be considered for reliable prediction of deformation characteristics at operating conditions. In addition to tests on macroscopic samples, limited amount of work was performed on solder-bump arrays (Pb5Sn and Sn-Ag).

4:05 PM

Creep of Cu and Ag Particle-Reinforced Composite Solder Joints at Elevated Temperatures: *Fu Guo*[1]; J. P. Lucas[1]; K. N. Subramanian[1]; T. R. Bieler[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824 USA

High temperature creep is an important issue in the study of solders especially when one considers that the service temperature for solders is usually above one half of their melting point in degrees absolute. Creep properties were determined for solder joints ~ 100 micron thick. The solder joints were prepared primarily with eutectic Sn-Ag solder containing 10 v% and 20 v% of about 5 micron size Cu and Ag reinforcing particles. Creep tests were conducted at 25, 85 and 150 degrees Celsius. Qualitative and quantitative evaluation of creep behavior was carried out using the

laser-ablation pattern mapping technique. Evolution of creep strain as a function of normalized position (near interface, near the center of the joint) was quantified and microstructurally analyzed. Various creep properties such as global and localized creep strain, variation of creep strain and strain-rate, and the onset of tertiary creep were determined. Creep deformation behavior of the Cu and Ag particle-reinforced composite solders were compared with those of the non-composite eutectic Sn-Ag solders.

4:25 PM
Electromigration in Thin Strips of Eutectic SnPb Solder: *K. N. Tu*[1]; [1]UCLA, Mats. Sci. and Eng., Los Angeles, CA 90095 USA

Room temperature electromigration has been observed in thin strips of eutectic SnPb solder stressed by a current density of 10e5 amp/cm2. Hillocks and voids grow at the anode and cathode, respectively. While the dominant diffusion species is Sn in this two-phase alloy, the growth of the hillocks, surprisingly, originates from the Pb grains. A strong correlation between microstructure and electromigration has been observed. A discussion of the effect of surface oxide on vacancy flux will be given.

4:50 PM
Grain Boundary Crack Growth in Interconnects Under an Electric Current: Chao-Yuan Liu[1]; *Sanboh Lee*[1]; T.-J. Chuang[2]; [1]National Tsing Hua University, Dept. of Mats. Sci., 101 Kuang Fu Rd., Sec. 2, Hsinchu 30043 Taiwan; [2]National Institute of Standards and Technology, Ceramics Div., Gathersburg, MD 20899 USA

Failure of thin-film interconnects is a great issue in semiconductor device. Due to the high electric current density in interconnects, electromigration-induced atomic flux is recognized as one of the important mechanisms to failure of interconnect. For wide polycrystalline interconnects, atomic flux along grain boundaries is believed to be the major failure mechanism. In situ transmission observations revealed the void propagation along grain boundaries. Thus the steady state crack growth along a grain boundary in an interconnect subjected to a high current density is considered. The crack growth is achieved by the mass transport driven by surface curvature and electric field. For crack propagation not parallel to the remote electric field, the direction of electric field on one crack surface is opposite to that on the other crack surface. The governing equation is built up and then the numerical solution is solved. The crack growth rate and crack width are proportional to the applied electric field with power 1.5 and-0.5, respectively. The crack tip morphology map is divided into four regions. Case I is defined as that both crack angles are positive, Case II, one of crack angles is zero, and Case III, one crack angle is positive and the other is negative, Case IV corresponds to that crack growth is physically impossible to occur.

Processing and Properties of Structural Nanomaterials II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Powder Materials Committee, Alloy Phases Committee, Materials Processing & Manufacturing Division, ASM-MSCTS, Materials & Processing Committee

Program Organizers: Leon L. Shaw, University of Connecticut, Department of Metallurgy & Material Engineering, Storrs, CT 06269-3136 USA; Lawrence T. Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; Carl C. Koch, North Carolina State University, Department of Materials Science & Engineering, Raleigh, NC 27695 USA

Tuesday PM, November 2, 1999 — Room: Rookwood, Location: Omni Netherland Plaza Hotel

Session Chairs: Lawrence T Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; F. H. (Sam) Froes, University of Idaho, Insti. for Mats. and Adv. Proc., Moscow, ID USA

2:00 PM Invited
High-Strength Bulk Amorphous Alloys Containing Nanocrystalline Particles: *Akihisa Inoue*[1]; [1]Tohoku University, Instit. for Mats. Rsrch., 2-1-1 Katahira, Aoba-Ku, Sendai 980-8577 Japan

Since a series of discoveries of amorphous alloys with large supercooled liquid region before crystallization and high glass-forming ability in Mg-Ln-Ni-Cu, Ln-Al-Co-Ni-Cu, Zr-Al-Ni-Cu and Zr-Ti-Al-Ni-Cu systems for several years between 1988 and 1990, great attention has been paid to the formation and properties of bulk amorphous alloys. For the latest one decade, a number of bulk amorphous alloys have subsequently been synthesized in Zr-Ti-Be-Ni-Cu, Fe-(Al,Ga)-(P,C,B,Si), Pd-Cu-Ni-P, Fe-(Co,Ni)-(Zr,Hf,Nb)-B, Co-Fe-(Zr,Hf,Nb)-B and Ti-Zr-Ni-Cu-Sn systems. Among these bulk amorphous alloys, it has been clarified that the Zr-based amorphous alloys exhibit high static strength, high fatigue strength and high fracture toughness and the Fe-and Co-based amorphous alloys have good soft magnetic properties. The Zr-Al-Ni-Cu, Zr-Ti-Al-Ni-Cu and Zr-Ti-Be-Ni-Cu bulk amorphous alloys have already been commercialized as sporting goods materials with high strength and high toughness. More recently, we have found that remarkable improvement of static and dynamic mechanical strength is achieved for bulk Zr-based amorphous alloys containing nanocrystalline particles. The high tensile strength, bending strength and fracture toughness increases from 1700 MPa, 2100 MPa and 135 kJ/m2, respectively, for the bulk amorphous alloys to 1900 MPa, 4300 MPa and 165 kJ/m2, respectively, for the nanocrystalline bulk amorphous alloys. The nanocrystalline bulk amorphous alloys are formed in Zr-Al-Ni-Cu-M (M=Ag, Ti, Nb, Pd, Au or Pt) systems and the nanocrystalline particles have a size of 2 to 50 nm and an interparticle spacing of 4 to 60 nm. The finding of the further improvement of mechanical properties for the bulk nanocrystalline amorphous alloys by the dispersion of nanoscale crystalline particles also has some advantages of the decrease in cooling rate in the alloy preparation and the formation of the bulk amorphous alloy with larger scale. The nanocrystalline bulk amorphous alloys are expected to be more widely used as a new type of nonequilibrium material exceeding the bulk amorphous alloys.

2:30 PM
Applications of Zr-Based Bulk Glassy Alloys to Golf Clubs: *Masahide Onuki*[1]; Akihisa Inoue[2]; Tetsuo Yamaguchi[1]; Haruyoshi Minamiguchi[3]; Koichi Iwata[3]; [1]Sumitomo Rubber Industries Limited, Sports Goods Rsrch. Dept., 2-1-1 Tsutsui-cho, Chuo-ku,

Kobe 651-0071 Japan; [2]Tohoku University, Instit. for Mats. Rsrch., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [3]Sumitomo Rubber Industries Limited, 2-1-1 Tsutsui-cho, Chuo-ku, Kobe 651-0071 Japan

Bulk glassy alloys have high tensile strength, while the Young's modulus is lower by 20 to 40% than that for the corresponding crystalline alloys. These uniqe properties are effective to increase the coefficient of restitution at the impact between a golf club and a golf ball. Although conventional amorphous alloys have been prepared only in thin foil and ribbon forms, we have succeeded in producing a glassy Zr-Al-Ni-Cu alloy in a shell shape with a dimension of about 90 x 40mm and a thickness of 3mm by a newly developed manufacturing process. It exhibits excellent mechanical properties such as tensile strength of 1700MPa, Young's modulus of 81GPa and impact fracture toughness of 130kJ/m/m. These properties satisfy the needs for wood type golf club heads, even for the driver golf club heads. The glassy driver golf club with the shell shape in the impact region was confirmed to have a high coefficient of restitution.

2:55 PM
Interfacial Defect Structures in Nanostructured Materials: *Ilya A. Ovid'ko*[1]; [1]Russian Academy of Sciences, Instit. of Problems of Mech. Eng., Bolshoj 61, Vas.Ostrov, St. Petersburg 199178 Russia

Defect structures in the interfacial phase in nanostructured materials are theoretically examined. In particular, interfacial disclinations and dislocations, their dipoles, and interfacial dislocation-disclination networks in nanostructured films and bulk materials are theoretically described with the special attention being paid to contribution of such defect configurations to structural stability, transport and mechanical properties of nanostructured materials. The specific peculiarities of interfacial defects in nanostructured multilayer coatings are analysed.

3:20 PM Break

3:40 PM Invited
Thermal Spraying of Nanocrystalline Materials: *E. J. Lavernia*[1]; [1]University of California, Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., Irvine, CA 92697-2575 USA

Thermal spray processes, such as plasma and high velocity oxygen fuel (HVOF) techniques, have evolved into sophisticated and practical methods for the synthesis of engineering coatings. In particular, HVOF thermal spraying has attracted commercial interest for its ability to produce coatings combining high hardness and bond strength with minimal porosity, tailored thickness and surface roughness. In recent years, HVOF thermal spraying has been proven to successfully fabricate nanostructured coatings using nanocrystalline powder feedstock prepared by mechanical alloying/milling or chemically synthesis. The relatively low temperatures and short dwell time which the powder particles attain during HVOF thermal spraying appear to help preserving the nanocrystalline structure in the final coatings. High velocity oxy-fuel spraying combines complex processes of combustion and spraying combustion and gas dynamics (fluid and particle dynamics), particle flattening and solidification on the substrate surface to form a coating. The dynamic processes are further complicated for the thermal spraying of nanocrystalline materials as the particle morphology deviates from the conventional spherical powders used for thermal spraying. Ongoing research programs at UCI direct toward the exploration of nanostructured coating using HVOF thermal spray are also described. Recently, nanocrystalline Ni, Inconel 718, and 316-stainless steel feedstock powders prepared by methanol or cryogenic milling were thermally sprayed using Sulzer Metco DJ 2600 HVOF system and the resulting coatings remain nanocrystalline. Microhardness measurements performed on the nanocrystalline Ni, Inconel 718, and 316-stainless steel (methanol milled for 10 hours) coatings yield microhardness values that are approximately 20%, 60% and 36% higher than those of conventional sprayed coatings when air was used as a carrier gas during thermal spraying. Furthermore, the hardness values increase with increasing aspect ratio which suggest the morphology of the particles influences the properties of the resulting coatings. Further study is underway to investigate this phenomenon.

4:10 PM
Wear Behavior of Al2O3-TiO2 Coatings Thermally Sprayed Using Nanostructured Powders: Daniel Goberman[1]; *Leon L. Shaw*[1]; [1]University of Connecticut, Dept. of Metallu. and Mats. Eng., Storrs, CT 06269 USA

Wear behavior of Al2O3-13 wt.% TiO2 coatings formed via plasma spray approach using reconstituted nanosized Al2O3 and TiO2 powder feeds are described. The oxide coatings have been sprayed using various spray conditions including different nozzles. Sliding wear against silicon nitride and erosion wear under silicon carbide particle streams are evaluated. It is found that the wear resistance of the coatings formed using nanostructured powders is better than that of the counterparts formed using commercial powders. The wear behavior of these nanostructured coatings are discussed based on their microstructure, grain size, phase content and microhardness.

4:35 PM
Synthesis of Nanostructured Cr3C2-25(Ni20Cr) Coatings: *Jianhong He*[1]; Michael Ice[1]; Enrique J. Lavernia[1]; [1]University of California Irvine, Chem. and Biochem. Eng. and Mats. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA

With nanocrystalline Cr3C2-25 (Ni20Cr) feedstock powders produced by mechanical milling, a nanostructured coating has been synthesized using high velocity oxygen fuel thermal spraying. The properties of the nanostructured were compared to those of the conventional coating of the same composition using SEM, TEM and microhardness tests. The nanostructured Cr3C2-25 (Ni20Cr) coating synthesized in this study had an average carbide particle size of 24 nm. Discontinuous elongated amorphous phases were observed in the nanostructured coating. The conventional Cr3C2-25 (Ni20Cr) coatings of blend powder often had inhomogeneous microstructure. While uniform microstructure was observed in the nanostructured Cr3C2-25 (Ni20Cr) coating, uniformity of microstructure in the nanostructured coating was thought to attribute to individual synthesis process. The nanostructured Cr3C2-25 (Ni20Cr) coating yielded an average microhardness value of 1020 DPH300, which showed a 20% increase in microhardness from the conventional coating. The nanostructured Cr3C2-25 (Ni20Cr) coating had higher apparent fracture toughness than the conventional partner. The apparent mechanical property improvements in the nanostructured coating were resulted from the uniformity of microstructure and the intrinsically high performance of nanostructured materials. In addition, milling mechanism of powder system with hard particle and toughness binder duplex structure was also discussed. Key words: Nanostructured coating, amorphous phase, mechanical milling and HVOF.

P/M: Current Research and Industrial Practices: Combustion Synthesis, Shock Synthesis and Densification

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee

Program Organizers: F. D. S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgy Engineering, Rapid City, SD 57701-3995 USA; Enrique V. Barrera, Rice University, Metal Engineering & Materials Science Department, Houston, TX 77251 USA; G. M. Janowski, University of Alabama, Department of Materials & Mechanical Engineering, Birmingham, AL 35294-4461 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM — Room: Caprice 2&3
November 2, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Fernand D. S. Marquis, South Dakota School of Mines, College of Mat. Sci. and Eng., Rapid City, SD USA; Karl P. Staudhammer, Los Alamos National Laboratory, MS G770, Los Alamos, NM USA

2:00 PM Keynote
Combustion Synthesis-Densification of Metal-Ceramics Composites: *M. A. Meyers*[1]; Eugene A. Olevsky[2]; E. R. Strutt[1]; [1]UCSD, Dept. of AMES, Mail Code 0411, La Jolla, CA 92093 USA; [2]San Diego State University, Dept. of Mech. Eng., San Diego, CA USA

Combustion synthesis (also known as SHS) presents a bright potential for the synthesis of compounds with high degree of purity. However, for many reactions, the product is highly porous, and it must either be pulverized for subsequent densification, or densified while it is still hot and ductile. Densification has successfully been applied to a number of ceramic and metal-ceramic systems by (a) a high-speed forging technique, and (b) by a quasistatic pressing technique using a granular pressure transmitting medium. The quasistatic technique uses a graphite-alumina powder mixture which completely envelops the material being synthesized. The reactive mixture and pressure transmitting medium are placed in a piston and cylinder setup and the system is pressurized by uniaxial compression at a preestablished time after reaction completion. The state of stress is close to isostatic and the process is therefore termed quasi-isostatic pressing QIP Modeling of the densification was carried out using the Skorohod constitutive equation incorporating both densification and viscous effects; comparison of the results with experimental distortion obtained in indentation experiments enables obtaining equation parameters. The distortion undergone by the combustion synthesis products during QIP densification was modeled using the appropriately verified constitutive equation and assuming a linear elastic response for the granular pressure transmitting medium. Research supported by ARO MURI Program and by NSF Institute for Mechanics and Materials.

2:35 PM Invited
Recent Developments in Combustion Synthesis of Nanosize Silicon Nitride and Silicon Carbide Powders: *J. A. Puszynski*[1]; [1]South Dakota School of Mines and Technology, College of Mat. Sci. and Eng., 501 E St. Joseph St., Rapid City, SD 57701 USA

Synthesis of ceramic and intermetallic materials in a self-sustaining regime has been explored for more than twenty years. The main drawbacks of this approach are lack of homogeneity and relatively wide particle size distribution of resulting products. Recently, a significant progress has been made to control both phase composition and particle size of combustion synthesized ceramic powders. It has been demonstrated that an addition of certain volatile compounds such as HF, HCl, NH4HF2, NH4Cl, NH4F, KClO3, and Teflon, changes the overall mechanism of the reaction and leads to a formation of nanosize particles. Recently, both a-silicon nitride and silicon carbide with average particle size below 200 nm were synthesized in a self-sustaining regime by a nitridation of silicon at elevated nitrogen pressures and by a direct reaction between silicon and carbon at elevated argon pressure, respectively. Analysis of reaction conditions and product characterization will be presented in detail. Technological aspects of this new synthesis technique, issues associated with a reactor scale-up, and environmental impacts will be discussed as well.

3:05 PM Invited
Shock-Consolidation and Reaction Synthesis of Titanium-Silicon Ternary Carbide: *Naresh N. Thadhani*[1]; Jennifer Jordan[1]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

We will report on our work on shock consolidation of pre-alloyed Ti3SiC2 powder as well as reaction synthesis of shock-densified powder precursors to form the titanium-silicon ternary carbide phase. The shock-compression experiments were performed using a 80-mm diameter single-stage gas gun capable of fabricating three 12-mm diameter by 4-mm thick samples or one 50-mm diameter by 8-mm thick sample. The shock-loading pressure for impact experiments performed at velocites of 500-900 m/s were computed using AUTODYNE-2D numerical simulation. The consolidated pre-alloyed Ti3SiC2 samples revealed retention of the ternary phase with a microstructure showing extensive plastic strain, corresponding to a dislocation density of cold-rolled metals. Samples of the reacted shock-densified powder precursors showed formation of the Ti3SiC2 phase under conditions of defect-enhanced solid-state diffusion. In this paper we will describe the experimental approach, microstructural characteristics, and deformation behavior of shock-consolidated as well as reaction synthesized Ti3SiC2 ternary carbide. (Funded by ARO Grant No. DAAG55-98-1-0161)

3:35 PM Break

3:50 PM Invited
Advances in Shock-Induced Synthesis and Densification of Metal Silicides: *F. D. S. Marquis*[1]; S. S. Batsanov[2]; [1]South Dakota School of Mines and Technology, College of Mats. Sci. and Eng., Rapid City, SD 57701 USA; [2]Center for High Hydrodynamic Pressures, Moscow 141570 Russia

Previous work has established that the fundamental mechanisms controlling the chemical synthesis and densification of metal silicides are dominated by processes ocurring during the pulse rise (nanoseconds) and during the peak pressure state (microseconds). These processes lead to enhanced reactivity through shock induced particle: deformation, fragmentation, interpenetration and mixing and considerable temperature increases. This paper discusses the microstructures obtained in metal silicides a function of the chemical nature and characteristics of the powder mixtures, the experimental configurations, and the shock parameters.

4:15 PM
Densification Characteristics of Combustion Synthesized Alpha and Beta Sialons: R. R. Korlahalli[1]; A. DeGraw[2]; F. D. S. Marquis[1]; *J. A. Puszynski*[1]; [1]South Dakota School of Mines and Technology, College of Mats. Eng., Rapid City, SD 57701 USA; [2]Novel Technologies Inc., Morristown, TN 37814 USA

It has been demonstrated that both alpha and beta sialons can be successfully synthesized in a self-sustaining regime at elevated nitrogen pressures. The initial composition of reactants, their particle size as well as a degree of dilution with a final product of the reaction are very detrimental factors influencing the quality of synthesized materials. Combustion characteristics during the synthesis of various sialons will be presented. A special attention will be paid to a formation of yttria-and lithia-based a-sialons. There is a wide range of compositions, which leads to a complete development of solid solutions. The yttria-based sialons were combustion

synthesized with the yttria content varying between 0.2 and 0.8, whereas the content of lithia in lithia-based sialons varied between 0.2 to 1.2. All syntheses were carried out at nitrogen pressures above 25 atm. Series of scale-up experiments were conducted to find the optimum synthesis conditions. Combustion synthesized sialon powders with various compositions were sintered under nitrogen atmosphere at 1 atm without any sintering additives. Both densification characteristics and mechanical properties of densified sialons will be presented as well.

4:40 PM
Physical Properties of Combustion Synthesized Oxide Powders: *Joanna McKittrick*[1]; [1]University of California at San Diego, Dept. of Appl. Mech. and Eng. Sci. and Mats. Sci. Pgm., La Jolla, CA 92093-0411 USA

Rapid exothermic reactions are simple and practical techniques to form single phase, fine crystallite size materials in the as-synthesized condition. Combustion synthesis of metal nitrates (oxidizers) and a carbonaceous fuel is one such technique in which a self-sustaining, exothermic reaction can be ignited at relatively low temperatures. The combustion reaction is initiated in a muffle furnace or on a hot plate at a temperature < 500°C, much lower than the phase transition of the target material. In a typical reaction, the precursor mixture of water, metal nitrates and fuel decomposes, dehydrates and conflagrates after about 3-5 minutes. The resultant product is a voluminous, foamy powder that occupies the entire volume of the reaction vessel, typically a 300-mL Pyrex dish. This talk will discuss the synthesis of nanocrystalline, complex oxide compositions such as BaTiO3, LaFeO3, (Y1-xTbx)3Al5O12,(Y1-xCex)2SiO5, and (Ba1-xEux)MgAl14O23.The physical properties of the as-synthesized product are a function of the fuel/oxidizer ratio, type of fuel, position in reaction dish, precursor water content and heating rate. The adiabatic flame temperature is maximized when the fuel/oxidizer ratio is stoichiometric. Fuels that produce more gaseous products result in higher surface area, smaller crystallite size powders.

5:05 PM
Combined MA and CCS in the TiH2-B System: *S. Ozbilen*[1]; Abdulkadir Gullu[2]; [1]Technical University of Gazi, Metallu. and Mech. Eng., Ankara Turkey; [2]Gazi University of Tech. Education, Mech. Edu. Dept., Ankara Turkey

Combined unreactive mechanical alloying (MA) and controlled combustion synthesis (CCS) was carried out in the TiH2-B system. Samples with different TiH2 levels were studied to investigate the influence of the particle size of reactants on the level of exothermicity of the self-propagating reactions together with the other effects of mechanical alloying on CS technique. In the first step homogeneous mixtures of samples #1 to #3 were converted into fine, crystalline forms without compound formation. For this purpose, unreactive mechanical alloying for up to 8 hours of time under Ar with forced air cooling to keep the temperature at RT were utilised. MA'ed powder samples were then examined by XRD and SEM study. Homogeneously mixed, unreactively MA'ed and then cold-compressed pellets of samples #1-#3 (green compacts) were subsequently combustion synthesized in controlled fashion and in thermoexplosion mode under vacuum to develop Ti-boride compounds formation (TiB and TiB2 depending on the composition). Thermal analysis under vacuum by DTA was carried out on the green compacts. XRD and SEM investigation were used for the examination of unreactively MA'ed, cold-pressed, and as-reacted pellets via CS.

Refractory Metals & Alloys: A Symposium on Research, Development & Applications IV: Ta, W and Their Alloys

Sponsored by: Structural Materials Division, Refractory Metals Committee, Corrosion and Environmental Effects Committee
Program Organizers: Mehmet Uz, Lafayette College, Chemical Engineering, Easton, PA 18042-1775 USA; Ken Natesan, Argonne National Laboratory, ET/212, Argonne, IL 60439-4838 USA

Tuesday PM
November 2, 1999
Room: Caprice 1&4
Location: Omni Netherland Plaza Hotel

Session Chairs: Mehmet Uz, Lafayette College, Chem. Eng. Dept., Easton, PA 18042 USA; Christopher A. Michaluk, CABOT Performance Materials, Technical Marketing, Boyertown, PA USA

2:00 PM Invited
Characterization and Modeling of Tantalum Textures and Resultant Plastic Anisotropy: *John F. Bingert*[1]; Paul J. Maudlin[2]; George T. Gray[3]; [1]Los Alamos National Laboratory, Mats. Tech. Metallu., Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Textures in tantalum and tantalum alloy, their evolution, and their effects on deformation have been investigated with an emphasis on accurately predicting the anisotropic plastic response from textured polycrystals. The effect of deformation variables, such as strain path and slip modes, on wrought tantalum textures was experimentally and computationally explored. Deformation textures, derived from restricted crystallographic slip conditions that generate lattice rotations, can be modeled as a function of the prescribed deformation gradient. Polycrystal plasticity was simulated in this manner using either a modified Taylor approach or a self-consistent model. In addition to texture evolution, the models were used to probe the five-dimensional yield surface. The anisotropy of plastic flow was then described by a yield function constructed from either a piece-wise or quadratic fit of the five-dimensional surface. Plastic anisotropy was experimentally investigated by quasi-static compression and Taylor cylinder impact testing. Comparison of experimental impact-interface footprints to those from continuum code calculations incorporating plastic anisotropy show generally good agreement.

2:35 PM Invited
Experimental Investigation and Simulation of the Rolling and Annealing Textures of Tantalum: *Dierk R. Raabe*[1]; [1]Max-Planck-Institut fuer Eisenforschung, Max-Planck-Str. 1, Duesseldorf 40237 Germany

The paper gives an overview of the textures and microstructures observed in rolled and annealed Tantalum. Particular attention is drawn to the evolution of the rolling textures as a function of strain, to the temperature dependence of the annealing textures, to the influence of recovery on the annealing textures, and to the spatial inhomogeneity of texture and microstructure. The experimental investigations are made using quantitative texture analysis and metallography. The deformation simulations are conducted using Taylor-Bishop-Hill theory. The simulations of recovery and recrystallization are made using a cellular automaton approach. The simulations are in good accordance with experimental results.

3:10 PM Invited
Texture and Microstructural Development in Cold-Rolled and Annealed Pure Tantalum: *Hao Zhang*[1]; Rick O. Eller[1]; [1]Tosoh SMD Inc., 3600 Gantz Rd., Grove City, OH 43123 USA

The texture and microstructural development in cold-rolled and annealed pure tantalum plates was studied by using X-ray and metallography. Pole figures were measured at various rolling and annealing stages, and the orientation distribute functions (ODFs) at each stage were calculated using the POPLA package with Roe's notation for the three Euler angles, i.e., psi, theta and phi. Pure Ta billets were firstly cross-rolled cold into plates with a 55% reduction, which resulted in a mixture of {100}<100>, {100}<310>, {100}<110> and a weak {111}<211> textures. The Ta plates were then either continually cross-rolled to total a reduction above 90%, or underwent an intermediate annealing followed by further cross-roll to a total reduction above 90%. Finally all the worked Ta plates were annealed and fully recrystallized. The as-rolled Ta with the intermediate annealing showed strong dual {100}<100> and {111}<211> textures. The recrystallization texture consisted of a predominately {111}<uvw> fiber texture and a secondary {100}<210> rolling texture. The annealing was found to enhance the {111} texture. For the heavily worked Ta plates without undergoing the intermediate annealing, the texture consisted of {100}<210> and {100}<100> rolling textures, and {111}<uvw> near fiber texture. After final annealing, the fully recrystallized microstructure demonstrated a strong {111}<uvw> fiber texture and a secondary {100}<310> rolling texture. The texture consistency across the plates and the texture gradient through the depth was also studied and the results will be discussed.

3:30 PM Break

3:45 PM Invited
On Quantifying the Texture of Wrought Tantalum: *Christopher A. Michaluk*[1]; Paul S. Prevey[2]; David P. Field[3]; [1]Cabot Performance Materials, 144 Holly Rd., Boyertown, PA 19512-1607 USA; [2]Lambda Research, Cincinnati, OH USA; [3]TexSEM Laboratory, Draper, UT USA

Tantalum wrought forms are finding greater use in demanding applications where crystallographic texture directly influences the product performance. However, characterizing the texture of tantalum plate is not a trivial matter. Intrinsically, this highly dense refractory metal readily absorbs x-rays; furthermore, commercially produced tantalum plate stock has been found to exhibit sharp texture bands atop a through-thickness texture gradient. As such, conventional methods, which rely on x-ray diffraction to measure pole figures about the sample-normal direction, fail to provide quantitative information on the bulk texture of tantalum. Although neutron diffraction is a practical means for determining the global texture of tantalum, its use has been limited due to its costs and the availability of beam time. This paper describes two commercially viable alternative methodologies for quantifying the texture of tantalum: analysis of a composite sample by x-ray diffraction, and Orientation Imaging Microscopy. Each technique offers unique benefits, which are dependent on the end-use of the material being analyzed.

4:05 PM Invited
Engineering Tantalum Sputtering Targets for Use in the Damascene Process: *Christopher A. Michaluk*[1]; [1]Cabot Performance Materials, 114 Holly Rd., Boyertown, PA 19512-1607 USA

The damascene process is the next-generation semiconductor fabrication technique that is expected to utilize sputter deposited Ta or TaN to serve as the diffusion barrier between the Si substrate and the copper conductors and plugs. Challenges to the capabilities of conventional physical vapor deposition capabilities being posed by this emerging technology, while being address in part through an evolution in equipment design, will also require improvements in sputtering target materials. This paper discusses the role of purity, microstructure, and texture in tantalum targets as dictated by these future semiconductor applications. Recent advancements in tantalum target material technology, driven by the needs of the damascene process, are also presented.

4:25 PM Invited
Creation and Processing of Super Strengthened Tubes for High Temperature and Pressure Work Conditions: *Oleksandr Illich Dekhtyar*[1]; [1]Kurdumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Inhomogeneous Alloys, 36 Vernadsky St., Kiev 252142 Ukraine

The physical and technological foundations, equipment and instruments were elaborated for production of super strengthened effective cylindrical 10-20 mm diameters and 1-2 mm thickness tubes from tungsten single crystals. These tubes have a set of unique properties: unchanged crystallographic orientation of cylindrical surface (110) with maximal electron work function, impermeability for nuclear decay products, super high creep resistance at high temperatures and inner pressure. In TIC application tubes have longevity about 100,000 hours. A method of tubes production is based on oriented strips' twisting and spiral joints' welding. It was found the effect of dislocation creep exhaustion and diffusion creep role increasing during long time creep tests.

4:45 PM Invited
Mechanical Behavior of W-5%Ta Alloy: *Jian Hu*[1]; Jeffrey S. King[1]; Ralph Zee[1]; [1]Auburn University, Mats. Rsrch. and Educ. Ctr., 202 Ross Hall, Auburn, AL 36849 USA

W-5%Ta single crystals about 10 mm in diameter and 50 mm in length have been grown at Auburn University using electron beam zone melting technique. No significant segregation of tantalum was observed in the single crystals along growth direction and transverse direction. Tensile and compression specimens were prepared using electrical discharge machining and followed by electrical polishing to obtain satisfied surface condition. Typical tensile specimens have a gage length of 6-8 mm and a gage cross-section of 1.2x2.0 mm^2 before testing, and typical compression specimens have a diameter of 4.6 mm and a L/D ratio of 1:1. Testing was performed at room temperature for tensile test and at elevated temperature between 1273K and 1773K in high purity Ar atmosphere for compression test. The mechanical behavior of W-5%Ta single and poly-crystals has been investigated as a function of starting microstructure, testing temperature, orientation and strain rates, and compared with that of pure tungsten. The deformation behavior of the alloy has also been investigated using dislocation analysis and surface slip trace analysis. The results indicate that 5 percent tantalum addition remarkably strengthens tungsten, especially at elevated temperatures, and appears to improve the ductility of single crystalline tungsten at room temperature.

Symposium to Honor Professor Julia R. Weertman: Mechanical Metallurgy

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Tuesday PM
November 2, 1999
Room: Rosewood
Location: Omni Netherland Plaza Hotel

Session Chairs: Campbell Laird, University of Pennsylvania, Philadelphia, PA USA; Richard J. Arsenault, University of Maryland, College Park, MD USA

2:00 PM Invited
Vacancy-Producing Mechanism in B2 Structured Matrix Composites: K. Xu[1]; *R. J. Arsenault*[1]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., Metallu. Mats. Lab., College Park, MD 20742-2115 USA

The intermetallic compound, NiAl, has many attractive properties as a high temperature,structural material. However, its lack of creep resistance prevents practical applications. Adding ceramic reinforcements, such as TiB2 particles, Al2O3 particles or whiskers can significantly improve the strength of binary NiAl at high temperatures. However, the increase in the yield stress of the discontinuous NiAl matrix composites as compared with monolithic NiAl is difficult to explain. The purposes of this research were to understand the deformation mechanisms which cause the increase in strength achieved by adding TiB2 particles, A12O3 particles or whiskers to NiAl, and to recognize the principles of the deformation process in NiAl matrix composites. In order to accomplish this objective, mechanical properties and thermal activation parameters in NiAl matrix composites with different types, shapes and sizes of reinforcements have been systematically evaluated. Microstructures and dislocation structures in NiAl matrix composites have also been thoroughly characterized before and after deformation. It was found that the size of the reinforcement had a large influence on the microstructures of the composites, and the nominal activation energies for all the composites were the same and within the range of the activation energy of self-diffusion for pure NiAl. This indicated that the deformation mechanism was the same for all NiAl matrix composites. The thermally activated motion of jogged screw dislocations is postulated as the rate controlling mechanism of the deformation of NiAl matrix composites. However, this non-conservative motion of jogged screw dislocation theory requires only the appearance of vacancy producing (VP) jogs. A simple model which is based on the cross slip of screw dislocations in NiAl is proposed to account for the occurrence of VP jogs. A formulation of strain rate vs. stress for the VP jogged screw dislocation model was derived. By computer simulation, it was found that this mechanism was capable of predicting the temperature dependence of the yield stresses of NiAl composites. It was further concluded that the reinforcement addition only increased the non-thermally activated component of the yield stress.

2:30 PM
Experiments on Fracture Toughness of Thick-Wall Cylinder for Modes I, II, III: *Toshiari Saegusa*[1]; Namio Urabe[2]; Chihiro Ito[1]; Kohji Shirai[1]; Akio Kosaki[1]; [1]Central Resaerch Institute of Electric Power Industry (CRIEPI), Transport and Storage Team, 1646 Abiko, Nuclear Fuel Cycle Back-End Project, Abiko-city 270-1194 Japan; [2]KKS, 1-1 Minamiwatarida, Kawasaki-city, Kanagawa 210-0855 Japan

Generally, fracture toughness of metallic structure is evaluated sufficiently for a fracture Mode I where a tensile stress is applied normal to the faces of a crack. Whereas, real structures under accidental conditions may be also subjected to Mode II fracture where a shear stress is applied normal to the leading edge of the crack or Mode III fracture where a shear stress is applied parallel to the leading edge of the crack. Normally, it is assumed that general structures are assumed not to fracture under Mode II nor Mode III unless they fracture under Mode I condition. But supporting data for this assumption are not available from the literature. This paper shows experimental results of fracture toughness of Ductile Cast Iron (ASTM A-536) and Forged Steel (ASTM A350Gr.LF) for Modes I, II, and III at a temperature range between 73K and 298K. The results showed a following relationship. JIC < JIIC < JIIIC (Eq.1) Under small-scale yield conditions, we obtain the following relationship, since J-integral values are proportional to the square of fracture toughness values, KIC < KIIC < KIIIC (Eq.2) Using above experimental data and relations, fracture toughness of a thick-wall cylinder made of Ductile Cast Iron subjected to accidental drop conditions were evaluated analytically and demonstrated by experiments. The dimension of the thick-wall cylinder is as follows. Total length: 4.7m Outer diameter; 2.4m Inner diameter; 1.5m Wall thickness; 0.45m Total weight; 110tons Prior to the drop tests, an artificial flaw was introduced on the outer surface of the cylinder where the maximum stress will be induced by the drop test. For Mode I test, the cylinder was cooled down to 233K and dropped from a height of 9m on to an unyielding target. For a mixed Modes (II and III) test, the cylinder at 233K was dropped onto a mild steel bar of 0.2m in diameter and 0.3m in length. In both cases, no crack propagation was observed from the artificial flaw, and thus the integrity of the thick wall cylinder was demonstrated under accidental drop conditions.

2:45 PM
Micromechanics of Stress-Induced Martensitic Transformation in Mono-and Polycrystalline Shape Memory Alloys; Ni-Ti: Yuanchang Liang[1]; *T. Mori*[1]; Minoru Taya[1]; [1]University of Washington, Dept. of Mech. Eng., P.O. Box 352600, Seattle, WA 98195-2600 USA

Using a micromechanics approach, we first examine stress induced transformation in single crystals, by finding particular combinations (combined variants) of two basic (primitive) martensite variants which make the elastic energy vanish, when formed in thin plates. Under external stresses, the combined variants which a loading device produces with the least work are formed. Combined with the chemical energy change associated with the transformation and the energy dissipation needed to advance the martensite-parent phase interface, the external stress required to induce martensitic transformation in single crystals is determined, using an energy balance method. The stress state for the transformation to occur is, in general, on the boundary in the stresses-temperature diagram. The above method is extended to a polycrystal. The transformation strains (and geometry) of the combined variant, which is formed under stress, differ from grain to grain. The difference between the transformation strains and the amount of transformation develops internal stresses, which are evaluated with the Eshelby inclusion theory with an average field method. The amount of the transformation, internal stress and macroscopic strain are determined as a function of an external stress. A polycrystalline specimen is modeled as a material consisting of spherical grains with multiple fiber textures. A uniaxial stress is applied along the common axis parallel to the fiber axes of the textured grains. The uniform distribution of fiber textures of up to 25 different fiber axes are examined. It is found that after a short transition stage, all the grains transform under a load. The external uniaxial stress which induces this global transformation can be defined as the macroscopic yield (transformation) stress of a polycrystal. The stress saturates as the number of different textured grains increases. The

saturated stress is 1.18 times larger both in tension and in compression than the stress needed to induce the transformation in the single crystals, which transform most easily under uniaxial loading.

3:00 PM

Low-Cycle Fatigue Deformation Characteristics of Haynesâ HR-120â Alloy: *P. K. Liaw*[1]; Yuehui He[1]; L. Miller[1]; M. Huang[1]; C. R. Brooks[1]; R. R. Seeley[2]; D. L. Klarstrom[2]; B. Yang[1] [1]University of Tennessee, Dept. of Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International Inc., 1020 W. Park Ave., P.O. Box 9103, Kokomo, IN 46904-9013 USA

Low-cycle fatigue deformation characteristics of HAYNESâ HR 120â alloy at room and high temperatures were studied under axial strain control. Test results show that there is a significant effect of test temperature on the low-cycle fatigue behavior of HAYNESâ HR-120â alloy. It was found that the alloy could cyclically harden at moderately high temperatures (649°C and 871°C), but generally cyclically soften at room temperature (24°C) and high temperature (982°C). However, the variation of the stress amplitude with cycles at the temperatures of 24°C and 982°C depended on the total strain range. The significant cyclic hardening of the alloy occurred at the high total strain ranges of 1.5% and 2.0% at the beginning of the test at both 24°C and 982°C. Microstructural analyses indicated that the cyclic hardening behavior of the alloy at the test temperature of 649°C could be related to the formation of a number of deformation bands. Nevertheless, increasing the test temperature to 871°C, cyclic hardening was attributed to the precipitation of secondary-phase particles. Furthermore, it was also found that the coarsening of secondary-phase particles brought about cyclic softening of the alloy at the high temperature of 982°C. Coffin-Manson equations and Holloman equations were given for HAYNESâ HR-120â alloy at different temperatures.

3:15 PM

Indentation Size Effect: Indentation Creep and Strain Gradient Plasticity: *Donald S. Stone*[1]; *Abdelmageed A. Elmustafa*[1]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 US

The indentation size effect (ISE) of aluminum and a-brass was investigated. Samples with different levels of bulk work hardening and different surface preparations were tested. We demonstrate, using the Haasen plot formalism (mH versus H), the effects of work hardening on the strain rate sensitivity; these effects may be taken as a kinetic signature representative of dislocation strengthening mechanisms. We demonstrate that the ISE gives rise to the same kinetic signature as does work hardening and mechanical polishing. We conclude, therefore that the ISE is exclusively related to a dislocation mechanism. The data fit the strain gradient plasticity model (e.g., Fleck et al [1993], Poole et al [1996], and Ma and Clarke [1995]) well; furthermore, dynamic recovery has no direct effect on the magnitude of ISE and thus on the accumulation of geometrically necessary dislocations.

3:30 PM

J-Integral Design Curve for Safety Assessment of Casks: *Namio Urabe*[1]; A. Kosaki[2]; T. Saegusa[2]; [1]Kokankeisoku KK, 1-1 Minami watarida, Kawasaki-ku, Kawasaki 210-0855 Japan; [2]CRIEPI, 1646 Abiko, Abiko 270-1194 Japan

International Atomic Energy Agency regulates "Safe design of shipping packages against brittle fracture" for cask materials to prevent catastrophic failure under hypothetical accident conditions, such as 9-meter free drop test at-40 C. The regulation is, however, based on linear elastic fracture mechanics. Cask (spent nuclear fuel transport and/or storage container) materials, e.g., ductile cast iron (ASTM A847) or forged steel (ASTM A350 Gr. LF), exhibit ductile manner under impact loading conditions at and even below-40 C. Therefore, safety assessment methodologies on the basis of non-linear or elastic-plastic fracture mechanics are desirable. J-intergral values on large test panels (cut from cask bodies) with artificial surface cracks were empirically measured by means of strain-gauges and photo-elasticity techniques under both uniaxial and combined (tension and bending) loading modes. And results are compared with those of finite element calculations and also existing similar design curves.

3:45 PM Break

4:00 PM

Development of the Electrochemical Fatigue Sensor for Evaluating Fatigue Damage: Y. F. Li[1]; J. Wang[1]; M. Z. Wang[1]; J. DeLuccia[1]; *Campbell Laird*[1]; [1]University of Pennsylvania, School of Eng. and Appl. Sci., Philadelphia, PA 19104-6272 USA

The Electrochemical Fatigue Sensor (EFS) is a device which operates by an electrochemical-mechanical interaction and which can sense the type and extent of fatigue damage both before and after crack initiation. It was initially explored through studies on soft metals. Here we report efforts to determine the ability of the device to read damage in hardened commercial alloys: 7075 aluminum alloy, 4130 steel and Ti-6Al-4V, which one might reasonably expect to be most difficult to evaluate because damage tends to develop late in the fatigue life. Nevertheless, the EFS follows the damage and detects small cracks at sizes hitherto beyond the range of conventional NDE techniques. We also demonstrate that the device, which uses an electrolytic medium, does not degrade the fatigue properties of the material being evaluated if care is used in electrolyte selection and in choice of electrochemical conditions.

4:15 PM

Predicted Influence of Materials' Thermal Properties on Disc Brake Roughness: *Rena L. Hecht*[1]; D. L. Hartsock[1]; R. B. Dinwiddie[2]; W. D. Porter[2]; [1]Ford Motor Company, Ford Rsrch. Lab., Vehicle Safety Rsrch. Dept., MD 2115, SRL, Dearborn, MI 48121-2053 USA; [2]Oak Ridge National Laboratory, High Temp. Mats. Lab., Oak Ridge, TN 37831 USA

Passenger car disc brakes occasionally exhibit objectionable vibration during operation. Low frequency vibration (< 100 Hz) that occurs when the brakes are applied is called "brake roughness" or "judder" and is caused by a variation in torque at the friction interface. Brake torque variation can result from disc thickness variation and/or thermal distortions. Thermal distortions or hot spots may occur during a long brake drag such as a mountain descent due to the onset of thermal elastic instability (TEI). Models of TEI can be used to predict the propensity of a brake system to generate torque variation or roughness during long brake drags. TEI modeling requires accurate materials properties such as Young's modulus, Poisson's ratio, specific heat, thermal diffusivity, thermal conductivity, density and thermal expansion of both the disc and the brake pad materials. We have measured the thermal transport properties of a series of disc materials, gray cast irons and aluminum metal matrix composites, and several commercially available brake pad friction materials. For example, thermal diffusivity measured by the laser flash technique was found to vary from 0.002-0.009 cm2/sec for friction materials, 0.15-0.21 cm2/sec for gray cast irons, and 0.43-0.89 cm2/sec for aluminum metal matrix composites. These materials properties were used in a TEI finite element model of a mid-sized passenger car disc brake. Parametric analysis was done to understand the influence of materials properties on TEI. In particular, the parametric analysis shows that thermal properties such as diffusivity of the disc material affects the speed at which TEI and brake roughness are predicted to occur.

4:30 PM

Fatigue Crack Initiation Mechanism in ULTIMET® Alloy: *Liang Jiang*[1]; Charlie R. Brooks[1]; Peter K. Liaw[1]; Dwaine L. Klarstrom[2]; [1]University of Tennessee Knoxville, Mats. Sci. and Eng., Dougherty Eng. Bldg. 323, Knoxville, TN 37996-2200 USA; [2]Haynes International Inc., 1020 West Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

The process of fatigue crack initiation in ULTIMET® alloy, a Co-26Cr-9Ni (wt %) superalloy, by room temperature cyclic deformation, was studied. The microstructure of ULTIMET® alloy possessed relatively fine, uniform grains with annealing twins. The

microstructure was essentially an unstable face-centered-cubic (fcc) phase, from which the fcc phase to hexagonal close packed (hcp) phase transformation may be easily triggered at room temperature by mechanical deformation. Fatigue testing was coupled with optical microscopy, electron microscopy, and an acoustic emission (AE) system, which is a nondestructive evaluation technique, to study the crack initiation behavior. AE provides good indications for the fatigue damage of ULTIMET® alloy. Observations of the cracks at the electropolised specimen surface and studies of the fractography indicated that the cracks initiate from the sample surface and then propagate through one grain due to slip. The macrocracks subsequently traverse through the material due to stress concentrations from the initiated microcracks. The striations on the sample surface are periodically spaced as the result of the cyclic deformation. A model is proposed to explain the observed striation spacing. Note that ULTIMET® is a registered

Daily Personal Schedule

Wednesday, November 3, 1999

Time	Session Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
10:30 am				
11:00 am				
11:30 am				
12:00 pm				
12:30 pm				
1:00 pm				
1:30 pm				
2:00 pm				
2:30 pm				
3:00 pm				
3:30 pm				
4:00 pm				
4:30 pm				
5:00 pm				
5:30 pm				
6:00 pm				
6:30 pm				

trademark of Haynes International, Inc. This work is kindly supported by Haynes International, Inc. and National Science Foundation under contract numbers EEC-9527527 and DMI-9724476 with Ms. M. Poats and Dr. D. R. Durham as contract monitors, respectively.

4:45 PM
Detection and Evaluation of Carburized Degradation in HK-40 Cracking Tube: *Sang-Tae Kim*[1]; Chang-Gon Kim[1]; Kyong-Shik Cho[2]; [1]Yeungnam University, Dept. of Mech. Eng., Kyongsan 712-749 South Korea; [2]Korea Institute of Machinery and Materials, Changwon 641-010 South Korea

Carburized degradation in HK-40 cracking tube was evaluated by microhardness measurement and ultrasonic method. Ulrasonic method is well known as non-destructive test method and widely used to evaluate the material's damage caused by degradation practically. However, this method is just used for measuring the crack size and the thickness loss of tube. The purpose of this study is to investigate the carburized degradation in HK-40 cracking tube and the applicability of the ultrasonic technique for the evaluation of carburized material The miniaturized specimens(20¡¿20¡¿6.5 mm) are adopted from the HK-40 (25Cr-20Ni-0.4C) centrifugal cast tube after carburization treatment. Carburization was carried at 1200 ¡É by the pack method. The results of ultrasonic test present that the longitudinal wave velocity increased with the increase of carburized depth. The correlation between the longitudinal wave velocity and carburization was changed with the density and Young's modulus. With the obtained results from this study, it can be recognized that the technique using the ultrasonic velocity property is very useful method to evaluate the degree of carburized material non-destructively.

5:00 PM
Fatigue Behavior of Reactor Pressure Vessel Steels: *Jiunn-Yuan Huang*[1]; Charn-Ying Chen[1]; Jen-Hung Chen[1]; Roang-Ching Kuo[1]; Miinshiou Huang[2]; Tammy C. Smith[2]; Liang Jiang[2]; Doug Fielden[2]; Greg Jones[2]; Peter K. Liaw[2]; Jenn-Gwo Huang[3]; Sang-Tae Kim[4]; [1]Institute of Nuclear Energy Research (INER), P. O. Box 3-14, 1000, 1000 Wenhua Rd., Lungtan Taoyuan, Taiwan 325 ROC; [2]University of Tennessee, Mats. Sci. and Eng., 427-B Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [3]Taiwan Power Company, Taipei, Taiwan ROC; [4]Yeungnam University, Mech. Eng., Kyongsan South Korea

The fatigue behavior of SA 533 B-1 reactor pressure vessel (RPV) steels with sulfur content levels ranging from less than 0.01 weight percentage (wt.%) to higher than 0.03 wt.% was studied. An optical microscope, supplemented with an acoustic emission (AE) method, was employed to characterize the fatigue crack initiation and propagation behavior of RPV steels. Attempts were made to investigate the crack initiation features. Specifically, the influence of sulfur content on the fatigue behavior of RPV steels was examined in detail. MnS inclusions were characterized by quantitative optical metallography and scanning electron microscopy (SEM). Efforts were made to establish a quantitative relationship among the amount of MnS inclusions, fatigue life, and the fatigue cycle at which crack initiation occurred. Fatigue crack growth rates of RPV steels were determined and compared using different measurement techniques. The effects of MnS inclusions and load ratios on the crack propagation behavior were also investigated. Limited tests were conducted to investigate the frequency effect on fatigue crack initiation behavior. SEM was used to examine the fractographic features of fatigue-tested steel specimens. Transmission electron microscopy (TEM) was employed to investigate the roles of dislocation structures and microstructural characteristics in governing the crack initiation kinetics. The results of both SEM and TEM were used for modeling the fatigue crack initiation and propagation behavior.

Beryllium and Beryllium Alloys; Nuclear and Structural Applications: Beryllium Alloys and Physical Metallurgy

Sponsored by: Jt. ASM-MSCTS/TMS-SMD, Nuclear Materials Committee
Program Organizers: Michael F. Stevens, Los Alamos National Laboratory, MS G755, Los Alamos, NM 87545 USA; David J. Michel, Naval Research Laboratory, Code 6301, Washington, DC 20375 USA

Wednesday AM, November 3, 1999
Room: Salon M
Location: Omni Netherland Plaza Hotel

Session Chair: Michael F. Stevens, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

8:30 AM
Alloy Development in Beryllium-Based Materials: *Dan J. Thoma*[1]; Loren A. Jacobson[1]; Robert D. Field[1]; Robert J. Hanrahan[1]; Katherine C. Chen[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., Mail Stop G770, Los Alamos, NM 87545 USA

Beryllium offers unique advantages over other metals, owing primarily to its density-normalized properties and oxidation characteristics. Alloying in the metal (both intentional and unintentional) contributes significantly to the engineering response of the material. This study investigates the influence of alloying, particularly with respect to processing and properties. For example, in commercially pure beryllium, joining technologies are critically related to impurity control. Moreover, interstitial elements and inclusions have a direct impact on the deformation behavior of the material. With or without these impurities, the properties are anisotropic. Therefore, dilute substitutional alloying elements such as copper, palladium, and gold have been shown to result in more isotropic plastic behavior, while maintaining a solid-solution alloy. Finally, alloying of beryllium to intermetallics offers distinct advantages in the oxidation behavior as well mechanical properties of engineering materials. Specific examples of beryllium additions to B2 intermetallics (e.g., NiAl) and Laves phases (e.g., $NbCr_2$) will be illustrated.

9:00 AM
Solidification in Beryllium Aluminum Alloys: *Loren A. Jacobson*[1]; Paul W. Stanek[1]; [1]Los Alamos National Laboratory, Mail Stop G-770, Los Alamos, NM 87545 USA

The beryllium-aluminum system is a simple eutectic with a eutectic composition near 2.5 atom % beryllium. The liquidus on the beryllium rich side has a long portion that is nearly isothermal, and an inflection point, which implies a sub-liquidus miscibility gap. Most alloy compositions lie within the expected bounds of the miscibility gap, and so their solidification pathways can be very sensitive to a number of factors. This talk will consider a number of these factors, present some evidence for their relevance, and propose additional work that will be needed in order to clarify the fundamental issues associated with solidification and control of beryllium-aluminum alloy microstructures.

9:30 AM
Co-Deformation in Be-Al Composites: *David H. Carter*[1]; Mark A. M. Bourke[1]; Loren A. Jacobson[1]; Paul W. Stanek[1]; [1]Los Alamos National Laboratory, Mail Stop G-770, Los Alamos, NM 87545 USA

Beryllium-aluminum composites were produced that have excellent strength and ductility. These materials were hot isostatically pressed from powder which was rapidly solidified using a gas atomization process. Under rapid solidification, Be-Al undergoes

liquid phase separation, and upon complete solidification, two intimately interpenetrating phases exist. The resulting microstructure is a very fine, three-dimensional interpenetrating composite, where each phase is continuous. In order to better understand the ductility of these materials, neutron diffraction was used to measure the elastic strains in the individual phases as a function of applied stress. This data was used to quantify the deformation behavior of each phase. The Al phase is highly constrained by the Be phase due to the morphology of the composite as well as the high elastic modulus and low Poisson's ratio of Be. The results provide more insight into the overall deformation behavior of Be-Al.

9:55 AM Break

10:10 AM
The Influence of Alloying Elements on Deformed Semi-Products from AlBe Alloys: *Valery V. Savchuk*[1]; [1]Ulba Metallurgical Plant, Ust-Kamenogorsk Kazakhstan

Alloys doped with additions of magnesium, copper, zirconium, manganese and lithium were studied in this work. The best results for heat-treatable alloys were achieved with elements that were insoluble in, and did not interact with beryllium. Lithium may be the most suitable addition in this case. Investigation of the mechanical properties of extruded bars from AlBeMgLi alloys confirms this conclusion. Existing alloys respond to different types of heat treatment schedule, and the properties differ from the initial state after hardening and aging. The mechanical properties of alloys in different states are reported in this paper.

10:40 AM
Dynamic Behavior of Roll-Textured Beryllium as a Function of Temperature: *William R. Blumenthal*[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

The compressive stress-strain response of highly textured rolled-sheet beryllium was studied as a function of orientation and temperature at high strain rates up to 22% strain. X-ray diffraction measurements found a maximum in the c-axis texture of about 8 multiples of random distribution (m.r.d.) near the through-thickness direction. The texture results in a strong orientation dependence of the dynamic compression behavior over a range of temperatures between 77°K and 800°K. The dynamic compressive flow strength at room temperature in the through-thickness direction is almost a factor of two greater than in-plane directions. "Tensile-type" twins are prevalent after in-plane compression, but no evidence for twinning is observed after through-thickness compression up to 800°K. Work was performed under the auspices of the U.S. Department of Energy.

11:10 AM
Dip Brazing Aluminum-Beryllium Alloys: *W. J. Haws*[1]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA

Aluminum-beryllium alloys offer significant advantages over conventional aluminum alloys in avionics applications including lighter weight and higher stiffness. Dip brazing processes were developed for joining aluminum-beryllium alloys to fabricate avionics boxes from sheet materials. The results of dip brazing 7 different combinations of aluminum-beryllium alloys are presented along with baseline data for aluminum alloy 6061. Surface preparation, dip brazing parameters, shear test results and metallographic interpretation will be presented. Special surface preparation was required for the investment cast aluminum-beryllium alloys. This is the first significant characterization of shear strength of dip brazed aluminum-beryllium alloys. This work was funded by DARPA under Navy contract NOOO 14-96-C-0171.

Computational Materials Science at the Microstructural Scale: Microstructural Model Development and Mesoscale Mechanics Modeling

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Computational Materials Science & Engineering, Phase Transformations Committee

Program Organizers: Elizabeth Holm, Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185-0304 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark A. Miodownik, Sandia National Laboratories, Materials and Process Modeling, Albuquerque, NM 87185-1411 USA; J. P. Simmons, AFRL-MLLM, Dayton, OH 45433 USA; Prof. Peter W. Voorhees, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208

Wednesday AM — Room: Caprice 1&4
November 3, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Long-Qing Chen, Pennsylvania State University, Dept. of Mat. Sci. & Eng., University Park, PA 16802 USA; Elizabeth A. Holm, Sandia National Laboratories, Matls Theory & Computation, Albuquerque, NM 87185-0304 USA

8:30 AM Invited
Generalized Phase Field Approach for Computer Simulation of Microstructural Evolution: Incorporation of Rigid-Body Motion: Kai-Sheng Wu[1]; *Yunzhi Wang*[1]; Andrei Kazaryan[2]; Bruce R. Patton[2]; [1]The Ohio State University, Mats. Sci. and Eng., 2041 College St., Columbus, OH 43210 USA; [2]The Ohio State University, Dept. of Physics, 174 W. 18th St., Columbus, OH 43210 USA

A complete theoretical description of microstructural evolution in problems such as sintering and diffusion controlled high temperature creep in polycrystalline materials requires a treatment that incorporates multiple diffusion mechanisms as well as rigid-body motion of particles and grains. In this presentation, a new attempt to include rigid-body motion together with different diffusion mechanisms using a generalized phase field approach is introduced. A number of cases including pore shrinkage and cavity growth under external applied stress are discussed in the light of available analytical and experimental results. This work is supported by the National Science Foundation under grant DMR9703044 and the NSF Center for Industrial Sensors and Measurements under grant EEC-9523358.

9:10 AM
New Algorithms for Monte Carlo Simulation of Grain Growth and Recrystallization: *Anthony D. Rollett*[1]; Dierk Raabe[2]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]Max-Planck-Institut fuer Eisenforschung, Max-Planck-Str. 1, Duesseldorf 40237 Germany

Considerable insight into the processes of grain growth and recrystallization has been gained from computer simulation. This work has typically assumed uniform, isotropic boundary properties whereas in most materials the boundary properties depend on their crystallographic character. To account for this anisotropy the Potts model (Monte Carlo) for simulation of recrystallization has been modified to include a five parameter description of grain boundaries. The five parameter description accounts for the known dependence of properties such as energy and mobility on both misorientation and inclination. That is to say, both the difference in orientation of the crystal lattices on either side of a boundary, and the particular plane(s) comprising the boundary affect its prop-

erties. There is also a need to consider the different driving forces for grain growth and recrystallization and how best to account for them in the algorithms used for assigning transition probabilities in the Potts model. Certain aspects of these algorithms will be discussed in the context of anisotropic boundary properties. Quantitative descriptions of boundary properties are drawn from both experimental and theoretical work. Simulations of grain growth and recrystallization that illustrate the importance of boundary character are described.

9:30 AM
Scaling and Thermal Fluctuations in Potts-Based Monte Carlo Simulations: *Dierk R. Raabe*[1]; [1]Max-Planck-Institut fuer Eisenforschung, Max-Planck-Str.1, Duesseldorf 40237 Germany

The contribution consists of two parts. The first one introduces a method to scale Monte Carlo kinetics of the Potts model using rate theory. The method is particularly designed for Potts models using a non-conserved structural state variable which can assume a discrete number of degenerate ground states as commonly employed for the simulation of recrystallization and grain growth. The scaling method is based on the equivalence of single-site state switches in the Potts model and Turnbull's classical rate model of grain boundary motion mapped on a simulation lattice. The second part discusses the treatment of thermal fluctuations in the Potts model.

9:50 AM Break

10:00 AM Invited
Integral Materials Modeling: From Cellular Automata to Finite Elements: Guenter Gottstein[1]; V. Marx[1]; R. Sebald[1]; L. Lechtel[1]; R. Kopp[2]; R. Luce[2]; [1]Institut fur Metallkunde und Metallphysik, RWTH Aachen, Germany; [2]Institut fur Bildsame Formgebung, RWTH Aachen

Although recrystallization phenomena have been subject of scientific research for more than a century, prediction quality of microstructural evolution during recrystallization and grain growth is still poor. This is due to the fact that recrystallization is a non-equilibrium phenomenon which results from the thermal instability of the deformation microstructure. Instability conditions and kinetics are very sensitive to local conditions rather than to the average microstructure. Therefore, statistical models of recrystallization are of limited value. We present a mesoscopic approach of the problem by combining space resolved cellular automata codes to finite element calculations based on dislocation populations to account for work hardening. Interaction of recrystallization with precipitation is taken care of by determination of Zener drag from actually computed precipitate size distributions. Applications to hot forming process will be presented.

10:30 AM Invited
Simulating Microstructural Evolution in Deformed Polycrystals: *C. C. Battaile*[1]; T. E. Buchheit[1]; E. A. Holm[1]; G. W. Wellman[1]; M. K. Neilsen[1]; [1]Sandia National Laboratories, Albuquerque, NM 87185-1411 USA

Theories and simulations of normal grain growth can accurately predict the evolution of ideal microstructures. However, many technologically important materials are much more complex, and predicting the microstructures and properties of these systems requires more advanced models with realistic inputs. Recent computer simulations of microstructural evolution can predict complex 3-D grain structures with a natural variety of grain shapes and sizes. Polycrystal deformation simulations can provide information about local plasticity that accounts for the 3-D grain microstructures of real materials. By coupling the output from the polycrystal plasticity simulations with a modified Monte Carlo Potts model of grain growth, a meaningful picture of microstructural evolution in a deformed polycrystal is obtained. The coupling procedure will be discussed, and the dependence of coarsening behavior on the deformation history of a Cu polycrystal will be presented. This work was performed in part at Sandia National Laboratories under United States Department of Energy contract DE-AC04-94AL85000.

11:00 AM
Comparison Between Simulation and Experiment of a Polycrystal Plasticity Deformation Model: *Thomas E. Buchheit*[1]; David A. LaVan[1]; Gerald W. Wellman[2]; Micheal K. Neilsen[2]; [1]Sandia National Laboratories, Dept. 1805, MS 0333, P.O. Box 5800, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 9117, MS 0836, P.O. Box 5800, Albuquerque, NM 87185 USA

A material model which describes the rate dependent crystallographic slip of FCC metals has been implemented into JAS-3D, a quasistatic, large deformation, nonlinear finite element code. The resultant microstructural based elastic-plastic deformation model has successfully performed simulations of realistic looking 3-D polycrystalline microstructures generated using a Potts-model approach. These simulations have been as large as 50,000 elements composed of 200 randomly oriented grains. This type of model tracks grain orientation and predicts the evolution of sub-grains during deformation of a polycrystal. To determine the accuracy of the model, simulated results were compared with experimental measurements of the polycrystalline surface of an annealed OFC copper tension specimen characterized by electron backscattered Kikuchi pattern (EBKP) imaging. The copper specimens were worked then annealed prior to EBKP analysis and tension testing, producing a grain size of approximately 100 microns. Local grain orientation data before tension testing was fed directly into the polycrystal plasticity model. Performing EBKP on the same section of the sample after tension testing to 5%, 10% and 20% strain provided direct comparisons between simulated and experimental results. These direct comparisons between model and experiment isolates the issues of this quasi-continuum polycrystal plasticity modeling approach. These issues, including finite element mesh dependence on the simulated subgrain evolution, lack of a characteristic length scale and oversimplified grain boundaries, will be addressed during the presentation.

11:20 AM
Simulation of High Temperature Deformation Behavior of IN 718: Kai Y. Wang[1]; *Reza A. Mirshams*[1]; [1]Southern University and A&M College, Mech. Eng. Dept., P.O. Box 9987, Baton Rouge, LA 70813 USA

Constitutive relationships, for microstructural characteristics during hot deformation, have been developed on the basis of a dislocation density evolution model, derived from the original work by Sandstorm and Logneborg. The microstructure models, for work hardening, dynamic recovery, and dynamic recrystallization stages, are presented for IN 718 in high temperatures (around 1000°C) when the delta phase dissolved or not. Numerical solutions for the equations are made on Pietrzk's approach. Experimental validations are carried out using the published data in the literature for IN 718. The results indicate that the dislocation density evolution model for hot forging process predicts the hot flow stress behavior of IN 718 very closely with the experimental data.

11:40 AM
Thermo-Elastic Analysis of Functionally Graded Materials Using Voronoi Elements: *S. B. Biner*[1]; [1]Iowa State University, Metallu. & Ceramics, Ames Lab., 208 Metals Dev., Ames, IA 50011 USA

In this study, "Voronoi Cell Model" is developed for solving the evolution of stress-states in the heterogeneous solids. The method is based on the natural discretization of a multi-phase domain into basic structural elements by Dirichlet Tesselation. The results obtained were compared with standard finite element analyses for steady-state heat transfer analyses of bimaterials and functionally graded materials. The results obtained from the two numerical techniques compare favorably with each other. The advantages of using Voronoi Element analyses to study the micromechanistic behavior of heterogeneous solids are elucidated. This work was performed for the United States Department of Energy by Iowa

State University under contract W-7405-ENG-82. This research was supported by the Director of Energy Research, Office of Basic Sciences.

12:00 PM

OPEN

General Abstract Sessions: High Temperature Behavior I

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday AM — Room: Salon D&E
November 3, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: S. Ankem, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20742-2115 USA; Carl Boehlert, John Hopkins University, Dept. of Mech. Eng., Baltimore, MD 21218 USA

8:30 AM
Modeling of Thermophysical Properties of Pure Elements: *Qing Chen*[1]; Bo Sundman[1]; [1]Royal Institute of Technology, Div. of Computational Thermodynamics, Dept. of Mats. Sci. and Eng., Brinellvagen 23, Stockholm SE-100 44 Sweden

A thermodynamic database for pure elements contains the Gibbs energy functions of all elements in both stable and metastable structures. To obtain these functions, the temperature dependence of some thermophysical properties, for example, heat capacity, molar volume, and bulk modulus, must be adequately described. This is usually done by using simple polynomials of temperature to fit the experimental data as in available compilations or databases. The parameters fitted in this way are just isolated numbers and have no physical meaning. They are impossible to be used as a guide to predict parameters for metastable phases. In the present work, some physical models have been successfully applied to describe those thermophysical properties. It will be shown that the fitted parameters for heat capacity, molar volume and bulk modulus are indeed coherently related. Moreover, when lacking experimental data, those parameters can be calculated by using first principle theory. Examples on a couple of metallic elements will be shown.

8:50 AM
Coarsening of Grain Boundary Carbides in a Nickel-Base Ternary Alloy During Creep: *Carl Hiroshi Iwashita*[1]; Robert P. Wei[2]; [1]Lehigh University, Mats. Sci. and Eng., 226c Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA; [2]Lehigh University, Mech. Eng. and Mech., 327 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

Stress enhanced coarsening of grain boundary carbides in a Ni-18Cr-18Fe ternary alloy resulting from creep loading is analyzed. Grain boundary carbide distributions are determined from the material before and after creep testing. The extent of stress enhancement is determined by comparison of measurements from creep tested material with those from stress-free aged material. These measurements suggest that the coarsening kinetics are controlled by strongly stress-enhanced matrix and grain boundary diffusion, are analyzed with respect to existing coarsening theories. A new relationship for coarsening kinetics is presented to properly reflect the combined effects, and is correlated with the experimental data.

9:10 AM
Crystallography of Grain Boundary Carbides in a Nickel-Base Ternary Alloy During Creep: *Carl Hiroshi Iwashita*[1]; Robert P. Wei[2]; [1]Lehigh University, Mats. Sci. and Eng., 226c Sinclair Lab, 7 Asa Dr., Bethlehem, PA 18015 USA; [2]Lehigh University, Mech. Eng. and Mech., 327 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

The crystal structure of carbides and matrix near the grain boundary region of a Ni-18Cr-18Fe ternary alloy is examined. First, the identity of the grain boundary carbides is established as the face centered cubic Cr23C6 carbide. Next, the crystallographic relationships of these carbides to the matrix (<100>Matrix || <100>Cr23C6 and {100}Matrix || {100}Cr23C6) are determined on different boundary types (coherent and incoherent twins, and high angle random boundaries). Later, changes in the material caused by creep testing are examined, most notably the appearance of cavities adjacent to the carbides at incoherent carbide/matrix interfaces. The morphology of these cavities and relationship to the carbides are determined. The changes in the crystallographic relationships between carbide and matrix are discussed.

9:30 AM
Thermal Stability of Mechanical Alloyed Noncrystalline Ni-W Alloys: *Zhi He*[1]; Thomas H. Courtney[1]; [1]Michigan Technological University, Metallu. and Mats. Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

Mechanical alloying of Ni-W powder blends can generate noncrystalline structure containing nanometer scale remnant W particles. The amount of remnant W and the composition of the noncrystalline matrix depend on the alloy W content and the milling time. Using differential scanning calorimeter, x-ray diffraction and trasmission electron microscopy, we have investigated the thermal stability of such nanocrystalline alloys as they depend on W content (50 and 75at%W) and milling time (up to 40hr in a SPEX mill). Noncrystalline 50at%W alloys crystallize in three stages. During continuously heating, W first precipitates from the noncrystalline matrix. This is followed by partial crystallization (to a W supersaturated fcc phase) of the noncrystalline component. Several processes-crystallization of remaining noncrystalline component, additional precipitation of W, and intermetallic precipitation take place at higher temperature. The final crystallization products of 75at%W and 50at%W alloys are the same. However, the routes by which these form are different. The three stages observed in the decomposition of the 50at%W alloys are also found in the 75at%W alloys when the milling times are short. However, for longer milling times only one reaction, the high temperature one, is observed in the higher W content material. Possible causes for the observed

variations in the thermal stability are discussed. This work is supported by Army Research Office.

9:50 AM Break

10:00 AM
The Effect of Grain Size and Stress Level on the Ambient Temperature Creep Deformation of Titanium Ti-1.6%wt.V Alloy: *Ameet K. Aiyangar*[1]; Sreeramamurthy Ankem[1]; [1]University of Maryland, Mats. and Nuclear Eng., Room 1105, Bldg. 90, College Park, MD 20742 USA

Recently it has been reported that alpha Ti-0.4%wt.Mn alloy creeps at ambient temperature when the alloy is subjected to a stress of 95% yield stress. Further the extent of creep strain was found to increase with an increase in grain size and this was attributed to the phenomenon of Time Dependent Twinning in the coarse-grained materials. In the present investigation,the ambient temperature creep deformation behavior of another alpha Titanium alloy, Ti-1.6%wt.V, is studied in order to check whether the observations made earlier on Ti-0.4%wt.Mn alloy are also applicable to other alpha titanium alloys. The factors varied in the present investigation include grain size and stress level. The results of this investigation will be presented. This work is being supported by the Office of Naval Research under Grant No. N0001496101819

10:20 AM
A Preliminary Investigation of the Microhardness and Creep Resistance in a Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si Alloy: *Raymond J. Simpkins*[1]; Thomas R. Bieler[1]; [1]Michigan State University, Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA

The relationship between microstructure and mechanical properties has been extensively investigated in near-gamma TiAl alloys. There has been relatively little exploration of non-destructive testing methods to infer mechanical properties of this particular alloy system. In this investigation the relationship between room temperature microhardness of equiaxed and lamellar grains in Ti-47Al-2Nb-1Mn-0.5W-0.5Mo-0.2Si alloy and its primary creep resistance is examined to determine how the hardness and primary creep resistance are related. Samples of the as-HIPed specimen were exposed to single stage heat treatments, ranging from 900°C to 1177°C for 4, 5, and 10 hours, and a two-stage heat treatments at 1177°C/1010°C for 5 and 10 hours respectively to obtain varying microstructures. Energy Dispersive Spectroscopy (EDS) was utilized to examine fluctuation in composition of the equiaxed grains. Using polarized light techniques the equiaxed grains were characterized according to their respective brightness contrast and hardness under the microscope. Microhardness results indicated an average increase in hardness of both lamellar and equiaxed grains with each single stage heat treatment up to 1177°C. The average grain size decreased as the overall hardness of the equiaxed grains increased with increasing amounts of heat treatment. Primary creep experiments have revealed an increase in primary creep resistance with increasing heat treatment time and temperature.

10:40 AM
Failure Mode Classification of PSZ Thermal Barrier Coatings under Laser Thermal Shock Conditions: *X. Q. Ma*[1]; M. Takemoto[1]; K. Ono[2]; [1]Gakuin University, Faculty of Sci. Eng., Tokyo 157 Japan; [2]UCLA, Dept. of Mat. Sci. and Eng., Los Angeles, CA USA

The present work aims to investigation of the failure behaviors and mechanisms of plasma sprayed ZrO_2-8%Y_2O_3 thermal barrier coatings (TBCs) under different laser thermal shock conditions. With varying power from 30~100W, a CW CO_2 laser was used as high heat-flux source. The fractured-modes and dynamics of the TBCs with a NiCrAlY bond coat or not were elucidated by using an advanced acoustic emission technique. In the AE system, the AE signals from an out of plane displacement-type sensor were monitored, and employed for source simulation (i.e. fracture-mode, fractured volume and rise-time) of microcracking induced during the thermal shock test. The temperature profiles on the surface and at the interface of the TBCs were used for FEM simulation of thermal stress. The Fracture mode I was identified to be predominant for the TBC's failure (cracking, delimanation and spallation) while a great temperature gradient exists in the zirconia coat under the laser heat conditions. Key words: Coating, TBC, laser heating, failure, fractured mode, AE.

General Abstract Sessions: Advances in Processing and Heat Treatment II

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday AM
November 3, 1999
Room: Salon F&G
Location: Omni Netherland Plaza Hotel

Session Chair: Charles Henager, Jr., Structural Materials Development, Staff Scientist, Richland, WA 99352 USA

8:30 AM
An Examination of the Bainite Reaction in Fe-C-Mo: *R. E. Hackenberg*[1]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Mats. Sci., Thornton Hall, Charlottesville, VA 22903 USA

A steel of composition Fe-.24C-4.00Mo (wt%) has been investigated with the goal of determining the influence of molybdenum on the kinetics and morphology of the bainite reaction. Conventional TEM observations of the growth front of the ferrite + carbide aggregate, coupled with analytical TEM to determine the molybdenum distribution in this region were used toward this end. Particular attention has been paid to time-temperature envelope in the "bay" region on the T-T-T diagram, where an apparent growth stasis had been shown to occur, and where the postulated solute drag-like effect (SDLE) is expected to operate at maximum strength. The results will highlight the critical roles of interfacial structure and carbide precipitation in determining the migration behavior of the bainite-austenite growth front. Support by NSF-DMR is acknowledged.

8:50 AM
Effect of Mn on Pearlite Growth within the (g + a + Fe_3C) Three Phase Field of the Fe-C-Mn Phase Diagram: *C. R. Hutchinson*[1]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Mats. Sci., Thornton Hall, McCormick Rd., Charlottesville, VA 22903 USA

It is a well known effect that substitutional alloying elements have a very strong effect on the formation of pearlite. An attempt at understanding this effect must begin with an examination of the role of the alloying elements in altering the Fe-C phase diagram. Additions of Mn to the Fe-C system lowers the upper Ae1 and opens up an (g + a + Fe_3C) three phase field. The transformation to pearlite within this three phase field (which is necessarily less than 100% pearlite at equilibrium) exhibits some very interesting characteristics, namely a non-constant growth rate and a continually changing g/pearlite interfacial Mn concentration with time. Examination of the growth of pearlite within the three phase field is the topic of this investigation. Analytical transmission electron microscopy (ATEM) has been used to measure the g/a and g/Fe_3C interfacial Mn concentrations with time and an explanation for the unusual solute profiles and growth kinetics observed is presented in the framework of the local equilibrium model. Support by the NSF-DMR is acknowledged.

9:10 AM

Precipitation of the Al3(Sc,Zr) Phase in a Superplastic Aluminum Alloy: *John S. Vetrano*[1]; C. H. Henager[1]; S. M. Bruemmer[1]; [1]PNNL, MSIN P8-16, P.O. Box 999, Richland, WA 99352 USA

Scandium additions to aluminum-magnesium alloys precipitate as fine, coherent Al3Sc particles, which strengthen the material and inhibit recrystallization. Studies have shown that Zr additions to the ternary alloy system retard the overaging of the coherent particles. Fine-probe compositional analysis of the precipitates in the in the Al-Mg-Sc-Zr alloy revealed that Zr is present substitutionally for Sc in these fine precipitates. Additionally, Zr is present at higher concentrations at the periphery of the particles. This configuration hinders the overaging mechanisms and results in a more stable precipitate. Implications of this process on superplasticity and recrystallization will be presented. Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

9:30 AM

Variations in Chemical Composition of Precipitates in Al-Ag Alloys: *K. Matsuda*[1]; G. J. Shiflet[2]; S. Ikeno[1]; [1]Toyama University, Dept. of Sys. Eng. for Life Sci. and Eng., Toyama 930-8555 Japan; [2]University of Virginia, Mats. Sci. and Eng., Thornton Hall, Charlottesville, VA 22903 USA

There has been considerable work done concerning the development and role of interfacial structure during growth and dissolution of the Gamma' and the Gamma phases in Al-Ag alloys. The phase transformation involves Shockley partial dislocations and therefore represents one of the simplest solid-state diffusional transformations. However, there are fewer studies concerning chemical and structural changes associated with the transformation of Gamma' to Gamma. The Gamma' and Gamma phases in Al-Ag alloys have a similar crystal structure(HCP) and have somewhat different lattice parameters: viz., a=0.2858nm and c=0.4607nm, and a=0.2879nm and c=0.4573nm, respectively. Howe, Dahmen and Gronsky, found that the Gamma' phase has A-B-A-B stacking on an HCP lattice and that A-planes have pure silver and B-planes have aluminum and silver with an overall ratio of Al2Ag. The phase diagram shows that the equilibrium Gamma phase is not a line composition and that it has a range of silver concentration, so it can form from Ag3Al to Ag2Al. In this work, the chemical composition and information on crystal structure were obtained from precipitates aged at several temperatures in Al-16 and 30 mass%Ag alloys using a FE-TEM equipped with EDS. The silver composition in the precipitates increased from the ratio of Ag to Al (Ag/Al) = 1.5 to Ag/Al = 2 with increasing aging time and temperature, although crystal structure changes were not detected using convergent beam diffraction patterns.

9:50 AM Break

10:00 AM

An Age Hardening Study of Carbide Precipitation in a Ti-47Al-2Nb-2Mn—0.8vol%TiB_2 + 0.1 wt%C and its Effects on Mechanical Properties: *Raymond J. Simpkins*[1]; Thomas R. Bieler[1]; Paul A. McQuay[2]; [1]Michigan State University, Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA; [2]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49445 USA

The addition of carbon in both duplex and fully lamellar g-TiAl has yielded promising results on mechanical properties without significant decreases in ductility. In an attempt to determine the maximum strengthening effects of carbide precipitates in a Ti-47Al-2Nb-2Mn-0.8vol%TiB_2 +0.1wt%C alloy, solutionizing heat treatments ranging from 1000-1260°C to (1830-2300°F) for times up to 8 hours were conducted. Subsequent age hardening heat treatments ranging from 900-1200°F (1650-2200°F) were carried out to precipitate the carbon out of solution. Preliminary microscopy of the as-HIPed samples using Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) have revealed the formation of carbides in gamma grains, at gamma grain boundaries, around dislocations, and at a2/g interfaces. The precipitates in the alloy were characterized by shape, size, distribution, and orientation with its host matrix. Primary creep tests at 1500°F and 20ksi were conducted on the aged specimens to evaluate the effect of the heat treatment process in order to determine optimal strengthening effects of the carbide precipitates.

10:20 AM

Mixed Convection-Solidification in a Single-Component Liquid: *Noureddine Hannoun*[1]; Layachi Hadji[1]; [1]University of Alabama, Dept. of Math., P.O. Box 870350, 345 Gordon Palmer Hall, Tuscaloosa, AL 35487-0350 USA

We consider a horizontal layer of single-component liquid which is heated from below and solidified from above. A solid-liquid interface separates the liquid phase from the solid phase. As the heating intensity increases, which corresponds to an increase in the Rayleigh number Ra, steady cellular convection sets in the liquid when Ra exceeds a critical value. The convective motion is characterized by zones of up-flow of warm fluid separated by zones of down-flow of cold fluid which result in partial melting of the solid and the formation of crests and troughs at the solid-liquid interface. Previous experimental studies by Muller and Dietsche [Journal of Fluid Mechanics, vol. 161 (1985)] have shown that various roll-like patterns may occur depending on the value Ra and on the dimensionless thickness of the solid layer in the pre-convective regime. Specifically, bimodal patterns have been observed for moderately large Ra. In this study, we investigate numerically the configuration above and contrast our results to the experimental results obtained by the cited authors. The problem is governed by the Navier-Stokes equations and an energy equation valid for both the liquid and the solid phases. The solid-liquid interface is assumed deformable and its movement is on a much slower scale than the convection time scale. A two-dimensional version of the equations and boundary conditions are solved by the SIMPLER algorithm which incorporates an enthalpy formulation to simulate the phase-change. Emphasis is on isolating the two-dimensional patterns in the parameter space of the problem.

10:40 AM

Alloy Classification Based in DAS-Cooling Rate Experimental Equation: *María Eugenia Noguez*[1]; Guillermo F. Salas[1]; José G. Ramírez[1]; Teresita Robert[1]; [1]Universidad Nacional Autónoma de México, Ing. Metalúrgica, Facultad de Química, Congreso 88, Casa 4, Tlalpan, México, D.F. 14000 Mexico

Since the experimental equations relating secondary dendrite arm spacing and local solidification time or cooling rate, were developed. Later, theoretical models arrived to general similar equations. The reported equations (theoretical and experimental) have been considered simplified as l2= B (CR)-c,where l2 is Secondary Dendrite Arm Spacing (DAS), B and c are constants and CR is the Cooling Rate. B and c numbers have been collected from different authors and classified. The resulting equations were plotted, showing experimental and extrapolated cooling rate ranges. Discussion was focused to try to group Steel and Al, Cu, Ni, Fe and Ti alloys data according to these constants and is presented regarding the similarities and differences among the experimental and theoretical values within and among them.

11:00 AM

A Cellular Automaton Approach to Modeling of Structural Development in Electroslag Casting: Xiuqin Wei[1]; *Lang Zhou*[1]; [1]Nanchang University, Dept. of Mech. Eng., Nanchang 330029 ROC

A 3-D cellular automaton model of thermal transfer and solidification has been developed, aiming at a simulational study of the grain structure development in electroslag casting. The program we developed for simulation of the model allows the effects of both metallurgical factors, including solidification point, supercooling for nucleation and its scattering, and liquid/solid interface energy, and thermophysical factors, including heat conduction coefficients, heat transfer coefficients and latent heat, to be investigated. The effect of process control can be indirectly inspected with the simu-

lation by varying the melting rate. A box counting algorithm was employed to estimate the local curvature of liquid/solid interface. A series of simulated experiments of electroslag casting processes have been carried out. The simulation started from the beginning of the electroslag casting and proceeds by iteration of certain rules, during which a uniform constant slag temperature and a constant melting rate were assumed. It has been observed that a pool of molten metal forms and deepens gradually under constant melting rate. The deepening of the pool slows down with the simulated electroslag casting process, and the depth and shape of the pool tends to be steady after certain height of cast is formed. A columnar grain structure with the columns normal to the bottom of the molten metal pool was generally observed. Higher latent heat was found to enhance dendritic growth, while higher liquid/solid interface energy tend to inhibit dendritic development, although the effect of the interface energy was shown to be very weak. The results agree well with general observation of the grain structures in electroslag castings and demonstrate the applicability of cellular automaton modeling to structural development in casting.

General Abstract Sessions: Microstructure and Mechanical Properties I

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday AM
November 3, 1999

Room: Salon B&C
Location: Omni Netherland Plaza Hotel

Session Chairs: John Lewandowski, Case Western Reserve University, Dept. Mats. Sci. & Eng., Cleveland, OH 44106 USA; David A. Alven, Aerojet Ordance, TN, USA

8:30 AM
Fracture Resistance of a Zr-Ti-Ni-Cu-Be Bulk Amorphous Alloy: *Peravudh Lowhaphandu*[1]; Lorie A. Ludrosky[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Dept. Mats. Sci. and Eng., 10900 Euclid Ave., Cleveland, OH 44106 USA

The effects of changes in notch root radius (n.r.r.), fatigue precracking, and annealing treatments on the fracture toughness of a Zr-Ti-Ni-Cu-Be bulk amorphous alloy were investigated. The fracture toughness was determined via single-edge notched bend specimens under three point bend loading. It was found that the notch root radius, ranging from a fatigue precrack up to a 250 μm radius machined notch, had a strong effect on the magnitude of the fracture toughness. The effects of annealing treatments at temperatures below the supercooled liquid region for various time periods on the strength and notched fracture toughness were also determined. Increases in annealing time/temperature produced a severe reduction in notched toughness. The fracture surface features were characterized and related to the changes in fracture toughness recorded. The project is supported by AFOSR-AASERT-F49620-96-1-0228.

8:50 AM
Effects of Stress Triaxiality on Flow and Fracture of a Zr-Ti-Ni-Cu-Be Bulk Amorphous Alloy: *Peravudh Lowhaphandu*[1]; Snowy L. Montgomery[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Dept. Mats. Sci. and Eng., 10900 Euclid Ave., Cleveland, OH 44106 USA
Abstract Text Not Available

9:10 AM
Mechanical Anisotropy in Heavy Gauge Hot Band of AA7050 Aluminum Alloy: *Yansheng Liu*[1]; James G. Morris[1]; [1]University of Kentucky, Light Metals Rsrch. Lab., Dept. of Chem. and Mats. Eng., Lexington, KY 40506 USA

AA7050 aluminum alloy has high strength and is widely used in the aircraft industry. The mechanical anisotropy of this alloy is important to its application. Tensile strength difference has been observed in the rolling versus the transverse direction. Variation of tensile property is also observed in the normal direction to the rolling plane. Texture and microstructure were observed in order to understand the variation of mechanical properties of the alloy. Results of this analysis indicate that texture variation is responsible for the mechanical property change in the normal direction to the rolling plane.

9:30 AM
Effects of Changes in Grain Size on Flow and Fracture of Nb and Nb-1%Zr: *Deenesh Padhi*[1]; Anand V. Samant[2]; John J. Lewandowski[1]; [1]Case Western Reserve University, Mats. Sci. and Eng., Charles White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA; [2]Praxair Inc., Process and Systems R&D, 175 East Park Dr., P.O. Box 44, Tonawanda, NY 14151-0044 USA

The mechanical behavior of polycrystalline pure Nb and a Nb-Zr alloy was investigated under a variety of stress states, strain rates, and test temperatures. In addition to testing both smooth and notched specimens, instrumented impact tests were performed on as-received commercially pure Nb, on a Nb-Zr alloy and on vacuum heat treated Nb and the Nb-Zr alloy. The effects of changes in grain size on the flow and fracture of these materials under the conditions tested is reported.

9:50 AM Break

10:00 AM
Preliminary Experiments on an Advanced Deformation Simulator Apparatus: *Nishad S. Prabhu*[1]; [1]Case Western Reserve University, Mats. Sci. and Eng., Charles White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA

Compression tests have been conducted on a variety of metallic and polymeric materials in order to determine the effects of changes in deformation velocity on the flow stress of such materials. Recent equipment designed by MTS Systems Inc. provides the capability of simulating a variety of deformation processing sequences, including multiple deformation sequences at very high velocity (e.g.^{3}100 in/sec). A description of the equipment capabilities which includes high velocity and high load capacity (e.g. 110 kip) with the possibility of multi-hit sequences via the use of a separate 220 kip indexing actuator will be provided. The presentation will conclude with a summary of initial experiments conducted on a number of materials over a range of loading rates (e.g. 0.1 in/sec-100 in/sec) where either a multi-hit sequence (e.g. up to 5) or a single hit has been conducted to provide a total compressive strain of up to 0.7. Initial observations on material behavior and deformation induced microstructural changes will be provided. Experimental work is being partially supported by a MTS fellowship, with equipment support by NSF-DMI-9512296, Ohio Board of Regents, and The Case School of Engineering.

10:20 AM
Mechanical Properties of Reactively Sputtered (Ta, Si)N Hard Coating: J. W. Nah[1]; W. S. Choi[1]; *Sun-Keun Hwang*[1]; J. M. Lee[1]; [1]Inha University, Div. of Mats. Sci. and Eng., 253 Yong-Hyun Dong, Nam-Gu, Incheon 402-751 Korea

The mechanical properties and the chemical bonding characteristics of a ternary hard coating based on (Ta, Si)N deposited onto a high-speed tool steel by reactive sputtering were studied. From an Auger electron spectroscopy, it was found that the nitrogen content in the coating increased with the nitrogen gas flux during deposition. In as-deposited condition, the major phase of the coating was TaN containing Si in solution. The micro-hardness of the coat-

ing increased with the flux of the nitrogen gas during sputtering, reaching to 17GPa. Post-coating annealing heat treatment, particularly the one at 1000¡É, resulted in a change in the chemical composition of the coating, mainly because of a reverse diffusion of Fe and C from the substrate, which yielded a TaC phase. The hardness as well as the adhesion strength of the coating were increased by the annealing treatment at 1000¡É. The XPS analysis of the coating layer found that Fe and Si in the coating did not form chemical compound but enriched the interface of the coating and the substrate.

10:40 AM
Study of Prismatic and Basal Slip in Oriented Single Colony Crystals of an Alpha/Beta Titanium Alloy: *Satyarth Suri*[1]; Gopal Viswanathan[1]; T. Neeraj[1]; M. F. Savage[1]; M. J. Mills[1]; [1]The Ohio State University, Mats. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Titanium alloys are used in a number of aerospace, biomedical and naval applications due to their high specific strength, Youngs modulus and corrosion resistance. There have been reports in the literature that these alloys creep at room temperature at stresses below the yield strength. Deformation in polycrystalline Ti alloys typically occurs on the <a>-type prismatic slip system. Recently there have also been reports of a decrease in the dwell fatigue life of these alloys as compared to the low cycle fatigue life. The decrease in the dwell fatigue life of these alloys has been associated with <a>-type basal slip and preferential cleavage along the basal planes. The current work investigates both the prismatic and basal slip under constant strain rate and creep conditions in oriented single colony crystals of a near alpha-Ti alloy at room temperature. Details of the mechanisms of slip transmission across the alpha/beta interfaces for both the prismatic and basal slip orientations have been investigated. The mechanical behavior of the single colony crystals will be interpreted in terms of the details of the slip transmission process.

11:00 AM
Effect of Thermomechanical Processing on the Mechanical Anisotropy and Crystallographic Texture of DC Cast AA3003 Aluminum Sheet: *G. J. Liao*[1]; Y. Liu[1]; Y. L. Liu[1]; J. G. Morris[1]; [1]University of Kentucky, Light Metals Rsch. Lab., 175 Anderson Hall, Lexington, KY 40506 USA

The mechanical anisotropy of DC cast AA3003 hot hand was determined by measuring the earing behavior for different thermomechanical processes. The corresponding crystallographic texture was determined by pole figure measurement and analyzed by orientation distribution function (ODF) method. The texture characterization is correlated with the variation in earing percentage. The as-received hot band had a 45o earing and high deformation texture components, while annealing the hot band produced 90o earing. The annealed hot band had a recrystallized texture with a high volume fraction of the cube component {001}<100>. When the annealed hot band was cold rolled to different strains, its 90o earing percentage decreased with increase in the degree of cold rolling reduction and the earing changed from 90o to 45o at a cold rolling reduction of 45%. Accordingly, the softening texture components decreased and the deformation texture components increased with an increase of strain. However, the as-received material annealed after cold rolling had a critical change in mechanical anisotropy at a plastic strain of 70% which was higher than the annealed materials subjected to cold rolling. The former material showed a lower volume fraction of the cube component than the annealed hot band. On the other hand, the annealed hot band after cold rolling plus annealing exhibited 45o earing instead of 90o as would normally be expected, and this material showed a high randomly recrystallized texture with little accumulation of any particular orientation and a low cube component and a high R-cube component. The earing behavior can be indicated by the D value (D = S (Copper+Brass+S+R-Cube)-S(Goss+Cube)). Positive D values are associated with 45o earing and negative D values are associated with 90o earing.

Hydrogen Effects on Materials Behavior: Fracture in Hydrided and Amorphous Materials

Sponsored by: Structural Materials Division; ASM International: Materials Science, Critical Technology Section; Corrosion and Environmental Effects Committee
Program Organizers: Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Russell H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; A. W. Thompson, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

Wednesday AM
November 3, 1999
Room: Salon H&I
Location: Omni Netherland Plaza Hotel

Session Chairs: Petros Sofronis, University of Illinois, Theor. and Appl. Mech., Urbana, IL 61801 USA; Michael J. Morgan, Westinghouse Savannah River Company, Aiken, SC 29808-0001 USA

8:30 AM
Hydride Formation in Commercial Purity Titanium: Zhengfu Wang[1]; *Clyde L. Briant*[1]; K. Sharvan Kumar[1]; [1]Brown University, Dept. of Eng., P.O. Box D, Providence, RI 02806 USA

This paper will report a study on the critical potential for hydride formation in aqueous NaCl solutions. The results will show that the critical potential for hydride formation decreases significantly with increasing pH and also decreases with decreasing temperature. For example at 70C the critical potenital is-600mV SCE in a 3.5%NaCl solution at pH=1, whereas it is-1000mV SCE at pH=7. The effects of surface preparation and pre-straining will be discussed and also the time required to detect hydrides at a given potential. Comparison will be made between the critical potential measured in commercial purity titanium and in titanium alloys. Finally, the correlation between the critical potential and the degradation of mechanical properties will be considered. In this context it will be pointed out that the distribution of hydrides is of paramount importance. This work was supported by the Office of Naval Research.

8:50 AM
Microstructural Effects on Hydriding Kinetics in Alpha-Uranium: *Robert J. Hanrahan*[1]; John F. Bingert[1]; Dan J. Thoma[1]; Marilyn E. Hawley[1]; Geoffrey W. Brown[1]; [1]Los Alamos National Laboratory, TA 3, MS G770, Los Alamos, NM 87545 USA

The kinetics of the uranium-hydrogen reaction have been the subject of numerous studies. However, the mechanisms governing the initial stages of surface hydride nucleation and growth are controversial. In this work, we have used a combination of experimental techniques (such as optical microscopy with an environmental stage, orientation imaging microscopy (OIM) and atomic force microscopy (AFM)) to demonstrate that the nucleation of UH3 occurs within the near surface region of the metal. In addition, OIM has revealed that particular grains that show a greater susceptibility to hydride nucleation have lower density orientations perpendicular to the surface, and therefore most likely permits faster hydrogen diffusion. The hydride nucleation sites within the grains appear to be consistent with local chemical inhomogeneities acting as catalysts for the formation of UH3. The subsequent expansion of the hydride requires deformation and fracture of the metal, which explains the inverse correlation between strength and hydride growth rate observed in a-uranium.

9:10 AM
Crystallography and Phase Transformations in Ti-Al Based Metal Hydrides: *Bryan Molloseau*[1]; Marc De Graef[1]; [1]Carnegie Mellon University, Mats. Sci. and Eng., Roberts Engineering Hall, Rm. 136, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Transition metal intermetallics have potential applications in hydrogen-based propulsion systems for aircraft and future hypersonic vehicles. To obtain an in-depth understanding of the effect of hydrogen on the stability and properties of transition metals and their hydrides we have embarked upon a combined modeling and experimental study of a tetragonal hydride (q-phase) in the Ti-Al system. This hydride phase is obtained after high pressure, high temperature charging, and replaces all a_2 phase in a duplex microstructure. The unit cell of the hydride is body-centered tetragonal and we will present results from x-ray diffraction, neutron diffraction, electron diffraction and high resolution transmission electron microscopy detailing the transformation from a_2 to the heavily faulted q-phase.

9:30 AM
Thermohydrogen Processing of Titanium Alloys: *F. H.(Sam) Froes*[1]; Oleg N. Senkov[1]; Mutlu Cavusoglu[1]; Javaid I. Qazi[1]; William M. Mullins[2]; [1]University of Idaho, Instit. for Mats. and Adv. Processes, 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]US Army Research Office, P.O. Box 12211, Research Triangle Park, NC 27709-2211 USA

Use of hydrogen as a temporary alloying element in titanium alloys is an attractive method for enhancing processability including working, machining, sintering, compaction, etc., and also for controlling microstructure and thereby improving final mechanical properties. In present paper, the status of the methods and applications of the thermohydrogen processing of titanium alloys is reviewed. Effect of hydrogen alloying on the phases present, their composition, and kinetics of phase reactions are considered. The effect of hydrogen on hot and cold workability, composite-and powder-metallurgy-product processing, and microstructure modification of wrought and cast conventional alloys and intermetallics, including production of nanocrystalline structures is discussed. Recent results on the processing of nanocrystalline alloys and composites, as well as on studies of the workability of various titanium alloys at elevated temperatures will also be presented. Thermohydrogen processing has clear advantages in the development of improved microstructures and mechanical properties in titanium alloys. In the case of near net shapes it is the only method for significant microstructural modification. It allows energy saving in processing final products by improving the workability.

9:50 AM
Deformation and Fracture of Hydrided Zircaloy-4: D. W. Bates[1]; *R. S. Daum*[2]; D. A. Koss[2]; A. T. Motta[2]; [1]Currently U.S. Navy; [2]Pennsylvania State University, University Park, PA 16802 USA

During reactor exposure, Zircaloy-4 cladding tubes in pressurized water reactors undergo corrosion and hydriding, potentially affecting cladding mechanical properties. This study is investigating the effect of hydrides on the failure of Zircaloy-4 under conditions relevant to reactor-exposure hydriding, specifically the tendency to form hydrides locally near the outer (cool) surface of the Zircaloy cladding. We have tested unirradiated cladding tube material using ring specimens subjected to both plane-strain and uniaxial tension at room temperature and 300 degrees centigrade. Three material conditions have been examined: unhydrided, uniformly hydrided, and hydrided such that the hydrides are concentrated within a layer near the outer surface of the cladding tube. While a uniform distribution of hydrides does not affect ductility over a wide range of test conditions, specimens with hydride layers greater than 100um (> 740 wt ppm H) are embrittled. Conversely, specimens with hydride layers less than 100um (400-600 ppm H) exhibit ductility, suggesting a transition from ductile to brittle failure at a critical hydride layer thickness.

10:10 AM Break

10:20 AM
Hydrogen-Induced Effects on Barrier Materials for the Geological Repository: *Peter J. Bedrossian*[1]; Douglas L. Phinney[1]; Joseph C. Farmer[1]; [1]Lawrence Livermore Laboratory, Mats. Sci. & Tech., P.O. Box 808, L-350, Livermore, CA 94551 USA

Several of the alternative designs for the Engineered Barrier Systems at the proposed High-Level Waste repository at Yucca Mountain, Nevada include either Titanium Grade 7 or Grade 16. While exhibiting excellent corrosion resistance, these materials are prone to hydriding and may therefore be susceptible to hydrogen-induced cracking, swelling, blitering, and related phenomena. It is therefore crucial to determine the extent to which cathodic hydrogen charging, embrittlement, and volume expension may affect the performance of the proposed waste package designs. Using a combination of in situ atomic force microscopy (AFM) and Secondary Ion Mass Spectrometry (SIMS), we have observed blister formation during cathodic charging of titanium in aqueous solutions, leading to cracking of the surface after extended periods of cathodic charging. Decreasing the pH of the electrolyte accelerates the blister formation. The presence and the extent of blistering and swelling phenomena have been correlated with quantitative measurements of hydrogen uptake. This work was performed at Lawrence Livermore National Laboratory under the auspices of the US DOE under contract W-7405-Eng-48, and was partially supported by the Yucca Mountain Project.

10:40 AM
The Crack Propagation Behavior of Zircaloy-2 in High Pressure H_2+H_2O: *R. Grant Rowe*[1]; [1]GE Corporate Research and Development, P.O. Box 8, K-1, MB265, Schenectady, NY 12301 USA

It has been shown that in a high pressure H_2 gas environment at 325°C, Zircaloy-2 fractures in a brittle manner at crack propagation rates as high as 5 mm/s. The fracture mechanism, termed HGC, has been determined to be the rapid formation of a d-$ZrH_{1.5}$ hydride layer at the crack tip followed by brittle fracture and redevelopment of the layer by deformation enhanced H transport at the crack tip. A study of the effect of strain rate on HGC at 325°C showed that that at engineering strain rates below 10^{-3} s^{-1}, residual H_2O in the atmosphere (H_2O/H_2 @ 10^{-5}) prevented the initiation of HGC fracture while at higher surface strain rates, fracture occurred by brittle HGC. The present work examines rate of crack propagation in H_2+H_2O environments with various H_2O partial pressures. The interdependency of the environment and crack propagation rate on the mechanism of fracture will be discussed.

11:00 AM
Effect of Hydrogen Alloying on Mechanical Properties of Titanium: *Oleg N. Senkov*[1]; F. H.(Sam) Froes[1]; John J. Jonas[2]; [1]University of Idaho, Instit. for Mats. and Adv. Processes, 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]McGill University, Dept. of Metallu. Eng., 3610 University St., Montreal H3A 2B2 Canada

Effect of hydrogen alloying (up to 30 at.%) on elastic and plastic properties of titanium over the temperature range 20°C to 1000°C was studied. Elastic moduli slightly increased with an increase in the volume fraction of a hydride phase at room temperature. Solid solution of hydrogen in the alpha titanium decreased the shear and Young's moduli, increased the bulk modulus, Lame constant and Poisson's ratio, and decreased the yield stress, flow stresses and strain hardening. By contrast, alloying the beta titanium with hydrogen increased the shear and Young's moduli, decreased Poisson's ratio, did not appreciably affect the bulk modulus and Lame constant, and increased the yield stress, flow stress and strain hardening. The different effects of hydrogen on the elastic constants and plastic behavior of alpha and beta titanium are interpreted in terms of the influence of dissolved hydrogen on the stability of the hexagonal close-packed and body-centered cubic lattices in the vicin-

ity of the alpha-to-beta transformation.

11:20 AM
Effects of Hydrogen on the Fracture Behavior of Zr-Ti-Cu-Ni-Be Bulk Metallic Glass Alloys: *Daewoong Suh*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Dept. of Mats. Sci. and Eng., Stanford, CA 94305-2205 USA

Recently developed multicomponent Zr-Ti-Cu-Ni-Be bulk metallic glass alloys exhibit a range of unique properties including ultra-high strength, large elastic strains, and excellent corrosion and wear resistance. However, studies conducted on rapidly solidified amorphous metals suggest that these materials may be susceptible to hydrogen embrittlement. At present, there is little understanding of hydrogen effects on the fracture behavior and mechanisms by which hydrogen interacts with the amorphous structure. In the present study, the effects of hydrogen on the mechanical and fracture behavior of Zr41.25Ti13.75Cu12.5Ni10Be22.5 (at.%) bulk metallic glass alloys have been investigated. Fracture and fatigue crack growth behavior were examined after hydrogen charging specimens using a cathodic charging technique. It was shown that whereas the fracture toughness was degraded, fatigue crack growth was significantly retarded after charging. Such conflicting results are interpreted in terms of a mutual competition between degradation of the inherent resistance to crack extension and a reduced crack driving force by crack tip shielding mechanisms. They include crack deflection, roughness-induced crack closure and crack bridging. These processes are believed to counteract the embrittling effects of hydrogen. Fractographic studies revealed a marked difference after hydrogen charging indicating a fundamental difference in fracture and deformation processes. Microstructural characterization including XRD and TEM was performed with the aim of identifying changes due to hydrogen. Possible mechanisms are discussed with particular emphasis on the mechanical and chemical effects of hydrogen, and the separate role of intrinsic damage and extrinsic shielding mechanisms.

11:40 AM
The Effects of Hydride Cycling on the Mechanical Properties and Hydriding Thermodynamics of Pd-Based Alloys: *David F. Teter*[1]; Daniel J. Thoma[1]; [1]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA

Pd-based alloys have the unique characteristic that they retain structural integrity after cycling through the hydride phase several times as compared to most metals and intermetallics, which crack and form powders upon hydriding. Pd-based alloys become heavily deformed during hydriding, and consequently the hardness, i.e. yield strength, of the metal increases dramatically above that for the annealed condition. Also, cycling of the alloy through the hydride phase field alters the thermodynamics of hydride formation and decomposition. Cycled alloys tend to have flatter plateau pressures in pressure-composition-temperature isotherms than annealed alloys. The plateau pressure of cycled alloys can be lower, equal or higher than that of the annealed form depending upon alloy composition and temperature of hydriding. Changes in the thermodynamics and pressure-composition isotherms due to hydride cycling are explained in terms of the mechanical properties of the annealed and cycled forms of the alloy as well as the mechanism of hydride growth through these alloys.

12:00 PM
Influence of Delta-Phase, Degree of Homogenization and Cast Thickness on the Hydrogen Embrittlement of Cast Alloy 718: *Goeran Sjoeberg*[1]; Daniel Cornu[2]; Thomas Kruslind[1]; Jonas Ciardi[3]; David Pallois[2]; [1]Volvo Aero Corporation, Space Propulsion Div., Mail Stop 6670GS, SE-461, Trollhattan Sweden; [2]Societe Europeene De Propulsion, Materiaux Procedes, Foret de Vernon, Vernon, Cedex 27208 France; [3]Royal Institute of Technology, Dept. of Mats. Processing, S-100 44, Stockholm, Sweden

Embrittlement of cast alloy 718 was examined by conventional tensile testing and by sub-scale, in-situ, tensile testing in SEM of smooth specimens charged with hydrogen. The effects of delta-phase, level of homogenization and of cast thickness were studied. At 30 ppm, the hydrogen embrittlement, measured as the reduction of elongation at fracture, was of the same order of magnitude (63-71%) for both thicknesses and both levels of homogenization examined. For the delta-phase containing material (by deliberate precipitation) the elongation was reduced by 83%. Fractography on broken specimens and on polished specimens during the straining in the SEM supported conclusions from the mechanical data obtained. Premature fissures due to the presence of hydrogen were observed in grain boundaries, eutectic areas and especially at the delta-phase platelet/matrix interfaces.

Processing and Properties of Structural Nanomaterials III

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Powder Materials Committee, Alloy Phases Committee, Materials Processing & Manufacturing Division, ASM-MSCTS, Materials & Processing Committee

Program Organizers: Leon L. Shaw, University of Connecticut, Department of Metallurgy & Materials Engineering, Storrs, CT 06269-3136 USA; Lawrence T. Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; Carl C. Koch, North Carolina State University, Department of Materials Science & Engineering, Raleigh, NC 27695 USA

Wednesday AM — Room: Rookwood
November 3, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Carl C. Koch, North Carolina State University, Mats. Sci. and Eng., Raleigh, NC 27695 USA; Enrique J. Lavernia, University of California, Dept. of Chem. and Biochem. Eng. and Mats. Sci., Irvine, CA USA

8:30 AM Invited
Synthesis and Consolidation of Nanocrystalline Materials-An Overview: E. G. Baburaj[1]; *F. H. (Sam) Froes*[1]; O. N. Senkov[1]; [1]University of Idaho, Instit. for Mats. and Adv. Processes (IMAP), 321 Mines Bldg., Moscow, ID 83844-3026 USA

This paper reviews the research work, at IMAP, on the synthesis of nanocrystalline materials from precursor elements and compounds, and their consolidation. Nanocrystalline materials has been synthesized by a number of far from equilibrium processes, including mechanical alloying (MA), mechanochemical processing (MCP), supercritical fluid processing (SCFP), and severe plastic deformation (SPD). Examples of the materials include the Ti-Al based intermetallic compounds and composites produced by MA, Ti base alloys and metal carbides synthesized by MCP, thin film Cu produced by SCFP and Al-Fe alloys produced by SPD. Details of the processes for each category of these materials and the enhancement of properties achieved by the retention of nanoscale structures in consolidations will be presented. The potential of these processes to substitute for conventional methods of production will also be examined.

9:00 AM
A Model for Mechanical Alloying in SPEX-8000 Mill: *Wei Wang*[1]; Deliang Zhang[1]; Alan Langdon[1]; [1]The University of Waikato, Dept. of Tech., Private Bag 3105, Hamilton New Zealand

Mechanical alloying (MA) is a novel ball milling process where a powder mixture in a ball mill is subjected to high-energy collision between balls and between balls and the wall of vial. SPEX-8000 mill, as one type of MA mills, are widely used for MA in laboratories all over the world. To understand its mechanism, a modelling study on the milling process of a SPEX-8000 mill is carried out. The movements of the vial and the balls in the vail are described 3-dimensionally using plain mechanics equations. Rotation and slip-

pery of balls during impacts between ball to ball and ball to wall of vial are included in the consideration. Flow diagrams are introduced to facilitate numerical calculations. Some interesting results, such as the impact energy and the impact number as a function of ball number and ball mass, are presented and discussed.

9:25 AM
Nanostructured TiC Powder Prepared through Carbothermic Reduction Enhanced by High Energy Milling: Ruiming Ren[2]; Zhenguo Yang[1]; *Leon L. Shaw*[1]; [1]University of Connecticut, Dept. of Metallu. and Mats. Eng., Storrs, CT 06269 USA; [2]Dalian Railway Institute, Dept. of Mats. and Eng., Dalian 116028 ROC

This paper describes a novel process for producing nanostructured TiC powder through carbothermic reduction of TiO2 enhanced by mechanical activation. Various analytical tools including x-ray diffraction, scanning electron microscopy, transmission electron microscopy and thermogravimetric analysis have been utilized to study the structural and phase evolution during milling and carbothermic reduction. It is demonstrated that high energy milling prior to carbothermic reduction has substantially reduced the reaction time and temperature of carbothermic reduction. The enhanced carbothermic reduction has been attributed to the structural and energy state changes of the reactants caused by the mechanical treatment prior to the reaction. Furthermore, it is proposed that these structural and energy state changes contribute to the enhanced carbothermic reduction through the increased reaction kinetics as well as the increased reaction driving force.

9:50 AM
Mechanical Alloying of Ti-based MMC Alloys by the Zoz Attritor: *Sedat Özbilen*[1]; Cemil Çetinkaya[1]; [1]Gazi University, Teknik Egitim Fakültesi, Metal Egitimi Bölümü, Teknikokullar, Ankara, Turkey

Ti-based advanced class materials systems such as Ti-aluminides, Ti-MMC's and CMC's, and Ti-Al-X alloy powders were prepared by the Zoz attritor under controlled atmospheres and processing times. Processed powders by mechanical alloying were characterised by XRD and SEM investigation. High yield in the MA MA processed powders were discussed and compared with those processed by other types of attritors and mills.

10:15 AM Break

10:35 AM Invited
Nano Zirconia Powder Synthesized by Mechanochemical Processing: Aaron C. Dodd[1]; *Paul G. McCormick*[1]; [1]University of Western Australia, Special Rsrch. Ctr. for Adv. Mineral and Mats. Processing, Nedlands 6907 Australia

Recent research has shown that mechanochemical processing of chloride precursors with oxide exchange reagents can be used as a technically simple and versatile technique for manufacturing a wide range of ultrafine oxide powders. In this process the precursors react, either during milling or during subsequent heat treatment, to form a composite powder consisting of nanocrystalline oxide particles embedded within a soluble salt matrix. The ultrafine oxide powder is then recovered by removing the salt by-product with an appropriate solvent. This paper reports on recently developed extensions to this basic technique for manufacturing ultrafine powders of stabilised zirconia. Factors influencing the particle size and degree of agglomeration will be discussed.

11:05 AM
Synthesis of Nanostructured WC-12%Co Coating Using Mechanical Milling and HVOF Thermal Spraying: *Jianhong He*[1]; Michael Ice[1]; Enrique J. Lavernia[1]; [1]University of California Irvine, Chem. and Biochem. Eng. and Mats. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA

A nanostructured WC-12% Co coating was synthesized using mechanical milling and high velocity oxygen fuel (HVOF) thermal spraying. The variation of powder characteristics with milling time and the performance of the coatings were investigated using SEM, X-ray, TEM and microhardness measurements. There is no evidence that indicates the presence of an amorphous phase in the sintered WC-12% Co powder, and the binder phases in this powder are the hcp and fcc Co solid solutions. Mechanical milling of up to 20 hours did not lead to the formation of an amorphous phase in the sintered WC-12% Co powder. During the initial stages of the milling, the brittle W carbide powders were first fractured into fragments and then embedded into the binder phase. This process gradually formed polycrystal nanocomposite powders of the Co binder phase and W carbide particles. The conventional cold welding and fracturing processes primarily occurred amongst the Co binder powders and polycrystal composite powders. The nanostructured WC-12% Co coatings, synthesized in the present study, consist of an amorphous matrix and W carbides with an average particle diameter of 35 nm. The coating possesses an average microhardness of 1135 DPH, and higher apparent toughness than that of its conventional counterpart. KEYWORDS: Nanostructured materials, amorphous, mechanical milling, and thermal spraying.

11:30 AM
Solid State Reactions in Nanometer Scaled Diffusion Couples: *Deliang Zhang*[1]; Danyang Ying[1]; [1]The University of Waikato, Dept. of Tech., Private Bag 3105, Hamilton New Zealand

When two solid phases such as elemental metals are in contact to form a diffusion couple, solid state reactions may occur at the interface as long as there exists a thermodynamic driving force for the reactions. To facilitate the solid state reactions, sufficiently high mobility of atoms is also required, and thus the diffusion couple has to be heated to above a critical temperature before the reaction can occur. For a given diffusion couple, one might expect that the reaction temperature and the reaction outcome is fixed, but observations reported in literature indicate that the reaction temperature and associated reaction products are closely related to the scale of a diffusion couple. When the dimension of a diffusion couple is in nanometer scale, the reactions often occur at much lower temperatures, and in association with the low reaction temperature, metastable phases may form as a result of the reaction. In order to understand the relationships between the scales of a given diffusion couple, the reaction temperature and the phase formation, we have investigated solid state reactions in diffusion couples of micrometer and nanometer scales. This paper will describe and discuss the results of this investigation. The diffusion couples of different scales were obtained by high energy ball milling of mixtures of elemental powders to different stages. The kinetics of solid state reactions and resulting phase formation were studied by using differential scanning calorimeter and x-ray diffractometer.

11:55 AM
Ti-TiN MMC's by Reactive Mechanical Alloying: *Sedat Özbilen*[1]; Abdülkadir Güllü[2]; [1]Gazi University, Metallu. Edu. Dept., Ankara Turkey; [2]Gazi University, Mech. Educ. Dept., Ankara Turkey

Reactive mechanical alloying of TiH2, Ti-TiH2 powder mix and mechanical alloying of Ti powder mix for 10 uninterrupted hours with sealed vial was carried out under nitrogen. This was followed by annealing of MA'ed powders under nitrogen to 950°C for thermal treatment to determine the stability of nitrides forming. Representative samples of each powder mix RMA'ed and thermally treated were characterised by XRD using Cu K radiation and examined by Cambridge ST40 Stereoscan SEM operating under 25kV to determine morphology and crystal structure change of the powders. It was observed that RMA under nitrogen cause to the formation of TiN in all the samples studied. Annealing under nitrogen to 950°C for 1 hour leads to the formation of Ti-TiN-Ti2N multiphase material in samples 1 and 2 (albeit with different ratios); to that of Ti-TiN composite material in sample 3. This shows that sintering of the MA'ed powders of TiH2, Ti-TiH2 and Ti is a promising

way for producing ultrafine grained Ti-TiN composite materials with RMA processing under nitrogen.

P/M: Current Research and Industrial Practices: Reactor Design and Synthesis

Sponsored by: Materials Design and Manufacturing Division, Powder Materials Committee

Program Organizers: F. D. S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgy Engineering, Rapid City, SD 57701-3995 USA; Enrique V. Barrera, Rice University, Metal Engineering & Materials Science Department, Houston, TX 77251 USA; G. M. Janowski, University of Alabama, Department of Materialss & Mechanical Engineering, Birmingham, AL 35294-4461 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Wednesday AM
November 3, 1999

Room: Caprice 2&3
Location: Omni Netherland Plaza Hotel

Session Chairs: Naresh N. Thadhani, Georgia Institute of Technology, School of Mat. Sci. and Eng., Atlanta, GA USA; Vitalli N. Nesterenko, University of California-San Diego, Dept. of AMES, La Jolla, CAUSA

8:30 AM Keynote
Thermal Plasma Synthesis of Ceramic Powders: *Patrick R. Taylor*[1]; [1]University of Idaho, McClure Hall, Moscow, ID 83844-3024 USA

General reactor design considerations for the formation of several ceramic powders by thermal plasma processing are presented, as well as the possible chemical reactions involved in the processes under the high temperatures generated. Silicon carbide, titanium carbide, titanium diboride, zirconium carbide, titanium carbide-iron, boron nitride and barium strontium titanate powders have been synthesized. For each ceramic system the precursors are vaporized completely in the hot zone of the reactor where the plasma source is located; the vapors then travel down the temperature gradient to meet conditions where specific chemical reactions may occur (with subsequent nucleation and/or growth); this is followed by rapid quenching to minimize back reactions or additional growth, thus enhancing the formation of metastable compounds that are consequently stabilized at room temperature. A description of the reactor system is presented along with theoretical and experimental results and conclusions from experiments.

9:05 AM Invited
Fullerenes and Nanotubes: Current Research and Future Directions: *E. V. Barrera*[1]; [1]Rice University, Mech. Eng. and Mats. Sci. MS-321, Houston, TX 77005 USA

Since the discovery of fullerenes in 1985 and nanotubes in 1991, a great deal of effort has been spent on understanding their basic properties. Since that time, efforts have also been initiated to develop applications for these very interesting lightweight carbon molecules. This presentation will highlight the various investigations going on to develop applications for fullerenes and nanotubes. Only the work in Materials Science at Rice University will be emphasized yet some general applications will also be given. In this presentation the use of fullerenes for nanocrystalline materials, magnetic thin films, and ultra hard materials will be discussed. Mechanical, electrical and thermal applications of nanotubes will be presented. Their powder properties will be emphasized and our ability to process them into materials will be discussed. Support for this research has come from NSF grant no. DMR-9357505, NASA grant no. NGT9-23, and ONR grant no. N00014-99-1-0246.

9:35 AM
Purification and Functionalization of Vapor Grown Carbon Nanotubes and Single Walled Nanotubes: *K. Lozano*[1]; E. V. Barrera[2]; [1]NASA-Johnson Space Center, Bradley Files, Houston, TX 77058 USA; [2]Rice University, Mech. Eng. and Mat. Sci. MS-321, Houston, TX 77005 USA

We present an overview of the methods used to purify and functionalize two types of carbon nanotube materials. Vapor grown carbon fibers (VGCFs) have been purified by oxidation methods and subsequently functionalized by different acid treatments. Single-walled nanotubes are purified by a somewhat similar process and can now be functionalized by a fluoridation method. Final products tend to be either dry VGCFs or SWNTs in solution. SWNTs can also be processed as a "Bucky Paper." Analyses of these materials involves SEM, TEM, WDS, and IR methods. The current understanding of the availability and current conditions of nanotubes will be identified. Support for this research has come from NSF grant no. DMR-9357505 and NASA grant no. NGT9-23.

10:05 AM Break

10:20 AM Invited
Powder Metallurgy at Los Alamos National Laboratory–Then and Now: *Sherri R. Bingert*[1]; [1]Los Alamos National Laboratory, MST-6, Metallu. Mats. Sci. and Tech., P.O. Box 1663, MS G770, Los Alamos, NM 87545 USA

Powder metallurgy has been an active part of Los Alamos programs for over fifty years. Although not all state-of-the-art, the capabilities represent one of the widest and most unusual collections of processing equipment in the country, most of which has been used for fabrication of components for internal requirements and for research and development, or demonstrations of processing technology. The past, present, and future operational capabilities of the section will be presented and include: powder making, powder characterization, reactive powder handling, powder conditioning, hot and cold uniaxial pressing, hot and cold isostatic pressing, extrusion, powder rolling, sintering and heat treatment facilities, atmospheric and low pressure plasma spray, flame spray, wire arc spray, alloy development, rapid and directional solidification, and consolidation and micromechanics modeling. The team has worked on a large variety of materials over the course of the LANL history including most metals, many oxides, composites, and other unusual materials. Some select projects and results from past and present programs will be presented.

10:50 AM
Titanium-Titanium Nitride MMC's by Reactive Mechanical Alloying: S. Ozbilen[1]; [1]Gazi Technical University, Metallu. Eng., Teknikokullar, Ankar, Turkey

Reactive mechanical alloying of TiH2 (sample #1),Ti-TiH2 (sample #2) powder mix and mechanical alloying (MA) of Ti (sample #3) powder mix for 10 uninterrupted hours with sealed vial was carried out under nitrogen. This was followed by annealing of MA'ed powders under N2 to 950°C for thermal treatment to determine the stability of nitrides forming. Representative samples of each powder mix RMA'ed and thermally treated were characterised by XRD using Cu Ká radiation and examined by Cambridge ST40 Stereoscan SEM operating under 25kV to determine morphology and crystal structure change of the powders. It is observed that RMA under N2 cause to the formation of TiN in all the samples studied. Annealing under N2 to 950°C for 1 hour leads to the formation of Ti-TiN-Ti2N multi-phase material in samples #1 and #2 (albeit with different ratios); to that of Ti-TiN composite material in sample #3. This shows that sintering of the MA'ed powders of TiH2, Ti-TiH2 and Ti is a promising way for producing ultrafine grained titanium-titanium nitride composite materials with RMA processing under N2.

11:20 AM
SEM Investigation of Mechanically Alloyed Materials Sys-

tems by Zoz Attritor: *S. Ozbilen*[1]; Mehmet Erdogan[1]; [1]Gazi Technical University, Metallu. Eng., Teknikokullar, Ankara, Turkey

One component material systems of brittle, ductile and in-between nature and two component materials systems such as brittle-to-brittle, brittle-to-ductile and ductile-to-ductile nature were alloyed mechanically by using a horizontal type of an attritor, i. e., a Zoz Attritor under a specified time and MA atmosphere. All powders and powder mixtures of above specified nature of material systems before and after MA by Zoz attritor were examined in detail under SEM with respect to powder particle size, shape, amount, distribution and surface texture. With this way, the capacity of Zoz attritor was tested in terms of powder yield suitable for optimised sintering cycle following MA processing.

Symposium to Honor Professor Julia R. Weertman: Nanocrystalline Materials I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Wednesday AM
November 3, 1999
Room: Rosewood
Location: Omni Netherland Plaza Hotel

Session Chairs: David A Rigney, The Ohio State University; Hector A. Calderon, IPN, Dept. Ciencia de Materiales Mexico, D.F. 07300 Mexico

8:30 AM Invited
Microscopic Description of Plasticity in Computer Generated Metallic Nanophase Samples: *Helena Van Swygenhoven*[1]; Alfredo Caro[2]; Miklos Spaczer[1]; [1]Paul Scherrer Institute, GFA, Villigen-PSI ch-5232 Switzerland; [2]Centro Atomico Bariloche, Bariloche 8400 Argentina

Large scale molecular dynamics computer simulations are reported on the elastic and plastic behaviour of two model fcc metals, Ni and Cu, with different stacking fault energies and mean grain sizes in the range of 3-12 nm. The microstructure of the different samples is discussed in terms of density, excess energy and the presence of low angle and high angle grain boundaries. It is shown that there exists a critical grain size below which a transition from an intra-grain to a purely inter-grain plasticity occurs and that this transition is determined not only by the grain size but also by the stacking fault energy and the presence of many low angle grain boundaries. Below the critical grain size all plasticity is accommodated in the grain boundary (GB). The strain rates increase strongly with decreasing grain size. The deformation mechanism is discussed in terms of a model based on GB viscosity controlled by a self-diffusion mechanism at the disordered interface, activated by thermal energy and stress. Above the critical grain size the strain rates are less sensitive to the grain sizes and plasticity is partly accommodated in the GB, partly inside the grains by the creation of stacking faults. Accurate analysis of the atomic configurations shows that the stacking faults are produced by the passage of partial dislocations generated and absorbed in opposite grain boundaries. The release of local stress by the emission of partial dislocations seems to be large enough to inhibit further emission of a partial in the same or in a neighbouring plane, making mechanical twining a rare event that has not been observed in these model materials. Atomic displacement analysis shows that deformation starts often at triple points, with GB sliding followed by the creation of intra-grain Shockley partial dislocations which glide on slip systems that are not necessarily those favoured by the Schmid factor. A quantitative evaluation of the sliding mechanism and the dislocation activity is given and discussed in terms of the stacking fault energy and the presence of many low angle grain boundaries.

9:00 AM
The Limiting Strength Properties of Nanocrystalline Materials: Ronald W. Armstrong[1]; *Gary D. Hughes*[2]; [1]University of Maryland, Dept. Mech. Eng., College Park, MD 20742 USA; [2]Laboratory for Physical Sciences, 8050 Greenmead Dr., College Park, MD 20740 USA

Updated consideration is given to a number of exciting reasons for investigating the strength properties of ultrafine grain size materials, for example, relating to the increased importance of the interfacial energy versus the volume free energy of crystals at nanocrystalline grain sizes. Emphasis is given to the predicted band of results to be expected for the Hall-Petch (H-P) reciprocal square root of grain size dependence for strength measurements based on the dislocation pile-up theory involving small numbers of dislocations. Further connection is made between low temperature H-P measurements for nanocrystalline strength properties and such other model characterizations as: (1) the shear strength of perfect crystals; (2) tensile instability limitations evaluated from single crystal strain hardening coefficients; (3) theoretical H-P microstructural stress intensities for plastic yielding or cleavage cracking; and, (4) relaxation of the grain boundary obstacle by pile-up stress influence on Coble-type diffusional creep.

9:15 AM
A Comparison of Deformation and Fracture of Nanolaminate with Nanocrystals: *Fereshteh Ebrahimi*[1]; Qing Zhai[1]; Dan Kong[1]; Zunayed Ahmed[1]; Alirio J. Liscano[1]; [1]University of Florida, Mats. Sci. and Eng., 221 MAE, P.O. Box 116400, Gainesville, FL 32611 USA

Nanostructures can be produced by reducing the grain size of polycrystals to the nano-regime or by manufacturing laminated structures with nano-size layer thickness values. Very high strengths can be achieved in both structures, however, their deformation mechanisms may be different. One major difference is the existence of large internal stresses, which can develop during processing as well as during deformation, in laminated structures. In this paper the deformation and fracture behavior of nickel and copper nanocrystals are compared with Cu/Ag and Cu/Ni nanolaminates. These materials are produced by electrodeposition and characterized using TEM, SEM, x-ray diffraction and tensile testing. The results of this study reveal that for comparable strength levels the pure metals fracture by the so-called knife-edge behavior, however the laminated structures show a behavior typical of two-phase materials with evidences of brittle type fracture. The latter behavior is attributed to the development of very high local elastic internal stresses in the laminated structures.

9:30 AM
Characterization of Nanostructured Ti Based Intermetallics: *Hector Alfredo Calderon*[1]; Vicente Garibay-Febles[1]; Antonio Cabrera-Perez[1]; Jose Gerardo Cabañas-Moreno[1]; Minoru Umemoto[2]; Koichi Tsuchiya[2]; [1]IPN, Dept. Ciencia de Materiales, UPALM ED. 9, Apdo. Postal 75-707, Mexico, D.F. 07300 Mexico; [2]Toyohashi University of Technology, Prod. Sys. Eng., Tempakucho, Toyohashi, Aichi 441 Japan

Microstructural characterization and measurement of mechanical properties of nanocrystalline Ti-Al alloys are reported. Mechanical alloying and sinterization assisted by plasma techniques are used to produce these nanocrystalline alloys. The alloys investigated include TiAl+X and TiAl3+X where X represents an addi-

tional element such as Cr, Mn or Fe. Mechanical alloyed powders have a microstructure, which consist of an amorphous matrix containing small crystalline domains. X-ray diffraction, conventional transmission electron microscopy (TEM) and high resolution (HREM) are used to identify the crystallites and the amorphous matrix after different milling times. Sintered specimens have a crystalline microstructure with nanometer-size grains. We obtained a full dense material by plasma assisted sintering technique. Alloys based on TiAl-X (X represents Cr and Mn) have mainly the phases alpha2 (DO19) and gamma (L10) distributed homogeneously in the form of nanograins, resembling a globular structure. HREM is used to identify phase nature and distribution. Al3Ti-X (X represents Cr, Fe and Mn) alloys show only one cubic phase (ordered L12). Mechanical properties (hardness and compression tests) are reported as a function of alloy content and grain size for both types of alloys. TEM is used to characterize the deformation mechanism during deformation at room temperature. During compression tests of Al3Ti-X no plasticity is recorded; fracture appears before any plastic behavior. However a relative higher fracture stress is found for the Cr and Mn containing alloys. No dislocation activity has been identified most likely owing to the small grains produced in these alloys. The AlTi-X alloys present plasticity and have some dislocation activity owing to the relative large grain sizes observed.

9:45 AM
Mechanical Behavior of Bulk Nanostructured Metals and Metallic Glasses: *Walter W. Milligan*[1]; [1]Michigan Technological University, Dept. of Metallu. and Mats. Eng., Hougton, MI 49931 USA

Professor Julia Weertman was a pioneer in the area of bulk nanostructured metals, and one of the key people who inspired this work. An overview will be given of the deformation mechanisms and mechanical behavior in iron alloys with grain sizes between 45 nanometers and 10 micrometers. The finer-grained alloys exhibit mechanical behavior which is strikingly similar to amorphous materials such as metallic glasses and polymers, including shear banding on planes not predicted by continuum mechanics, and an apparently pressure-sensitive yield criterion. The current level of understanding and a description of the work in progress will be presented.

10:00 AM BREAK

10:20 AM Invited
Grain Boundaries and Interfaces in Nanostructured Materials: *Harriet Kung*[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanocrystalline materials have been attracting rapidly increasing interest in the last decade mainly due to the potential gain in a wide range of engineering applications. One characteristic feature of nanostructured materials is the high density of grain boundaries and interfaces. The high interface to volume ratio has contributed to interesting physical properties in the areas of magnetics, optics, and load-bearing applications. There exist several processing routes to obtain such materials at a laboratory scale such as powder compaction, rapid solidification, severe plastic deformation, and vapor deposition. The strength exhibited by these structures can reach within a factor of two to three of the theoretical strength. As the structural scale approaches the nanometer range, the role and characters of grain boundaries and interfaces in affecting the macroscopic mechanical properties become increasingly important and need to be elucidated. This talk will review the current research efforts concerning the characterization of grain boundaries and interfaces in nanocrystalline materials. Specifically, the effect of the different processing techniques (gas condensation and compaction, physical vapor deposition and co-deformation) on the texture, types, and structure of interfaces in nanostructured metals (Cu, Pd) and composites (Cu-Nb, Cu-Cr, Cu-Ag) will be presented. In addition, the role of interfaces in controlling the mechanical properties and stability of nanostructured materials will be discussed as well.

10:50 AM
Mechanical Mixing and the Development of Nanocrystalline Material during the Sliding of Metals: *David A. Rigney*[1]; James E. Hammerberg[2]; [1]Ohio State University, Mats. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, Applied Theoretical & Computational Physics, Los Alamos, NM 87545 USA

Sliding contact of metals causes dramatic structural and chemical changes in the material adjacent to the interface. Details vary considerably for different choices of materials and sliding conditions. However, certain common features are well documented in the tribological literature. Careful studies of worn surfaces and of cross-sections provide evidence of large plastic strains and strain gradients and large strain rates and strain rate gradients. There is also good evidence for transfer, reactions with chemical species in the environment and mechanical mixing processes which affect friction and wear. These processes generate nanocrystalline material near the sliding interface and in the wear debris. Molecular dynamics simulations using more than 60,000 atoms help us to understand how this nanocrystalline material is generated during sliding. In particular, they reveal that the mechanically mixed layer and the nanocrystalline material have the same thickness, suggesting a mechanism for the generation of nanocrystalline material by mechanical processes.

11:05 AM
Towards a Topological Description of Nearly-Crystalline Networks: *Linn W. Hobbs*[1]; C. Exther Jesurum[1]; Alexander Coventry[1]; Vinay Pulim[1]; Bonnie Berger[1]; [1]MIT, LWH, 77 Massachusetts Ave., Rm. 1304962, Cambridge, MA 02139-4307 USA

"Crystallinity" is well defined as long-range translational and orientational order and describable in the traditional but arcane language of symmetry relations embodied in crystallography. The "amorphous" state is not similarly well defined or described, and certainly is not accessible through the concept of symmetry operations. In the world of nanomaterials, this somewhat artificial distinction in atomic arrangement blurs; and, indeed, models of the amorphous state have been proposed which amount to nanocrystalline assemblages (often polymorphic) with blurred boundaries. What both forms of atomic arrangement have in common is topology (or, loosely, connectivity), and there are topological rules—just like crystallographic rules—which limit the possible atomic arrangements, which are in any event few. The ability to sustain an arrangement in an amorphous state at all can, in fact, be shown to depend in a clear way on connectivity—or, more formally, on constraint theory. In this contribution, an algorithmic approach is described for generating atomic arrangements with local assembly rules, and a language for describing the resulting arrangements is laid out, both of which are applicable to crystalline and non-crystalline arrangements alike. The approach is based on local coordination units and is applied here to the tetrahedral net-

works formed by SiO2, Si3N4, SiC and Si. Some interesting consequences of amorphizability are shown to follow.

11:20 AM
Microstructural Evolution, Microhardness and Thermal Stability of SPTS-Processed Cu: *Honggang Jiang*[1]; Yuntian T. Zhu[1]; Igor V. Alexandrov[2]; Terry C. Lowe[1]; Ruslan Z. Valiev[2]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Ufa State Aviation Technical University, Instit. of Physics of Adv. Mats., Ufa 450000 Russia

Severe plastic torsional straining (SPTS) was used to prepare ultrafine-grained Cu materials. The evolution of their microstructure and microhardness with increasing torsional strains as well as their thermal stability was studied. Cellular subgrains with low-angle grain boundaries were first formed at low strain. Some of the low-angle grain Boundaries transformed to high-angle grain boundaries at higher strains, resulting in grain refinement. Correspondingly, microhardness was found to increase with increasing torsional strain. X-ray diffraction patterns showed the formation of crystallographic texture during the SPTS. SPTS also produced high internal stress and nonequilibrium grain boundaries. Differential scanning calorimetry (DSC) study revealed an exothermal peak between 180 and 280°C. Calculation of the energy released suggests that the peak was caused by both grain growth (recrystallization) and dislocation density reduction (recovery). Annealing twins were formed during the recrystallization. Microhardness started to drop at a very low temperature of 50°C, indicating a very low thermal stability.

11:35 AM
Grain-Size-Yield Stress Relationship: Analysis and Computation: *Marc Andre Meyers*[1]; David J. Benson[1]; Edward Fu[1]; [1]UCSD, Dept. of AMES, Mail Code 0411, La Jolla, CA 92093 USA

The seminal contributions of Julia Weertman to our understanding of the mechanical properties of nanocrystalline materials will be briefly reviewed. A constitutive equation predicting the effect of grain size on the yield stress of metals, based on the model proposed by M. A. Meyers and E. Ashworth (Phil. Mag.46(1982)73), is discussed and extended to the nanocrystalline regime. At large grain sizes, it has the Hall-Petch form, and in the nanocrystalline domain the slope gradually decreases until it asymptotically approaches the flow stress of the grain boundaries. The material is envisaged as a composite, comprised of the grain interior, with flow stress sigma(f), and grain boundary work-hardened layer, with flow stress sigma(fgb). Three principal factors contribute to the grain-boundary hardening: 1. the grain boundaries act as barriers to plastic flow; 2. the grain boundaries act as dislocation sources; 3. elastic anisotropy causes additional stresses in grain-boundary surroundings.The predictions of this model are compared with experimental measurements over the mono,micro, and nanocystalline domains. Computational predictions are made of plastic flow as a function of grain size incorporating elastic and plastic anisotropy as well as differences of dislocation accumulation rate in grain boundary regions and grain interiors.This is the first plasticity calculation that accounts for grain size effects in a physically-based fashion. Research supported by US ARO MURI Program at UCSD.

ASM/TMS Distinguished Lectureship in Materials and Society

11:45-12:45 PM
Room: Mayflower I & II
Omni Netherland Plaza Hotel

Lecturer: Dr. Mary Lowe Good
Topic: Materials in the 21st Century: Global Innovation vs. Discovery

About the Lecturer: Dr. Mary L. Good is the Donaghey University Professor at the University of Arkansas, Little Rock and serves as the managing member for Venture Capital Investors, LLC, a group of Arkansas Business Leaders who expect to foster growth in the area through the opportunistic support of technology-based enterprises. Dr. Good also presently servers on the Board of Biogen, a successful biotech company in Cambridge, Massachusetts; IDEXX Laboratories of Westbrook, Maine; and the Lockheed Martin Energy Research Corporation Board of Oak Ridge, Tennessee.

Beryllium and Beryllium Alloys; Nuclear and Structural Applications: Beryllium Alloys-Industrial Applications

Sponsored by: Jt. ASM-MSCTS/TMS-SMC, Nuclear Materials Committee
Program Organizers: Michael F. Stevens, Los Alamos National Laboratory, MS G755, Los Alamos, NM 87545 USA; David J. Michel, Naval Research Laboratory, Code 6301, Washington, DC 20375 USA

Wednesday PM
November 3, 1999
Room: Salon M
Location: Omni Netherland Plaza Hotel

Session Chair: Robert Hanrahan, Los Alamos National Lab, MST-6, Los Alamos, NM 87545 USA

2:00 PM
Near-Net Shape HIPing of Beryllium Powder: *Paul W. Stanek*[1]; B. Reardon[1]; [1]Los Alamos National Laboratory, Mail Stop G-770, Los Alamos, NM 87545 USA

The driving forces for near-net shape (NNS) beryllium part production will be discussed in the context of economics as well as ES&H. The production of NNS beryllium parts using hot isostatic pressing (HIPing) in conjunction with computer modeling and the advances in these two techniques will also be discussed. The advances in HIPing involve incorporating the assistance of MATSYS, Inc. to determine the in situ density changes of the beryllium powder. Furthermore, controlled experiments to determine the impact of friction between the powder and the steel HIP container on the final HIPed part will be presented. Computer modeling, in terms of finite element as well as micromechanical, has proven beneficial to NNS production. These benefits range from optimizing HIP schedules, to improving HIP can design, to reducing the total number of HIP experiments necessary to produce a NNS part. The intimate relationship between experiment and modeling necessary for NNS part production will be discussed in terms of experimental design and in novel ways of linking experiments with models.

2:25 PM
Equal Channel Angular Extrusion (ECAE) of Beryllium: *R. D. Field*[1]; K. Ted Hartwig[2]; Carl T. Necker[1]; [1]Los Alamos National Laboratory, MST-6, Mail Stop G770, Los Alamos, NM 87544 USA; [2]Texas A&M University, Dept. of Mech.Eng., College Station, TX 77843-3123 USA

The Equal Channel Angular Extrusion (ECAE) technique has been applied to a P/M source Be alloy. Two passes have been successfully completed, using two different processing routes, on Ni canned billets of Be at 400°C. No cracking was observed in the billets, and significant grain refinement was achieved. In this presentation, microstructural features of the extruded billets will be presented, including fine recrystallized grain structures after heat treatments up to 800°C. Dislocation structures will be discussed, including evidence for c+a slip in the as-extruded material found by transmission electron microscopy. Significant crystallographic texture

has been found to develop during the ECAE process, which will be discussed in terms of the unique deformation mode of the process.

2:50 PM
Fabrication and Filling of NIF-Sized Beryllium Alloy Capsules: *Robert W. Margevicius*[1]; L. J. Salzer[1]; A. Nobile[1]; M. A. Salazar[1]; L. R. Foreman[1]; [1]Los Alamos National Laboratory, MS G770, Los Alamos, NM 87545 USA

Beryllium as the ablator for a hydrogen-filled NIF capsule offers many advantages. However, fabrication of a hollow beryllium capsule without any processing inhomogeneities (e.g., weld joints or plugs) remains a technological challenge. One method being pursued is the joining of beryllium hemispheres. Three areas of active interest are materials fabrication, joining, and filling. Be-Cu alloy materials were processed by both cast and powder metallurgy routes. The cast material was typically purer with a large grain size; the powder metallurgy alloy had a slightly higher oxide content but its grain size remained small. Based on past efforts, a modified brazing technique with Cu or Al was used to join the two halves. The joints were microstructurally undetectable in many places, however, optimization of this process is expected to produce better joints still. High resolution x-ray radiography has been valuable in determining braze-line composition profiles. Filling with isotopic hydrogen was investigated by two methods. The first involved diffusion-filling of empty, hollow spheres. Spheres were joined (as described above) under vacuum. The spheres were then placed in a high pressure, high temperature deuterium cell. Internal pressures to 6.7 MPa were obtained by this method. The second method involved trapping gas in the hollow cavity during the brazing cycle. Technical barriers to this method still need to be overcome. This talk will present the results from the work involving material fabrication, hemisphere joining, and hydrogen filling.

3:15 PM
Rapid Net Shape Forming of Spherical Beryllium Powder into a Complex Shape: *D. E. Dombrowski*[1]; W. J. Haws[1]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA

Rapid Net Shape Forming is an innovative manufacturing process which can produce intricate net shape products. In the final step of the process, a metallic powder in a seamless can is consolidated to full density by Hot Isostatic Pressing (HIP). Application of this process to beryllium powder made by inert gas atomization will be described. The process has been successfully demonstrated with a simple I-beam shaped part and a complex shape used in the aerospace industry. This work was funded under DARPA cooperative agreement no. NOOO 14-95-2-0019.

3:40 PM Break

3:50 PM
Be-Al Arc Welding Process Development for Casting Repair: *Brian J. Smith*[1]; [1]Starmet Corporation, 2229 Main St., Concord, MA 01742 USA

The Beralcast family of Be-Al alloys represents a recently developed metallurgical alternative to cast Al alloys for many different applications. Casting repair or re-work processes, such as welding, have also been developed to increase foundry yields. Welding presents similar technical challenges as casting because the substrate metal is melted and re-solidified all along the weld fusion line. Problems such as solidifications shrinkage, hot-cracking, Al segregation and gas porosity had been encountered in arc welded Beralcast castings. A process development program was performed to establish the arc welding process for Beralcast Be-Al castings. The weld development program evaluated many weld process variables such as current mode, shielding gas composition and purity, filler metal composition, machine settings and pre-and post-weld heat treatments and determined their effect on resultant weld characteristics. All welds were characterized using non-destructive and destructive testing techniques. A review of weld and HAZ microstructures and weld tensile properties will be provided and related to the weld process settings.

4:15 PM
Sheet Forming Aluminum-Beryllium Alloys: *W. J. Haws*[1]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA

Processes for hot bending aluminum-beryllium sheet to 90' were developed. Forming experiments were performed between 4OOoF and 12OOoF. Optimum forming temperatures were determined for two gages of cross-rolled AlBeMet (R 162 (62% Be) sheet. Minimum bend radius ratios for both gages were determined. Smaller bend radius ratios were obtained compared to previous work on aluminum-beryllium alloys. Specimens were examined metallographically to determine if cracking had occurred during bending. Cracking, when it occurred, was observed to occur both the aluminum phase or at the aluminum-beryllium interfaces. Cracking within the beryllium phase was not observed. This work was funded by DARPA under Navy contract NOOO 14-96-C-0 1 7 1.

4:40 PM
Producing Ultra Light Weight Aluminum Beryllium Panels Using the Low Density Core Process: *M. Svilar*[1]; D. S. Shih[2]; P. Stanek[3]; [1]Brush-Wellman Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA; [2]The Boeing Company, Mail Code S1111041, P.O. Box 516, St. Louis, MO 63166-0516 USA; [3]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA

Ultra Light Weight porous core 38% Al-62% Be alloy sandwich panels have been produced in the laboratory using Boeing's proprietary Low-Density-Core (LDC) technology originally developed for titanium. The expanded core Al-Be panels had a core porosity exceeding 40 % as measured by volume with the porous core thickness comprising 60% of the overall sheet thickness. Based on structural finite element models, these values are expected to increase the specific stiffness 20 % to a calculated value of approximately 110 GPa/(g/CM3) compared to the experimentally determined value 91 GPa/(g/CM3) for standard solid AlBeMet\@ 162 sheet. Potential advantages of the LDC AlBeMet 162 panels are superior specific stiffness in all orientations, and elevated service temperature as well as costs benefits from metal working processing compared to composite lay-up fabrication. This work was sponsored by DARPA and ONR, contract number NOOO 14-96-C-03 98.

5:05 PM
Plasma Spray-Forming of Beryllium Components: *Richard G. Castro*[1]; Kendall J. Hollis[1]; Keith E. Elliott[1]; [1]Los Alamos National Laboratory, MS G770, Los Alamos, NM 87545 USA

Plasma spray technology has been used extensively for applying coatings and producing spray-formed components of metals, ceramics, intermetallics and cermets. During the 1960's and '70's plasma spray technology was investigated by Union Carbide Speedway Laboratories and the Atomic Weapons Establishment for producing beryllium spray-formed components for defense related applications. Most recently, beryllium plasma spray technology was investigated at Los Alamos National Laboratory for producing thick (>10mm) beryllium armor tiles for first wall applications for the International Thermonuclear Experimental Reactor (ITER), the next generation magnetic fusion energy devices. Information will be presented on the manufacturing techniques used for producing plasma sprayed-formed components of beryllium, and the mechanical and thermal properties of the deposited materials. Discussions will also be presented on the potential use of this technology for producing spray-formed components of aluminum-beryllium alloys.

Computational Materials Science at the Microstructural Scale: Mesoscale Models for Solidification

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Computational Materials Science & Engineering, Phase Transformations Committee

Program Organizers: Elizabeth Holm, Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185-0304 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark A. Miodownik, Sandia National Laboratories, Materials and Process Modeling, Albuquerque, NM 87185-1411 USA; J. P. Simmons, AFRL-MLLM, Dayton, OH 45433 USA; Prof. Peter W. Voorhees, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208

Wednesday PM
November 3, 1999

Room: Caprice 1&4
Location: Omni Netherland Plaza Hotel

Session Chair: Peter W. Voorhees, Northwestern University, Dept. of Mats. Sci. & Eng., Evanston, IL 60208-3108 USA

2:00 PM Invited
Phase-Field Modeling of Solidification Microstructures: *Alain Serge Karma*[1]; [1]Northeastern University, Physics Dept., 360 Huntington Ave., Boston, MA 02115 USA

One of the main present challenge for the phase-field approach is to cope with the disparity of lengthscales between the atomic scale capillary length and the microstructural scale, several orders of magnitude larger. The first part of this talk will present recent progress made in coping with this difficulty that combines an improved asymptotic analysis of the phase-field model and a Monte-Carlo based algorithm for the efficient solution of the large scale diffusion field. This algorithm offers the same advantage as adaptive mesh refinement, in that the computational cost scales like the area of the growing structure rather than the volume, but is simpler to implement numerically in three dimensions without significant compromise in accuracy. This approach now makes it possible to model three-dimensional dendritic growth in a range of dimensionless undercooling (0.01-0.1) that is directly relevant for experiments. The second part of this talk will focus on the application of the phase-field approach to model the formation of eutectic colony structures during directional solidification of ternary alloys. In this case modeling confirms the prediction of an oscillatory morphological instability, in agreement with recent experiments, but also reveals difficulties in modeling (solid-solid-liquid) tri-junctions within a phase-field approach and provides new insight into what controls their mobility.

2:40 PM
Computational Simulation of Convection Influenced Solidification: *Damir Juric*[1]; [1]Georgia Institute of Technology, School of Mech. Eng., Atlanta, GA 30332-0405 USA

A front tracking method is used to directly simulate the solidification of pure materials and alloys in the presence of fluid convection. The method is based on an explicit Lagrangian tracking of the phase interface and combines the ability to model phase change, complex interface dynamics, fluid flow, heat and solute transport. The method allows for fully coupled solid and fluid motion and shrinkage or expansion upon solidification. The tracked phase boundary is treated as an imbedded interface through which the conservation laws are supplemented by the appropriate local interfacial delta function source terms for interphase mass transfer, latent heat and solute rejection. We use the method to simulate the growth of dendritic structures of a pure material in a shear flow and compare the results to those with no flow. The growth of binary alloy grains in a shear flow and the coupling of the grain motion to the flow is investigated.

3:00 PM
Phase-Field Simulations of Dendritic Growth with Convection: Xinglin Tong[1]; *Christoph Beckermann*[1]; Alain Karma[2]; [1]University of Iowa, Dept. Mech. Eng., 2412 SC, Iowa City, IA 52242 USA; [2]Northeastern University, Dept. Physics, 112 Dana Research Ctr., Boston, MA 02115 USA

A phase-field model is presented for numerically simulating dendritic growth in the presence of melt convection. The flow field is obtained by solving the Navier-Stokes equations modified to account for the interfacial drag in the diffuse interface region. The model is validated for several limiting cases involving flow in regions of complex structure. Results are presented for the growth of a single equiaxed dendrite of pure materials having different surface tension anisotropies. The dendrite tip operating state selection and the development of thermal noise-induced sidebranching in the presence of flow are analyzed in detail.

3:20 PM Break

3:40 PM Invited
Direct Numerical Simulation of Dendritic Microstructures: *Jon Dantzig*[1]; N. Provatas[1]; N. Goldenfeld[2]; [1]University of Illinois, Mech. & Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]University of Illinois, Dept. of Physics, 1206 W. Green St., Urbana, IL 61801 USA

Understanding pattern selection during dendritic solidification is an important problem for materials scientists and engineers. The microstructure formed during solidification affects the properties of the material in service, and cannot be changed readily by subsequent solid-state processing. In this talk, we describe recent computations using phase-field models to directly simulate dendritic growth, in order to relate the microstructural features to the processing conditions. In this method, the liquid-solid interface is modeled as a diffuse region whose thickness is characterized by an order parameter, known as the phase field. One of the difficulties encountered when applying the phase field method is the conflicting requirements of high resolution needed to successfully capture the physical phenomena at the interface, and the simultaneous need to fully resolve the diffusion field ahead of the advancing front. We employ an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion field. Examples are given for pure, isolated dendrites, and for directionally solidified alloy dendrites, with comparisons to experimental observations.

4:20 PM
Obtaining Parameters of the Phase Field Model from Atomistic Simulations: *Jeffrey J. Hoyt*[1]; Mark D. Asta[1]; [1]Sandia National Laboratories, Computat. Mats. Sci. Dept., MS 9161, Livermore, CA 94550 USA

The phase field model has been proven to be quite effective in the numerical modeling of solidification and other phase transformations. In order to model real alloy systems however, several materials parameters are required as input to the phase field model and these parameters are often very difficult to measure experimentally. In this talk it will be shown how molecular dynamics simulations utilizing interatomic potentials derived from the embedded atom method can provide the necessary phase field parameters. Specifically, the case of binary solidification will be investigated where three quantities are required: the diffusion coefficient in the liquid, the solid-liquid interface velocity as a function of undercooling and the solid-liquid interfacial free energy. All parameters have been computed for Cu and Ni and the interface properties have been determined as a function of cyrstallographic orientation for the three low index directions 100, 110 and 111.

4:40 PM
Modeling of Primary Dendrite Arm Spacings in Al-Si-Mg Tenary Alloys: *Qingyou Han*[1]; Srinath Viswanathan[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

A finite difference model for the growth of a cellular and dendritic array in multi-component alloys has been used to predict the primary dendrite arm spacings in Al-Si-Mg alloys. The primary spacing selection mechanism is considered and treated in the model. Thermodynamic calculations have been carried out to obtain data on liquidus slopes and solute distribution coefficients in Al-Si-Mg alloys. The data are used in the model to predict primary dendrite spacing. Comparison of the theoretical predictions with experiments shows good agreement. The model is then used to discuss the effect of temperature gradient and growth velocity on primary dendrite arm spacings. Simple analytical equations for primary dendrite arm spacing as a function of temperature gradient and growth velocity are developed.

5:00 PM
Asymptotic Analysis of Particle Engulfment: *Layachi Hadji*[1]; [1]University of Alabama, Math. Dept., Box 870350, 345 Gordon Palmer Hall, Tuscaloosa, AL 35487-0350 USA

An asymptotic analysis is conducted on the interaction between an insoluble spherical particle and an advancing solid-liquid interface when the particle is in the near-contact region, i.e. gap thickness is much smaller than the particle's radius. The analysis considers only thermal effects in a pure substance. The interface equilibrium temperature includes the undercooling effects due to the front curvature and to the long-range intermolecular forces in the thin melt film behind the particle. The small gap analysis allows the determination of the crystal-melt shape near the particle from which the minimum gap thickness is obtained. The latter is then used to calculate the forces that act on the particle under steady-state conditions. These forces consist of the viscous drag force, which opposes the movement of the particle, and the Van der Waals force which pushes the particle away from the front. A force balance then yields an expression for the growth rate which separates pushing from engulfment. This expression depicts explicitely the dependence of the threshold velocity on the physical parameters.

General Abstract Sessions: High Temperature Behavior II

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday PM, November 3, 1999
Room: Salon D&E
Location: Omni Netherland Plaza Hotel

Session Chairs: Michael J. Mills, Ohio State University, Department of Materials Science & Engineering, Columbus, OH 43210 USA; Carl Boehlert, John Hopkins University, Dept. of Mech. Eng., Baltimore, MD 21218 USA

2:00 PM
The Effect of Trace Levels of Nickel on Creep Behavior of Ti-6Al-2Sn-4Zr-2Mo: *Robert Walter Hayes*[1]; Gobal Viswanathan[2]; Michael Mills[2]; [1]Metals Technology Inc., Northridge, CA 91324 USA; [2]Ohio State University, Columbus, OH 43210 USA

The effect of trace levels of Ni on the high temperature creep behavior of the alloy Ti-6Al-2Sn-4Zr-2Mo has been investigated over the temperature range 510 to 565°C at stress levels ranging from 172 to 413 MPa. Material containing high and low levels of Ni were heat treated in order to produce a lath type microstructure. Although the intent was to produce identical microstructures, actual results reveal that the microstructures differ somewhat between the high and low Ni material. The creep results indicate that increased levels of Ni lead to increased minimum creep rates and this arguement is strengthened by the fact that although the high Ni material posseses a more creep resistant microstructure, the creep rates are higher at identical temperatures and stress levels relative to the low Ni material. The creep stress exponents obtained for both materials suggest a dislocation creep mechanism and are in good agreement with a recently proposed modified jogged-screw model for creep of gamma TiAl. The apparent activation energy for creep decreases with increasing trace levels of Ni, consistent with intrinsic self-diffusion values recently reported for alpha titanium. These results will be discussed in the framework of the modified jogged-screw model for creep. Detailed microstructural analysis will be presented in a companion paper.

2:20 PM
A Study on Creep Deformation Structures in a Ti-6Al-2Sn-4Zr-2Mo Alloy: *Gopal Babu Viswanathan*[1]; Robert W. Hayes[2]; Michael J. Mills[1]; [1]Ohio State University, 477 Watts Hall, 2041 College Rd., Columbus, OH USA; [2]Metals Technology, 19801 Nordhoff St., Northridge, CA 91324 USA

In a companion talk, the high temperature creep behavior of a Ti-6Al-2Sn-4Zr-2Mo (T-6242) alloy having a lath microstructure with trace amounts of Ni is discussed. There, it was shown that increased amounts of Ni increased the minimum creep rates over the temperature range 510-565°C in the applied stress levels ranging from 170-410 MPa. In addition to the increased minimum creep rates, the activation energy values were found to decrease with increased Ni content. In this study, the dislocation structures after the creep deformation have been analyzed. Preliminary analysis reveal that the dislocation density is relatively very high in the alpha phase compared to that of the beta phase, suggesting that the creep deformation is dominated by the a-phase. a-<11-20> type prism slip dominates the deformation in the a phase, though a-<11-20> pyramidal slip has been observed in those grains favorably oriented towards the stress axis. The a-<11-20> type dislocations however are mostly aligned in screw orientation and seem jogged along screw segments. Detailed analysis of these dislocations and other microstructural features along with possible creep mechanism that might be operative in these alloys will be presented.

2:40 PM
Study of the Effect of SRO on the Mechanical Behavior of Ti6wt%Al: *T. Neeraj*[1]; J. L. Robertson[2]; G. B. Viswanathan[1]; M. J. Mills[1]; [1]Ohio State University, 477 Watts Hall, 2041 College Rd., Columbus, OH 43201 USA; [2]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Building 7962 MS 6393, Oak Ridge, TN 37831-6393 USA

Titanium alloys are used extensively as a structural material because of its high strength to weight ratio. However, these materials creep extensively at room temperature at stress levels far below the yield strength. Recently, it has been shown that the reason for this behavior is the low work hardening exponent exhibited by titanium alloys. The alpha phase of commercial titanium alloys deform in a planar fashion leading to low work hardening in the material. It has been suggested in the literature that short range ordering (SRO) of Ti and Al atoms promote planar slip within the alpha phase of titanium alloys. In this study, a single phase Ti-6wt% Al alloy has been chosen to study the alpha phase behavior. The SRO in the alloy has been characterized using neutron diffraction after various heat treatments. This was followed by mechani-

cal testing under both creep and constant strain rate conditions. The resulting dislocation microstructures have been characterized using transmission electron microscopy.

3:00 PM
Creep Behavior of Nb-11Al-41Ti-1.5Mo-1.5Cr: *Robert Walter Hayes*[1]; Wole Soboyejo[2]; [1]Metals Technology Inc., 19801 Nordhoff St., Northridge, CA 91324 USA; [2]Ohio State University, Dept. of Mats. Sci. and Eng., Columbus, OH 43210 USA

The high temperature creep behavior of the Nb-based intermetallic alloy Nb-11Al-41Ti-1.5Mo-1.5Cr (at. pct.) has been investigated over the temperature range 650 to 760°C at stress levels ranging from 69.4 to 275.7 MPa. The material was forged and direct aged at 750°C for 25 hours. The microstructure of this alloy consists of a fine mixture of orthorhombic and ordered beta phase (B2) with the orthorhombic as the predominant phase. Creep specimens were obtained in the longitudinal and transverse orientations. The apparent activation energy for creep as well as the creep stress exponents were measured in both orientations. The apparent creep activation energies and stress exponents differ between the two orientations indicating an orientation dependence of the creep behavior under the present experimental conditions. The discussion of the results will provide some suggestions for the observed creep behavior. Ideas for further research on this important class of materials will also be presented.

3:20 PM
Dislocation Processes and Deformation Behavior in Polycrystalline Fe-Ni-Al Intermetallics: *Paul R. Brenner*[1]; M. F. Savage[1]; M. J. Mills[1]; R. D. Noebe[2]; [1]Ohio State University, Mats. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]NASA Lewis Research Center, Cleveland, OH USA

Study of the ternary Fe-Ni-Al system accomplishes further development of alternative high temperature, high performance alloys while expanding our understanding of the effects of large solute additions on the slip behavior and mechanical response of an ordered system. A study of deformation behavior over a range of temperatures up to 1000 C will be coupled with an analysis of slip and dislocation mechanisms. FeAl and NiAl systems exhibit extremes in behavior which could be complementary for high temperature, high performance applications. Alloys of the FeAl system exhibit relatively low intrinsic strengths and moderate ductility at room temperature whereas the NiAl system exhibits high intrinsic strengths and little ductility at room temperature. The ternary Fe-Ni-Al system enables systematic study of the behavior of the isomorphous B2 ordered structure. The system exhibits a pseudo-binary regime for Al compositions below 50 at%, enabling a study of alloys varying the content of Fe and Ni while Al content is held at 40 at%. This characteristic of the Fe-Ni-Al ternary system offers a unique opportunity to explore the effects of large solute additions on slip behavior and dislocation mechanisms such as previously observed slip transitions from a<111> type slip to a<010> slip which occurs on the iron-rich side of the phase field.

3:40 PM Break

3:50 PM
Selected Observations and Analysis for Diffusion in the Fe-Ni-Al System at 1000°C: *Yong-Ho Sohn*[2]; Mysore A. Dayananda[1]; [1]Purdue University, School of Mats. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907-1289 USA; [2]University of Connecticut, Dept. of Metall. and Mats. Eng., 97 North Eagleville Rd., Storrs, CT 06040 USA

Isothermal multiphase diffusion in the Fe-Ni-Al system at 1000°C was experimentally investigated by using solid-solid diffusion couples assembled with beta(B2), beta'(bcc), gamma(fcc) and (beta+gamma) alloys. The couples were examined for diffusion structures by optical microscopy and scanning electron microscopy and analyzed for concentration profiles and diffusion paths by electron microprobe analysis. Unusual diffusion structures and diffusion paths were observed for couples with terminal alloys in the vicinity of the miscibility gap between the beta and beta' phases; relative extrema in concentration profiles and development of zero-flux planes were frequently encountered. Ternary interdiffusion coefficients were calculated on the basis of the Boltzmann-Matano analysis over a wide range of compositions in the beta and beta' phase regions; ternary intrinsic diffusion coefficients were also determined on the basis of marker motion studies. In addition, average ternary interdiffusion coefficients were evaluated over selected composition ranges from single diffusion couples. These results will be presented and discussed to assess the relative diffusion behavior of the components, the ternary diffusional interactions, and the development of diffusion paths and diffusion structures in the Fe-Ni-Al system. This research carried out at Purdue University was supported by Argonne National laboratory.

4:10 PM
Formation of Ternary Phases to Inhibit Stress Corrosion Cracking in Al-Mg-Mn Alloys: *Mark C. Carroll*[1]; P. I. Gouma[1]; M. J. Mills[1]; G. S. Daehn[1]; Brady R. Dunbar[2]; [1]The Ohio State University, Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Century Aluminum Corporation, P.O. Box 98, Ravenswood, WV USA

The versatility and low cost of aluminum alloys of the Al-Mg-Mn 5000 series have made them ideal in a wide range of applications where a balance is required between strength, corrosion resistance, and cost. Alloys such as 5083, 5086 and 5456, which contain magnesium in levels above the room temperature saturation limit, still maintain a great deal of popularity although stress corrosion cracking (SCC) concerns exist in these and other high-Mg (above 3.5 wt%) alloys due to precipitation of the Al-Mg beta phase (Al8Mg5) at grain boundaries. Additions of minor amounts of elements such as zinc and copper to these high-Mg systems have been shown to precipitate along with Mg as ternary Al-Mg-X phases. The characteristics of these phases will differ from that of the binary Al-Mg beta-phase precipitates, both in structure and in the corrosion mechanisms by which they interact with the matrix or the precipitate-free zones (PFZs) which border grain boundaries. An evaluation of the characteristics of high-Mg materials alloyed with these elements, clarified through transmission electron microscopy (TEM), will be addressed. The physical structure of these phases along with the potential ramifications on the overall corrosion properties of the alloys which contain them will be discussed.

General Abstract Sessions: Advances in Processing and Heat Treatment III

Sponsored by: TMS
Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Lab., PO Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday PM	Room: Salon F&G
November 3, 1999	Location: Omni Netherland Plaza Hotel

Session Chair: Thomas R. Bieler, Michigan State University, East Lansing, MI 48824-1226 USA

2:00 PM
Effects of Minor Alloying Additions in As-Cast FeAl-Based Materials Revealed through Differential Scanning Calorimetry (DSC) And Optical Microscopy: *Aszetta Denise Jordan*[1]; Oswald N.C. Uwakweh[2]; Phil J. Maziasz[3]; [1]University Of Cincinnati, 5990 N. Pete Dawson Rd., Tucson, AZ 85704 USA; [2]University Of Cincinnati, Mats. Sci. and Eng., 498 Rhodes Hall, Cincinnati, OH 45221-0012 USA; [3]Oak Ridge National Laboratory,

Metals and Ceramics, P.O. Box 2008, Bldg. 4500 S MS6115, Oak Ridge, TN 37831-6115 USA

Several as-cast FeAl-based materials with minor additions of Mo, Zr, C and B elements were studied using Differential Scanning Calorimetry (DSC) and optical microscopy. Through DSC measurements, the materials in their as-cast condition were found to exhibit a metastable transition below 850°C which could subsequently be eliminated through annealing. It is proposed that the metastable transition is attributed to a processing-induced phenomena which results via compositional inhomogeneity during cooling of the cast material or from artifacts introduced during sample preparation prior to DSC measurements. Optical microscopy of the cast material revealed that minor elemental additions significantly influence or alter the microstructure producing grain structures ranging from equiaxed to dendritic type growths.

2:20 PM

Axi-Symmetric Filling Model of a Wheel Mold: *Melissa I. Bloch*[1]; Daniel P. Cook[2]; Armand J. Beaudoin[1]; Jon A. Dantzig[1]; [1]University of Illinois, Mech. and Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Reynolds Metals Company, Corp. Rsrch. and Dev., Corp. Tech. Ctr., 13203 N. Enon Church Rd., Chester, VA 23831 USA

Low pressure die casting is an important process for the manufacturing of automotive parts. Lower filling rates reduce melt atomization found in conventional die casting, but other defects which lead to the production of scrap material can still occur. These include cold folds, misruns and other defects associated with the filling process. In this work, we model the filling process during low pressure die casting of automotive wheels. We compare our computational results with in-plant observations, and identify mechanisms for defect formation and remediation.

2:40 PM

Recovery of Some Indispensible Values from Welding Electrode Mantling Scrape: *Suzan S. Ibrahim*[1]; Alber A. Sadek[2]; [1]Central Metallurgical Research and Development Institute, Mineral Processing; [2]Central Metallurgical Research and Development Institute, Welding Tech. Dept.

Undoubtedly the most widely used arc-welding process is the manual metal arc welding (MMAW). It is possible to weld a variety of metals with the same equipment simply by changing the electrode type. The main reason for using a flux covering in MMAW is to protect the molten metal from atmospheric contamination. At the same time, the flux fulfills a number of functions, all of which contribute to the success of the welding operation. However, during fabrication of flux covered electrodes, the residual flux forms a considerable problem. Depending on its storage condition, production schedule, grain size and etc., some companies reused the residual flux within the range of 5 to 10% of the lump sum of the total charge. However, this process is inadequate. Thus, two typical grape welding electrode covering flux (scrap samples, coded A & B) were supplied from a national welding electrode manufacturing company, to recover their valuable containing materials. Mineralogical and chemical investigation showed that 56.4% and 89.72% by weight of the scrap samples contained valuable constituents of various forms of iron, manganese and titanium minerals. They were found in appreciable amounts, reaching 16.24%, 6.69% Fe2O3, 4.5%, 3.88% MnO2 and 13.43%, 33.79% TiO2 in the two samples respectively. Aggresive attention was conducted, at appropriate conditions to librate the metallic scrap constitutes from the binders, soft material which is shown to be bentonite in the first sample (A) and cellulosic binder in the second one (B). Hydrocyclon classification was carried out to reject the binding material found below 35 mm. Tabling technique was conducted to remove light silica sands. Magnetic separation was finally employed to separate the metallic values from each other depending on their variation in their magnetic properties. The studies showed the amenability of the welding scrap wastes to be recovered into valuables especially ilmenite, leucoxene and rutile by simple physical separation techniques.

3:00 PM

Deposition and Properties of Non-Equilibrium Thin Films by Sputtering: *Jinn P. Chu*[1]; W. C. Luu[1]; J. K. Wu[1]; S. J. Lee[1]; C. W. Chang[1]; [1]National Taiwan Ocean University, Instit. of Mats. Eng., No. 2 Pei-Ning Rd., Keelung 20224 Taiwan

Physical vapor deposition techniques such as sputtering can be characterized by film growth far from equilibrium due to the rate of arrival of material at the growing surface and the short accommodation time of the atoms at the film/vapor interface. Due to the growth dynamics occurring during deposition the quenching rate of atoms from the vapor phase at the film surface can approach 10^{15} K/sec. As a result of non-equilibrium processing conditions, unique thin film material compositions, microstructures and properties are possible. Examples of non-equilibrium thin films fabricated by sputter deposition in our laboratory, including A-15 Cr, Cu-C and Cu-Mo alloy films, will be presented. The influence of processing condition on the crystallinity, microstructure, properties and elevated temperature characteristics of the films will be discussed.

3:20 PM

Recent Developments in the Debate about the Formation Mechanism of Precipitate Plates: *Hubert I. Aaronson*[1]; B. C. Muddle[2]; J. F. Nie[2]; [1]Monash University, Dept. of Matls. Eng., Clayton Vic.3168 Australia; [2]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., Pittsburgh, PA 15213 USA

Although this debate developed ca. 1930, significant progress toward closure has been made only recently. Cahn (1994) carefully defined the conditions under which a lattice correspondence can be maintained during diffusional growth. Muddle, Nie and Hugo (1994) demonstrated that accurate fulfillment of the surface relief and crystallographic specifications of the phenomenological theory of martensite crystallography (PTMC) can obtain for plates differing continuously in composition from their matrix throughout the growth process in substitutional alloys. Recently, Christian (1997) accepted that an IPS surface relief and quantitative applicability of the PTMC cannot uniquely identify the growth mechanism as martensitic. Christian also asked why the PTMC is applicable to transformations not requiring a glissile interfacial structure. By comparison with results from model transformations, it is shown here that the PTMC also applies to sessile structures. Only invariant plane broad faces are required. Lateral migration of growth ledges can then simulate the geometry (though notthe mechanism) of martensitic growth.

General Abstract Sessions: Microstructure and Mechanical Properties II

Sponsored by: TMS

Program Organizers: James C. Earthman, University of California, Department of Chemical Engineering & Materials Science, Irvine, CA 92717-2535 USA; Richard Wright, Idaho National Engineering Laboratory, P.O. Box 1625 MS 2218, Idaho Falls, ID 83415-2218 USA

Wednesday PM — Room: Salon B&C
November 3, 1999 — Location: Omni Netherland Plaza Hotel

Session Chair: Yue Hui He, University of Tennessee, Dept. of Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA

2:00 PM

Three-Dimensional Visualization and Analysis of Microstructure: *George Spanos*[1]; Robert O. Rosenberg[1]; Milo V. Kral[2]; [1]Naval Research Laboratory, Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375-5000 USA; [2]University of Canterbury, Dept. of Mech. Eng., Private Bag 4800, Christchurch, New Zealand

Three-dimensional (3-D) visualization and/or virtual reality techniques are commonly used in many fields including (but not limited to) medical research and applications, computational chemistry, military applications, and the entertainment industry. The huge strides made in the last decade in computer hardware and software have made 3-D visualization an incredibly powerful research tool, yet the materials research community has not taken full advantage of these capabilities. In particular, despite the importance of understanding the true 3-D morphology, distribution, and interconnectivity of solid-state precipitates and grains in opaque bulk materials, microstructural characterization is usually accomplished by methods that enable only two-dimensional (2-D) observations. For instance, conventional optical and scanning electron microscopy (SEM) techniques are typically restricted to observations of 2-D planes of polish, while transmission electron microscopy (TEM) is limited to thin foils (approximately 100 nm) of material. This talk will review experimental and computer techniques that we have employed to reconstruct, visualize, and analyze the true 3-D nature of microstructures. This will include descriptions of: (1) serial sectioning and image acquisition methods, (2) image enhancement and quantification techniques, and (3) a variety of computer hardware systems and software algorithms, including a virtual reality "cave" environment.

2:20 PM

Nickel Titanium Shape Memory Alloy Reinforced Aluminum Composites: *Glen Andrew Porter*[1]; Peter Kevin Liaw[1]; Terry Tiegs[2]; [3]K. Wu; [1]University of Tennessee, Knoxville, Dept. of Mat. Sci., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metal & Cera. Div.; [3]Florida Int. University, Miami, FL 33714 USA

A shape memory alloy (NiTi) has been distributed throughout an aluminum matrix, using powder metallurgy processing, in the hopes of using the shape memory effect to achieve strengthening in the aluminum matrix. In order to accomplish this, the NiTi powder must first be reduced in size to enhance interfacial bonding and composite deformation characteristics. This is done through a tedious and time-consuming mechanical milling process. Since this process alters the phase of the NiTi, it must be followed with an annealing process to restore the shape memory effect. Also, since the shape memory effect is so strongly dependent on the composition of the NiTi, the powder must also be treated with a coating in order to resist diffusion of the matrix during pressing. An oxide coating has been investigated with moderate success. The process of hot pressing also lends difficulty because the time at elevated pressures and temperatures result in further diffusion which destroys the delicate shape memory effect. However, without a sufficient pressure and temperature in hot pressing, a good density cannot be achieved. Internal voids due to a poor density may become sites for crack initiation upon loading, thus weakening the material. The shape memory effect is activated by cold rolling the samples at-50 C. This will not only deform the matrix, but also each NiTi particle within the matrix. (The aluminum is softer than the NiTi austenite phase, but harder than the martensite phase.) Upon reheating to the austenite phase, the NiTi will return to its original shape, (within 8-9% of deformation) while embedded in the aluminum matrix which has a much lesser degree of strain. This will create residual, internal stresses around each particle which will strengthen the material in a similar fashion as thermal stresses strengthen a ceramic/metal matrix composite upon cooling from manufacture. It is theoretically possible to have a 30% increase in yield strength in the material, due to localized stresses in the matrix created by the shape memory effect of the embedded NiTi particles. The presence of the shape memory effect will be detected through digital scanning calorimetry (DSC). The presence of residual localized stresses in the matrix will be determined by X-ray diffraction (XRD) techniques. The mechanical characterization will be conducted with a new MTS high cycle fatigue test frame. The modes of crack growth as well as the cross-sectional microstructures of the composites will be examined by scanning electron microscopy (SEM). The SEM is equipped with an energy-dispersive spectrometer (EDS), which will allow us to distinguish the extent of diffusion between the particles and matrix.

2:40 PM

High Frequency Fatigue Crack Propagation in Ni Superalloys: Walter W. Milligan[1]; *Santo A. Padula*[1]; Amit Shyam[1]; David L. Davidson[2]; [1]Michigan Tech University, Metallu. Dept., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Southwest Research Institute, P.O. Box 28510, San Antonio, TX 78228 USA

Fatigue crack propagation experiments were performed at frequencies up to 1,000 Hz on polycrystalline and single crystalline nickel-base superalloys. Tests were conducted at room temperature and 650°C. Thresholds and FCP rates were determined as a function of frequency, temperature, load ratio, and microstructure. In situ experiments conducted at 2 kHz in an SEM loading stage will also be described.

3:00 PM

Load Transfer at Imperfect Interfaces-Dislocation-Like Model: *H. Y. (Sean) Yu*[1]; Y. N. Wei[2]; Fu-Pen Chiang[2]; [1]Naval Research Laboratory, Multifunctional Mats. Branch., Washington, DC 20375 USA; [2]State University of New York at Stony Brook, Dept. of Mech. Eng., Stony Brook, NY 11794 USA

A dislocation-like model that described the boundary conditions of an imperfect interface is verified experimentally. The boundary conditions to be modeled are that the radial and the tangential tractions are continuous across the interface and the displacements may be discontinuous from one solid to another. The discontinuity of displacement across the interface is assumed linearly proportional to the displacement at the interface of the constituent where the stress source is. The effect of the imperfect interface on the load transfer is studied by photoelastically measuring the elastic deformation in bimaterials due to an inclusion with pure dilatational eigenstrain. The maximum shear stress distributions measured from the isochromatic fringe patterns are in good agreement with the theoretical calculations. The results show that an imperfect interface could be viewed as a continuum entity with interface rigidities as proposed by the dislocation-like model.

3:20 PM Break

3:30 PM

Impact Damage and Residual Stress Effects on a Threshold-Based Model of Fatigue Behavior of Gamma TiAl: *Ryan M. Smith*[1]; Elen G. Koharian[1]; Trevor S. Harding[1]; J. Wayne Jones[1]; [1]University of Michigan, Mats. Sci. and Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Recently, gamma titanium aluminides have received significant attention as potential materials in aerospace applications such as turbine blades. Their high specific strength and stiffness and comparatively low density point to potential weight savings when compared to current materials. However, titanium aluminides exhibit relatively low ductility and limited fatigue crack growth resistance which results in reduced damage tolerance. This situation is exacerbated when foreign object damage eliminates the fatigue crack initiation lifetime. These conditions necessitate a threshold-based design approach to fatigue of g-TiAl. The current study explores impact damage geometry and the potential role of residual stress on a threshold-based model of fatigue behavior in two g-TiAl alloys: a duplex Ti-47.9Al-2.0Cr-1.9Nb (at%) alloy and a lamellar Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si (at%) alloy. Crack shapes are quantified through optical micrographs of the heat-tinted fracture surface and confirmed with SEM micrographs of the fracture surface morphology. Results show that surface crack measurements exaggerate the extent of internal damage and that cracks tend to form as corner cracks except at more severe impact levels. The extent of plastic deformation as the damage site is estimated by microhardness measurements around the impact site. The potential effects on fatigue lifetime of residual stresses arising from this deformation are compared through fatigue-testing of specimens with and without a post-impact annealing. The deformation

field is strongly geometry dependent and its fatigue effects are most significant in severely impacted specimens.

3:50 PM
IR Thermography: A New Technique to Study Cyclic Fatigue: Hsin Wang[1]; *Liang Jiang*[2]; Peter K. Liaw[2]; Charlie R. Brooks[2]; Dwaine L. Klarstrom[3]; Rodger Seely[3]; [1]Oak Ridge National Laboratory, High Temp. Mats. Lab., Oak Ridge, TN 37831 USA; [2]University of Tennessee Knoxville, Dept. of Mats. Sci. and Eng., 323 Dougherty Eng. Bldg., Knoxville, TN 37996 USA; [3]Haynes International Inc., Kokomo, IN 46904 USA

Infrared (IR) imaging has been used to monitor temperature changes during high-cycle fatigue tests of cobalt and nickel-based superalloys. Although a temperature gradient along the specimen always exists during the test, three distinct regions have been found in the temperature vs. the number of cycles plot. A temperature rise in the first region is associated with internal friction and defect formation in the materials. The second region is a thermal equilibrium stage between the sample and the testing system. In the third region, a rapid temperature rise starts several thousands cycles before the final rupture. Using the image analysis software, the crack causing final failure can be clearly identified as a hot spot/area. Thermoelastic effect was also observed using high-speed IR imaging. This technique is being used as a new tool to study fatigue behavior of metals and ceramics. This work is supported by the National Science Foundation under Grant No. DMI-9724476, Haynes International, Inc., and by the U.S. DOE, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the HTML User Program under contract DE-AC05-96OR22464, managed by Lockheed Martin Energy Research Corporation.

4:10 PM
The Evaluation of Effect of Yield Stress on Delayed Hydride Cracking in Zr-2.5Nb Alloys: *In Sup Kim*[1]; Je Yong Oh[1]; [1]Korea Advanced Institute of Science and Technology, Dept. Nuclear Eng., Yusong-gu, Kusong-dong 373-1, Taejon 305-701 South Korea

The delayed hydride cracking (DHC) in pressure tubes of a CANDU reactor is caused by diffusion of hydrogen atoms and reprecipitation of hydride under stress. However it is hard to quantitatively evaluate the factors that affect the DHC because they are related to each other. Yield stress, one of the major parameters, is varied by temperature, heat-treatment, irradiation, and texture. It was, therefore, difficult to compare DHC velocity at one temperature and yield stress to those in other conditions. In this paper, DHC velocity was normalized and the relation between yield stress and DHC velocity was represented in one master curve. The equation from the master curve could explain the difference between theoretical activation energy and experimental activation energy in DHC. The difference was found to result mainly from decreasing yield stress with temperature.

Hydrogen Effects on Materials Behavior: Crack Growth Susceptibility

Sponsored by: Structural Materials Division; ASM International: Materials, Science, Critical Technology Section; Corrosion and Environmental Effects Committee
Program Organizers: Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Russell H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; A. W. Thompson, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

Wednesday PM, November 3, 1999
Room: Salon H&I
Location: Omni Netherland Plaza Hotel

Session Chairs: Anthony W. Thompson, Lawrence Berkeley Laboratory, Berkeley, CA 94720 USA; Daniel Eliezer, Ben-Gurion University of the Negev, Beer-Sheva, Israel

2:00 PM
Effects of Mixed Mode I/III Loading on Environment-Induced Cracking: *Russell H. Jones*[1]; Huaxin Li[2]; J. P. Hirth[3]; [1]Pacific Northwest National Laboratory, Mats. Dept., P.O. Box 999, MSIN P8-15, Richland, WA 99352 USA; [2]Energizer Company, Cleveland, OH 44101 USA; [3]Washington State University, Mech. and Mats. Dept., Pullman, WA 99164-2920 USA

Many materials that exhibit ductile, tough behavior during conventional Mode I testing exhibit reduced toughness when loaded with combinations of Mode I and III. A minimum in the energy for crack initiation and stable crack growth has been demonstrated for ductile metals with mixed mode loading with crack planes at angles between 40 to 60 degrees relative to Mode I loading. Internal hydrogen further reduces these energies in a ferritic/martensitic steel with the effect being relatively equal in pure Mode I and mixed mode loading conditions. Hydrogen effects can rationalize a number of observations of Mode I crack growth rates being faster than Mode III rates for environment-induced cracking. Where hydrogen is clearly not a factor, such as brass in ammoniacal solutions, cracks loaded in Mode I and III grow at similar rates. This research was funded by the Division of Materials Sciences of the Offices of Basic Energy Sciences of the U.S. Department of Energy

2:20 PM
Mechanisms for Fatigue Crack Growth in 7075-T6 Aluminum Alloy: *Evan J. Dolley*[1]; Robert P. Wei[1]; [1]Lehigh University, Mech. Eng. and Mech., Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

Environmentally enhanced fatigue crack growth (or corrosion fatigue) in aluminum alloys has been attributed to hydrogen embrittlement (HE). In this paper, experimental evidence on corrosion fatigue crack growth (CFCG) in a 7075-T6 aluminum alloy, in dry air and 0.5M NaCl solutions (with different concentrations of dissolved oxygen), is presented in support of the HE mechanism. CFCG in the NaCl solutions may be separated into two regimes: a long-crack regime and a chemically short-crack regime. In the long-crack regime, CFCG rates are about 10X that in a dry environment. In the chemically short-crack regime, the growth rates are further increased by a factor of two and gradually decrease to the steady state growth rates of the long-crack regime; the additional increase is associated with dissolved oxygen in the solutions. Changes in the micromechanism for crack growth were observed between dry air to the NaCl solutions. Further changes were noted between the chemically short and long-crack regimes. Crack growth in dry air was predominantly transgranular, and etch-pit analyses showed that it occurred along the {100} crystallographic planes. Cracking in the NaCl solutions was also transgranular, but occurred along {100} and {110} planes. The fractional area of the {110} facets,

however, tended to be higher at the shorter crack lengths in the chemically short crack regime, and is correlated with the increased CFCG rates in the aerated solutions. The cracking response is discussed in terms of influence of oxygen on hydrogen production, and on the influence of hydrogen on the micromechanism and kinetics of crack growth. A superposition model is used to predict FCG rates in terms of the fractional area of the {110} fracture. This research is supported by the Air Force Office of Scientific Research under Grant F49620-1-98-0198 and by the Federal Aviation Administration under Grant 92-G-0006.

2:40 PM

Hydrogen Effects on the Fracture Toughness Properties of Austenitic Stainless Steel Weldments: *Michael J. Morgan*[1]; Scott L. West[1]; Michael H. Tosten[1]; Glenn K. Chapman[1]; [1]Westinghouse Savannah River Company, Savannhah River Tech. Ctr., Aiken, SC 29808-0001 USA

In this study, the effects of weld ferrite content on the fracture toughness properties of hydrogen-exposed Types 304L and 21-6-9 stainless steel weldments were investigated. Fracture toughness samples were cut from weldments that were made with specific ferrite contents ranging from 4 to 33% by using automatic gas tungsten arc welding and different filler wires. J-integral fracture toughness properties were measured at room temperature before and after exposure to hydrogen gas at 623 K and 69 MPa. For welds with up to 8% ferrite, the microstructure consisted of discontinuous skeletal ferrite present in a predominant austenite matrix. For welds containing more than 20% ferrite, the microstructure consisted of nearly continuous skeletal/acicular/cell-like ferrite present in a plate-like and globular austenite matrix. At low levels, ferrite had a beneficial effect on fracture toughness. Weldments containing discontinuous skeletal ferrite had fracture toughness properties two to three times higher than as-forged stainless steels. On the other hand, ferrite had a detrimental effect on the fracture toughness of hydrogen-exposed weldments. Weldments exposed to hydrogen had lower fracture toughness values and different fracture modes than unexposed weldments and similarly exposed as-forged steels. In weldments with discontinuous ferrite, hydrogen caused fractured along austenite-ferrite interfaces whereas in weldments with continuous ferrite, hydrogen caused fractured by cleavage completely through the ferrite phase. The information in this article was developed during the course of work under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

3:00 PM

Effects of Internal and External Hydrogen on Inconel 718: *R. J. Walter*[1]; J. D. Frandsen[1]; [1]Boeing Defense & Space Group, MS IB-33, 6633 Canoga Ave., P.O. Box 7922, Canoga Park, CA 91309-7922 USA

Internal hydrogen embrittlement (IHE) and hydrogen environment embrittlement (HEE) tensile tests were performed on Inconel 718. For the IHE tests, the specimens were precharged to ~90 ppm hydrogen by exposure to 34.5 MPa H2 at 650°C. The HEE tests were performed in 34.5 MPa H2. Parameters evaluated were test temperature and strain rate for smooth and notch specimen geometries. Embrittlement decreased significantly with increasing strain rate at ambient temperature for both IHE and HEE. The strain rate effect decreased with increasing temperatures. For IHE, the strain rate effect was negligible at 260°C, and for HEE the strain rate effect was negligible at 400°C. IHE and HEE were most severe at ambient temperature. Although embrittlement decreased with increasing temperature, both HEE and IHE were significant at 650°C. At low temperatures, IHE was more severe than HEE. With increasing temperature, the difference between IHE and HEE decreased. IHE and HEE were essentially the same at temperatures above 350°C. At 350°C, the equilibrium hydrogen concentration in Inconel 718 is about 50% lower than the hydrogen content of the precharged IHE specimens. Dislocation hydrogen sweeping of surface absorbed hydrogen was the likely transport mechanism for increasing the hydrogen concentration in the HEE tests sufficiently to produce the same degree of embrittlement as that of the higher hydrogen content IHE specimens. The main IHE fracture characteristic was formation of large, brittle flat facets, which decreased with increasing test temperature. The IHE fracture matrix surrounding the large facets ranged between brittle fine faceted to microvoid ductility depending upon strain rate, specimen geometry as well as temperature. The HEE fractures were characteristically fine featured, transgranular and brittle with a significant portion forming a “saw tooth” crystallographic pattern. Both IHE and HEE fractures were predominantly along the {111} slip and twin boundaries. With respect to embrittlement mechanism, it was postulated that dislocation hydrogen sweeping and hydrogen enhanced localized plasticity were active in HEE and IHE for concentrating hydrogen along {111} slip and twin planes. Final brittle failure occurred by hydrogen induced planer decohesion.

3:20 PM

Hydrogen Effects on IN-100: *D. B. Allen*[1]; D. P. DeLuca[1]; R. J. Zaehring[1]; [1]Pratt & Whitney, P.O. Box 109600, West Palm Beach, FL 33410-9600 USA

A recent investigation into the effects of hydrogen on the wrought powder Ni base superalloy IN-100 was conducted. Metallographic preparation of hydrogen-exposed specimens revealed that regions of high hydrogen concentration reacted differently to etchants. Hydrogen ingress into the material was modeled and compared to the penetration depths obtained by etching and the effects of hydrogen on microscopic fracture modes were studied. The micromechanics of fracture, hydrogen permeation and metallographic effects are discussed.

3:40 PM Break

3:50 PM

Unanticipated Mechanical Property Degradation of IN 100 in High Pressure Hydrogen and Hydrogen Rich Steam: *S. J. Gentz*[1]; J. D. Haynes[2]; J. W. Sheldon[2]; [1]NASA-Marshall Space Flight Center, MS: EH22, Bldg. 4612, Huntsville, AL 35812 USA; [2]United Technologies-Pratt Whitney, P.O. Box 109600, West Palm Beach, FL 33410-9600 USA

IN 100 (PWA-SP 1074), a powder metallurgy nickel base super alloy is used in the Space Shuttle Main Engine (SSME) High Pressure Fuel and Oxidizer Turbopumps (HPFTPs and HPOTPs). This material is exposed to a variety of severe thermal and environmental conditions throughout the start transient, main stage, and shut down cycles of the SSME. Previous experience for most materials indicated that one of the most life limiting exposures is to ambient temperature high pressure gaseous hydrogen. Recent developmental testing of the HPFTP revealed hydrogen assisted cracking in the Turbine Housing. Thermal and structural analyses, supported by instrumented hot fire units, suggested the cracking area of the housing was being exposed to 300 to 500 F high pressure hydrogen. A characterization program was initiated to increase the understanding of IN 100 in this temperature and environmental regime. This paper presents the findings of this material property characterization investigation and of the examination of a hydrogen barrier coating and an alternative thermal treatment to improve the hydrogen properties of IN 100. Data presented will show that hydrogen rich steam has a greater effect on properties than pure hydrogen and that the minimum strength properties occur at a temperature greater than ambient for IN 100.

4:10 PM

Behavior of Direct Fabricated 304L SS in Hydrogen Environments: *Joel A. Philliber*[1]; Brian P. Somerday[1]; John E. Smugeresky[1]; Michelle L. Griffith[2]; [1]Sandia National Laboratories, P.O. Box 969, M/S 9403, Livermore, CA 94551 USA; [2]Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185 USA

Direct fabrication of metals through the LENSÔ processing can enhance mechanical properties over those observed in conventionally processed materials. In 316 stainless steels, the yield stress can be doubled with no ductility loss. This behavior is attributed to the small grain/cell size (5 micron) and relatively low dislocation

densities that are inherent in LENSÔ processed materials. Fabricating 304L SS with the LENSÔ process to create a similar microstructure may result in an increased resistance to solute hydrogen. The small grain size should provide ample hydrogen trapping sites at grain boundaries while limiting the amount of hydrogen at void nucleating particles. Additionally, the relatively low dislocation density should allow moderate ductility to be retained. A preliminary study was conducted to evaluate the ability of LENSÔ processed 304L SS to resist hydrogen embrittlement. Increased yield strengths were obtained with the LENSÔ processed 304L SS over conventional wrought and annealed material. The ductility of the LENSÔ material was also greater than that of the conventional 304L. LENSÔ fabricated tensile bars were charged to saturation in hydrogen gas at 300°C and pressures up to 140 MPa. Tensile testing was done in air at room temperature. The resistance of this material to hydrogen embrittlement and the underlying mechanisms will be discussed. This work is supported by the U.S. Department of Energy under contract #DE-AC04-94L85000.

4:30 PM

Effect of Hydrogen on Microvoid Fracture of 304L Stainless Steel: *Brian P. Somerday*[1]; Mark F. Horstemeyer[1]; [1]Sandia National Laboratories, Mats. & Eng. Sci. Ctr., P.O. Box 969, Livermore, CA 94551 USA

Solute hydrogen degrades the ductility and fracture toughness of 304L stainless steel at 25°C. Despite the salient effect of hydrogen on fracture resistance, the fracture mode is not significantly altered. Fracture progresses by microvoid nucleation, growth, and coalescence independent of hydrogen content. Although hydrogen clearly accelerates microvoid fracture, it is uncertain how each stage of the process is affected. The objective of this work is to quantitatively characterize the effect of hydrogen on microvoid nucleation, growth, and coalescence in low-sulfur 304L stainless steel. Notched tensile specimens are charged to saturation in hydrogen gas (up to 140 MPa pressure) at 300 C then tested under a constant displacement rate at 25°C. The experiments are interrupted prior to final fracture at strains from 75% to 95% of the fracture strain. The specimens are sectioned longitudinally and polished to reveal the extent of microvoid damage in the notch section. The number density and area fraction of voids are measured as a function of strain. These data are compared to similar measurements of damage in uncharged specimens. The experimental results are compared to initial efforts to numerically model microvoid fracture using finite-element and embedded-atom methods. (This work is supported by the U.S. Dept. of Energy under contract #DE-AC04-94L85000.)

4:50 PM

Hydrogen Effects on Crack Growth at Elevated Temperatures in IN903: *N. R. Moody*[1]; M. I. Baskes[1]; S. L. Robinson[1]; M. W. Perra[1]; [1]Sandia National Laboratories, P. O. Box 969, Livermore, CA 94551-0969 USA

Austenitic superalloys are often used in hydrogen and hydrogen-producing environments because of their resistance to hydrogen effects. However, they are susceptible to crack growth even at elevated temperatures. We therefore employed hydrogen charged samples to determine slow crack growth susceptibility in the iron-based superalloy IN903 at temperatures ranging from 253 K to 373 K. The measured crack growth rates increased by more than two orders of magnitude as temperature increased from 253 to 298 K and then decreased with further increases in temperature. Nevertheless, fracture in all samples initiated by fracture of matrix carbides followed by microvoid formation at slip band intersections and failure of interconnecting slip band segments. Materials and mechanics approaches were then combined to model the fracture process and crack growth rates. Application of the model to the test results supports the observation that microvoid formation at slip band intersections is the critical event in the fracture process. It further shows that the interaction between crack tip stress fields and trap sites controls crack growth susceptibility. This interaction with additional trap sites at temperatures above 298 K leads to the marked decrease in crack growth rates and increase in threshold stress intensities. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

Processing and Properties of Structural Nanomaterials IV

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Powder Materials Committee, Alloy Phases Committee, Materials Processing & Manufacturing Division, ASM-MSCTS, Materials & Processing Committee

Program Organizers: Leon L. Shaw, University of Connecticut, Department of Metallurgy & Materials Engineering, Storrs, CT 06269-3136 USA; Lawrence T. Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; Carl C. Koch, North Carolina State University, Department of Materials Science & Engineering, Raleigh, NC 27695 USA

Wednesday PM — Room: Rookwood
November 3, 1999 — Location: Omni Netherland Plaza Hotel

Session Chairs: Paul G. McCormick, University of Western Australia, Special Res. Center for Adv. Min. and Mats. Proc., Nedlands, WA 6907 Australia; Ke Lu, Institute of Metal Research, State Key Lab for RSA, Shenyang, China

2:00 PM Invited

High Pressure/Low Temperature Sintering of Nanophase Ceramic Powders: *B. H. Kear*[1]; [1]Rutgers University, Ctr. for Nanomaterials Rsrch., Piscataway, NJ 08855-0909 USA

A hot pressing method for consolidation of nanophase ceramic powder compacts without inducing significant grain growth has been developed. The method involves the simultaneous application of high pressure (up to 8 GPa) at relatively low temperature (< 0.5 Tm). High compaction pressure causes particle deformation, such that the green density increases with pressure up to a maximum at ~8 GPa. Low sintering temperature mitigates grain growth during the consolidation process. Another favorable factor is the occurrence of a pressure-induced phase transformation (typically from a metastable structure to a more stable structure), accompanied by a significant reduction in free volume (>3 vol.%). Such a transformation assisted consolidation has been successfully applied to produce sintered oxide and non-oxide bulk nanocrystalline (<100 nm grain size) ceramics, starting with even finer-scale ceramic nanopowders. Under appropriate conditions, a sintered grain size can be realized that is actually smaller than the original powder particles size.

2:30 PM Invited

Processing of High Hardness-High Toughness Alumina Matrix Composites: *Rajiv S. Mishra*[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. and Mats. Sci., One Shields Ave., Davis, CA 95616 USA

Ceramic-matrix composites with nanocrystalline (grain size less than ~100 nm) matrix provide new opportunities, both scientific and technological. The experimental results on creep and toughness do not follow the existing theoretical framework. The knowledge gap and opportunities to develop nanocrystalline ceramic-matrix composites will be highlighted. We have been able to synthesize fully dense nanocrystalline alumina matrix composites with grain size as fine as 30-40 nm. The results show significantly higher hardness and toughness in nanocomposites. Specific examples of Al_2O_3-Diamond, Al_2O_3-ZrO_2 and Al_2O_3-Nb nanocomposites will be presented. The hardness values of fully dense nanocrystalline Al_2O_3-10 vol. % Nb composite range from

20-23 GPa. The indentation toughness is > 8 MPa $m^{1/2}$ for the specimen sintered at 900°C and 2 GPa for 60 minutes. The present results show an increase in fracture toughness by more than 250% while retaining the hardness as compared to microcrystalline single phase alumina.

3:00 PM Invited
Processing and Properties of Nanophase Non-Oxide Ceramics: *Robert Vaßen*[1]; *Detlev Stöver*[1]; [1]Forschungszentrum Jülich GmbH, IWV 1, Jülich 52425 Germany

The present paper describes recent activities in the development of non-oxide nanophase ceramics. Examples on the processing of TiN, TiB2, Si3N4, SiC and others will be given. Only a few of the described activities ended up with both high densities (i.e. >95% of the Theoretical Density (TD)) and grain sizes below 100 nm. A possible reason for these behavior of non-oxide ceramics is the contamination of the surface by oxygen. For SiC, it is shown in more detail that the oxide layer on the surface influences the densification behavior. It favors non-densifying sintering mechanisms and hence coarsening of the microstructure. A processing route was developed which effectively reduces the oxide layer. Subsequent Hot Isostatic Pressing (HIP) of these SiC samples led to densities above 97%TD and grain sizes below 100 nm. A further improvement of the grain size to density ratio is expected from an optimized de-agglomeration of the nanophase powders during wet chemical processing. Results will be presented. Finally, a short overview of the properties of nanophase non-oxide ceramics will be given, e.g. hardness, fracture toughness, thermal diffusivity, superplastic deformation. Due to the fact that the manufacture of ceramics with grain size below 100 nm turns out to be difficult also results of samples with slightly larger grain sizes are included.

3:30 PM Break

3:50 PM Invited
Viewing Nanoparticles Sinter: Combined Studies of Computer Simulation and Electron Microscopy: *Robert S. Averback*[1]; [1]University of Illinois, Dept. of Mats. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

By directly observing nanoparticles while they sinter, using a combination of molecular dynamics (MD) computer simulations and in situ electron microscopy (TEM), a rich variety of behaviors was found. Our MD simulations revealed that the capillary forces associated with nanoparticles in contact play an important role in the sintering behavior. For crystalline Cu particles, these forces cause mass transport by generating dislocations, while interfacial torques cause contacting grains to rotate into low energy configurations, even at 80 K. Rapid sintering in crystalline and amorphous CuTi nanoparticles was also observed, but in this case by viscous flow within the interface. We also found that soft particles soft landing on hard substrates undergo shear such that the first few atomic layers of the nanoparticle become instantaneously epitaxial upon contact. This behavior was confirmed by TEM observations. We also showed by TEM that hard nanoparticles soft landing on soft substrates burrow into the substrate. These various effects will be discussed in terms of the extremely high shear stresses developed in nanoparticles systems.

4:20 PM Invited
Critical Issues in Superplasticity of Nanocrystalline Materials: *Rajiv S. Mishra*[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. and Mats. Sci., One Shields Ave., Davis, CA 95616 USA

The possibility of enhanced superplastic properties in nanocrystalline materials has been discussed for a decade now. In the last few years, tensile superplasticity has been observed in a number of nanocrystalline materials. The emerging trends suggest that the superplastic behavior in nanocrystalline state is not a simple scaling of the microcrystalline superplasticity. In this brief review, we outline the critical issues related to superplasticity in nanocrystalline materials. Nanocrystalline materials exhibit superplasticity at significantly lower temperatures. But even at these relatively lower temperatures, some grain growth occurs. The onset of grain growth limits the temperature range over which the parametric dependencies for superplasticity can be determined for a fundamental understanding. The kinetics of superplastic deformation in nanocrystalline state is compared with that in the microcrystalline state and the predictions of theoretical models. On a normalized basis, the superplasticity kinetics starts to decrease below ~0.2-1.0 mm range, depending on the material. The influence of grain size on each step involved during slip-accommodated grain boundary sliding is discussed to explain this transition. The authors gratefully acknowledge the support from the National Science Foundation under grants NSF-DMR-9630881.

4:50 PM
Grain Growth in Ti-Aluminide (TiAl) Type of Advanced Alloys Processed by Mechanical Alloying and Hot Isostatic Pressing: *Sedat Özbilen*[1]; Mehmet Erdogan[1]; [1]Gazi University, Metallu. Edu. Dept., Teknikokullar, Ankara, Turkey

Grain growth in Ti-based alloys such as Ti-aluminides (TiAl) with or without B additions has been investigated. For this purpose, mechanically alloyed and hot isostatically pressed powder materials were annealed at different temperatures for different annealing times. TEM study of these samples were carried out for grain size determination. Kinetics of grain growth in TiAl alloy matrix with grain boundary movement inhibitors (i.e., B additions) were studied. The results were compared with those reported in the literature. Attention was paid to the stability of nano grain structure and the effect of grain boundary inhibitors to the shape and size change of the grains.

P/M: Current Research and Industrial Practices: Net Shape Powder Parts

Sponsored by: Materials Design and Manufacturing Division, Powder Materials Committee
Program Organizers: F. D. S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metals Engineering, Rapid City, SD 57701-3995 USA; Enrique V. Barrera, Rice University, Metal Engineering & Materials Science Department, Houston, TX 77251 USA; G. M. Janowski, University of Alabama, Department of Materials & Mechanical Engineering, Birmingham, AL 35294-4461 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM	Room: Caprice 2&3
November 3, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Animesh Bose, Powdermet, Unit A, Sun Valley, CA 91352 USA; Henry R. Piehler, Carnegie Mellon University, Mats. Sci. and Eng., Pittsburgh, PA USA; Francis S. Biancanielo, U.S. Department of Commerce, National Institute of Standards and Technology, USA

2:00 PM Keynote
Direct Laser Powder Deposition: *James W. Sears*[1]; [1]Lockheed Martin Corporation, Bldg. D2, Rm. 114, 1 River Rd., PO Box 1072, Schenectady, NY 12301-1072 USA

Recent developments in the field of Laser Cladding and Rapid Prototyping have led to direct manufacturing of net shape metal components by laser fusion of powder alloys. This process is known by names such as Selective Laser Sintering (SLS), Directed Light Fabrication (DLF), Laser Engineered Net Shaping (LENS), and Direct Metal Deposition (DMD), to name a few. However for this talk it shall be called Direct Laser Powder Deposition (DLPD). DLPD involves fusing metal alloy powders in the focal point of a laser (or lasers) that is (are) being controlled by Computer Aided

Design-Computer Aided Manufacturing (CAD-CAM) technology. DLPD has the capability to produce fully dense components with little need for subsequent processing. Research and development of DLPD is being conducted throughout the world. The list of facilities conducting work in this area continues to growth (over 25 identified in the United States alone). Developments in DLPD are currently being pursued through two different techniques, SLS and laser cladding. In SLS a layer of powder is rolled out for each laser pass. While in the laser cladding technique the powder is fed into the focal point of the laser where it is melted and then solidifies to a form shape. This talk will elaborate on the state of these developments.

2:35 PM Invited
Net Shape Powder Forming Using Magnepress™ DMC Technology: *Bhanu Chelluri*[1]; [1]IAP Research Inc., 2763 Culver Ave., Dayton, OH 45429 USA

Magnepress™ DMC is an innovative net shape powder pressing technology that uses magnetic pulse forces to achieve full density in several material systems. Since the compaction duration is less than 1 millisecond, important dynamic effects occur in the powders which, when combined with the full density, yield properties for high performance. In ferrous alloys, the resulting properties are high strength and ductility that are close to those of forged and wrought materials. Additionally special microstructures and grain sizes of the starting powders can be preserved after Dynamic Magnetic Compaction (DMC). In this presentation, an overview of the process, the properties of various compacted materials, the technology features such as size, shape and dimensional tolerances that can be achieved in finished parts will be described.

3:05 PM
Improving Toughness in MIM Parts: Joseph W. Newkirk[1]; *J. Alan Sago*[1]; [1]University of Missouri-Rolla, Dept. of Metallu. Eng., Rolla, MO 65409 USA

Metal injection molded parts have received wide acceptance in many fields due to their lower cost and good strength and hardness properties. However, many applications of MIM parts are limited by toughness considerations. Studies have shown that the toughness values of individual MIM parts can vary widely. The factors that determine both the toughness value and reproducibility is being studied. The effect of processing variables and microstructural features on toughness will be reported on in this presentation. Recommendations for both improving toughness and the reproducibility of toughness from part-to-part will be presented.

3:35 PM Break

3:50 PM
Recent Advances in Ferrous Powder Metallurgy: *Nikhilesh Chawla*[1]; Fred Semel[1]; Sydney Luk[1]; Sim Narasimhan[1]; [1]Hoeganaes Corporation, Rsrch. and Dev. Div., 1001 Taylors Ln., Cinnaminson, NJ 08077 USA

Ferrous powder metallurgy (P/M) has advanced significantly over the last several years. Near net shape, high strength, and cost effective P/M parts are being used in several automotive applications including transmission gears and connecting rods. This talk will address recent advances in the area of ferrous powder for the P/M industry. It will be shown that ferrous P/M encompasses a combination of physical metallurgy, in alloy development, as well as polymer and surface science, in developing binder treatment for powder blending technology. The development of a molybdenum prealloyed powder for increased density, as well as binder treated powders for better control of alloying additions and increased compaction rates will be presented. Emerging technologies and an outlook for P/M development in the 21st century will be discussed.

4:20 PM
Mechanical Properties of Powder Injection Molded Gas and Water Atomised 316L Stainless Steel Powder Mixtures: *S. Ozbilen*[1]; Murat Dalkylyc[1]; [1]Gazi Technical University, Metallu. Eng., Teknikokullar, Ankara Turkey

Gas and water atomised 316L stainless steel powders with different sizes and shapes were blended with different ratios. These blends were mixed with proprietry binder formula to prepare the designed feedstocks for molding. Molded feedstocks were then subjected to binder removal and sintering under controlled atmospheres to obtain maximum possible sintered densities. These were followed by pre-designed heat treatment schedules to get the optimum mechanical properties from the powder alloy system studied in the present work. Recent results will be presented and discussed.

4:50 PM
The P/M of 58Fe-31Mn-10Al-1C Alloy: *Shih-Chin Chang*[1]; Shih-Nan Yen[1]; Chun-Sien Lin[2]; [1]National Tsing-Hua University, Mats. Sci. and Eng., Hsinchu 30043 Taiwan; [2]Gloria Heavy Industrial Corporation, R & D, 35, Hsin Chung Rd., Hsin Ying Taiwan

The optimum process of making mechanical alloyed P/M 58Fe-31Mn-10Al-1C alloy was determined in this work. After 12 hr ball milling of the mixture of commercially available pure powder of elements, the intermetallic phases of FeAl, Fe3Al and Fe3AlC0.5 were found. After compaction under a pressure of 550 Mpa, sintering were carried out in different H2 + Ar atmospheres. A single austenitic structure with annealing twins was formed after one hour sintering. The highest sintered density and best mechanical properties of the specimens were obtained with the sintering atmosphere of 20 vol% hydrogen. The sintering density was 6.37 g/cm3 and the mechanical properties were: hardness of HRC 35, yield strength of 1122 Mpa and fracture strength of 1207 MPa.

Symposium to Honor Professor Julia R. Weertman: Nanocrystalline Materials II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37996-2200 USA; David Dunand, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Greg Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Wednesday PM	Room: Rosewood
November 3, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Jeffrey A. Eastman, Argonne National Laboratory, Mats. Sci. Div., Argonne, IL 60439 USA; Carl C. Koch, North Carolina State University, Raleigh, NC 27695 USA

2:00 PM Invited
Deformation and Fracture of Amorphous, Nanocrystalline, and Amorphous/Nanocrystalline Materials: *Carl C. Koch*[1]; [1]North Carolina State University, Mats. Sci. and Eng., Box 7907, Raleigh, NC 27695 USA

Julia Weertman and her co-workers have lead in both experimental studies and interpretation of the mechanical behavior of nanostructured materials. This paper will review her work, as well as that of others, which suggest what may be useful analogies in the mechanical behavior of amorphous and nanocrystalline materials. As early as 1983, Donovan and Stobbs observed similar deformation behavior between amorphous and nanocrystalline Pd-20 at.% Si. In both cases deformation proceeded by localized shear bands. The shear band deformation can result in significant macroscopic plasticity in constrained compression such as rolling, but almost no macroscopic plastic deformation in tension. This is a consequence of the essentially zero strain hardening such that the materials are per-

fectly plastic and the yield strength is equal to the fracture stress. This deformation behavior has been studied in metallic glasses and amorphous polymers and more recently in nanostructured materials. Speculation on the nature of the microscopic deformation mechanisms in both amorphous and nanostructured materials will be presented. In addition, the potentially useful combination of strength and ductility observed in some two-phase amorphous/nanocrystalline alloys will be discussed.

2:30 PM Invited

Consolidation and Characterization of Nanocrystalline Cu and Cu-Ni Powders Prepared by Mechanical Alloying and Milling: *Jose Gerardo Cabañas-Moreno*[1]; Alicia Rodríguez-Pulido[2]; Hector A. Calderon[1]; Koichi Tsuchiya[3]; Minoru Umemoto[3]; Julia R. Weertman[4]; [1]Instituto Politecnico Nacional, Dept. Ciencia de Materiales, UPALM Ed. #9, Apdo. Postal 75-373, Mexico, D. F. 07300 Mexico; [2]Instituto Tecnológico de Saltillo, Dept. Metal-Mecánica, B. Venustiano Carranza 2400, Apdo Postal 600, Saltillo, Coahuila 25280 Mexico; [3]Toyohashi University of Technology, Product. Sys. Eng., Tempaku-cho, Toyohashi, Aichi 441 Japan; [4]Northwestern University, Mats. Sci. and Eng., The Tech. Instit., Evanston, IL USA

Mechanical alloying (MA) and mechanical milling (MM) are processes which can be readily used to prepare sizable quantities of metallic materials having grain sizes in the range of 10-50 nm. However, materials produced by MA and MM are usually produced in form of powders in widely variable states of agglomeration and with particle sizes ranging from just below 1mm and up to several tens of micrometers, depending on the particular material as well as the milling equipment employed in their production. Evidently, in order to obtain a bulk product, the powders resulting from MA or MM require subsequent consolidation and thermal treatments which may change, among other things, their nanocrystalline nature. Because of the potential advantages to be gained in cost and speed/volume of production by using MA and MM in the preparation of nanocrystalline metals and alloys, we have explored the making of Cu and Cu-Ni alloyed powders by MM and MA, respectively. In order to limit the coarsening of their original nanocrystalline structure, these powders have been subsequently hot consolidated by means of a plasma-assisted sintering process, using maximum sintering temperatures of about 773K applied for 600 s. In collaboration with Prof. J. R. Weertman's group, the structure and properties of the metallic materials prepared in this way have been characterized. These results will be presented and compared with the ones derived from Prof. Weertman's pioneering work on nanocrystalline Cu produced by other processes. Work supported by a CONACYT-NSF Bilateral Project Grant.

3:00 PM

Grain-Size Dependent Mechanical and Thermal Properties of Nanostructured Yttria-Stabilized Zirconia Coatings: *Jeffrey A. Eastman*[1]; Guido Soyez[1]; Ronald J. DiMelfi[2]; Loren J. Thompson[1]; Jitendra P. Singh[3]; Karl L. Merkle[1]; [1]Argonne National Laboratory, Mats. Sci. Div., 9700 S. Cass Ave., Bldg. 212, Argonne, IL 60439 USA; [2]Argonne National Laboratory, Reactor Eng. Div., 9700 S. Cass Ave., Bldg. 208, Argonne, IL 60439 USA; [3]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Bldg. 212, Argonne, IL 60439 USA

The seminal work of Prof. J. R. Weertman and co-workers has contributed substantially to current understanding of the mechanical behavior of nanocrystalline materials (see [1] for a recent summary). Through their work, it has been demonstrated that normally ductile materials such as fcc metals exhibit increased strength and reduced ductility as grain size is decreased into the nanometer regime. In contrast, nanocrystalline ceramics show great potential for increased ductility compared to their coarse-grained counterparts. As described in [1], significant porosity in samples often prevents determination of the intrinsic grain-size dependent behavior of nanocrystalline materials. When prepared through traditional powder processing routes, fully dense nanocrystalline ceramics are particularly difficult to produce. In the current work, metal-organic chemical vapor deposition (MOCVD) has been used to produce nanocrystalline yttria-stabilized zirconia (YSZ) coatings with no apparent porosity. Processing parameters can be varied to produce controlled grain sizes as small as 10-15 nm or as large as epitaxial single crystals. Biaxial disk-bend tests on YSZ coatings deposited onto both metallic or ceramic substrates are being performed to determine if expected improvements in ductility and fracture resistance accompany grain size reduction. The thermal conductivity of these coatings is also being measured. Approximately a factor-of-two reduction in thermal conductivity is seen at ambient temperature for the smallest-grained coatings compared to the conductivity of coarse-grained or single crystal coatings. A combination of lower thermal conductivity and improved mechanical behavior would make nanostructured zirconia coatings excellent candidates for future applications as thermal barriers. Measurements of the thermal and mechanical behavior of commercial YSZ thermal barrier coatings using the same techniques will also be described and discussed. This work is supported by the U. S. Department of Energy, BES-DMS, under Contract W-31-109-Eng-38 and by a grant from Argonne's Coordinating Council for Science and Technology. [1] J.R. Weertman, D. Farkas, K. Hemker, H. Kung, M. Mayo, R. Mitra, and H. Van Swygenhoven, MRS Bulletin, February, 1999, pp. 44-50.

3:15 PM

An Overview and Prospectus of the Mechanical Behavior of Nanostructured Materials: *Terry C. Lowe*[1]; Yuntian T. Zhu[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., MST-DO, MS G754, Los Alamos, NM 87545 USA

Quoting Neil Lane, Science Advisor to the President of the United States "If I were asked for an area of science and engineering that will most likely produce the breakthroughs of tomorrow, I would point to nanoscale science and engineering." Recent efforts to understand the mechanical behavior of nanostructured materials have helped build the path to fulfill this projection. In this overview we summarize steps along the way to our current understanding, including key contributions by Julia Weertman. Then we identify some emerging challenges and provide a prospectus on the directions for future research, addressing both experimental and computational science. Finally, we examine the connection between the evolution of nanostructured materials and the integration of the physical and biological sciences.

3:30 PM BREAK

3:50 PM

Microsample Tensile Testing of Nanocrystalline Metals and Single-Crystalline: *Marc Zupan*[1]; M. Legros[2]; Brian Elliott[3]; K. J. Hemker[1]; [1]John Hopkins University, Dept. of Mech. Eng., Baltimore, MD 21218 USA; [2]Ecole des Mines/Parc de Saurupt, Lab de Physique des Materiaux, Nancy 54042 France; [3]McKinsey & Company, 600 Campus Dr., Florham Park, NJ 07932 USA

The development of a "microsample" tensile testing machine has greatly facilitated the mechanical testing of small specimens; microsamples 25-500 microns thick and 200-300 microns wide are now routinely tested in this machine. The non-contact optical interferometric strain/displacement gage (ISDG) used to measure strain in the gage of the microsamples possesses aresolution of 5 microstrain, and resistive heating can be used to heat the microsamples to temperatures in excess of 1300K. Microsample tensile tests of nanocrystalline pure metals have provided evidence of tensile strengths in excess of 1GPa, limited ductility, and measurements of elastic moduli that are consistent with bulk values. The results of these experiments will be discussed in terms of the underlying nanostructure and an apparent lack of dislocation activity. By contrast, single-crystalline microsamples of Ti-55.5at%Al have been tested in tension at temperatures ranging from 873K to 1173K. The results of these tensile experiments have been compared with the compressive strength of this material, and a tension-compression (T/C) asymmetry has been observed for three

Daily Personal Schedule

Thursday, November 4, 1999

Time	Session Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
10:30 am				
11:00 am				
11:30 am				
12:00 pm				
12:30 pm				
1:00 pm				
1:30 pm				
2:00 pm				
2:30 pm				
3:00 pm				
3:30 pm				
4:00 pm				
4:30 pm				
5:00 pm				
5:30 pm				
6:00 pm				
6:30 pm				

different orientations. This T/C asymmetry will be related to the dissociation, motion, and cross-slip locking of superdislocations in the ordered L10 structure.

4:20 PM
A Comparison of the Deformation Bahavior of Ultra Fine Grained Copper Produced by Particulate Processing and Bulk Deformation Processing: *Shankar M. L. Sastry*[1]; Suryanarayanan R. Iyer[2]; Virgil Provenzano[3]; Lynn Kurihara[3]; [1]Washington University, Mech. Eng., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]Applied Materials Inc., Santa Clara, CA 95050 USA; [3]Naval Research Laboratory, Code 6320, 4555 Overlook Ave., Washington, DC 20375 USA

Mechanical properties of nanocrystalline materials synthesized by a variety of methods and consolidated by different processing routes have been attributed to nanocrystalline structure without due consideration given to the impurities present, nanoporosity of the consolidated products, and the nature of inter particle and inter agglomerate bonding. Contamination is a serious issue in processing of nanocrystalline materials; yet control and minimization of impurities such as carbon, oxygen, nitrogen, and hydrogen has not been adequately addressed in most of the investigations. Adsorbed impurities, oxide films on particles surfaces, and agglomeration of fine particles invariably result in poor inter-particle and inter-agglomerate bonding although the consolidated products may exhibit close to theoretical densities. Evaluation of properties of such nanocrystalline materials masks the 'intrinsic' effects of nanocrystalline and nanograin structure and leads to erroneous conclusions regarding nanocrystalline material behavior. With the objective of determining the intrinsic and extrinsic effects on the mechanical properties, ultra fine grained copper samples were prepared by two methods: (i) consolidation of nanocrystalline particles produced by solution phase synthesis and POLYOL process, and (ii) severe plastic deformation (SPD) by equal channel angular extrusion of bulk copper. The mechanical properties of the alloy samples were determined by microhardness measurements, compression testing, and three-point bend testing. Whereas the particulate processed copper exhibited high hardness values, the specimens failed without exhibiting any plastic deformation in 3-point bend tests. The ECAE copper exhibited a good combination of high hardness and room temperature ductility. The results will be discussed in terms of the intrinsic and extrinsic factors governing the deformation of ultra fine grained copper.

4:35 PM
Deadhesion Properties of Nanocrystalline Films by Nanoindentation: Alex A. Volinsky[1]; *William W. Gerberich*[1]; Natalia I. Tymiak[1]; [1]University of Minnesota, CEMS, 421 Washington Ave. S.E., Minneapolis, MN 55455 USA

To evaluate adhesion of nanocrystalline films two requirements are knowledge of the film's constitutive properties and reliable technique for measuring interfacial delamination. Both of these can be provided by nanoindentation techniques. Atomic force microscopy identification of grain sizes in the range of 40 to 200 nm for film (thickness ranging from 40 nm to 3 microns) have been determined for sputter-deposited Cu films on SiO2/Si. From a range of nanoindentation experiments, both yield strength and modulus have been extracted for use in strain energy density calculations. This has been used with dislocation emission arguments to estimate the plastic energy dissipation in films thicker than 100 nm. For those thinner, a true surface energy is expected. Measurements in nanocrystalline Cu thin films deposited on SiO2/Si wafers confirm this. The measurement technique also consists of nanoindentation-induced microcracks into a W superlayer which triggers film delamination. On the driving force side, superlayer micromechanics measures values of 0.6 to 100 J/m^2, consistent with resistance side estimates from plastic energy dissipation. As such, the ratcheting effect of fracture toughness dependence, yp[ys] on the true surface energy, ys, is estimated.

4:50 PM
Characterization of Ultrafine Particles Produced by DC Plasma Jet Method: *Koichi Tsuchiya*[1]; Y. Todaka[1]; M. Umemoto[1]; [1]Toyohashi University of Technology, Dept. of Product. Sys. Eng., Toyohashi, Aichi 441-8580 Japan

Ultra-fine particles (UFPs) of various metals, oxides and nitrides have been drawing much attention since they often exhibit anomalous physical and chemical properties which can not be seen in bulk materials. In the present paper structure and properties of UFPs of various metals produced by DC plasma jet method will be presented with a special emphasis on Fe-Cu binary alloy. In this method, elemental powders of pure Fe and Cu were fed directly into Ar arc plasma. They were instantaneously vaporized by extremely high temperature of plasma and gaseous atoms form atomic clusters which grow into UFPs. Analytical high resolution electron microscopy revealed that the obtained UFPs are solid solution with a wide range of composition. Magnetic properties of these UFPs will be also reported.

Computational Materials Science at the Microstructural Scale: Microstructural Evolution in the Solid State

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Computational Materials Science & Engineering, Phase Transformations Committee
Program Organizers: Elizabeth Holm, Sandia National Labs, P.O. Box 5800, Albuquerque, NM 87185-0304 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark A. Miodownik, Sandia National Laboratories, Materials and Process Modeling, Albuquerque, NM 87185-1411 USA; J. P. Simmons, AFRL-MLLM, Dayton, OH 45433 USA; Prof. Peter W. Voorhees, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208

Thursday AM
November 4, 1999
Room: Caprice 1&4
Location: Omni Netherland Plaza Hotel

Session Chair: Jeff P. Simmons, AFRL, MLLM, Dayton, OH 45433 USA

8:30 AM Invited
Simulation of Recrystallization By Combining a Probabilistic Cellular Automaton with a Crystal Plasticity Finite Element Model: *Dierk R. Raabe*[1]; Richard C. Becker[2]; [1]Max-Planck-Institut fuer Eisenforschung, Max-Planck-Str. 1, Duesseldorf 40237 Germany; [2]Alcoa, Tech. Ctr., Alcoa Center, PA 15069 USA

We present partially discrete simulations of the formation of texture and microstructure in heterogeneously deformed aluminum during static primary recrystallization. Discrete predictions are of interest because they allow one to consider local grain boundary characteristics (mobility, energy) and local driving forces. The simulations are based on applying a probabilistic cellular automaton to spatially discrete deformation data obtained by a viscoplastic crystal plasticity finite element simulation. We will discuss the simulation methods, the mapping of the finite element interpolation points on the regular cellular automaton lattice, the used nucleation models, and a number of metallurgical results such as the textures, microstructures, and kinetics. The obtained results show that deviations from the classical Avrami behavior can be discussed in

terms of grain clustering and partial recovery of grains which have low driving forces, insufficient misorientations with respect to the growing nuclei, or insufficient nucleation rates.

9:10 AM

Modeling Dislocation Cell Structure Evolution: *Mark A. Miodownik*[1]; David J. Srolovitz[2]; Peter Smereka[2]; Elizabeth A. Holm[1]; [1]Sandia National Laboratories, Albuquerque, NM 87185-1411 USA; [2]University of Michigan, Ann Arbor, MI 48109 USA

Plastic flow in metals occurs by a dislocation mechanism in which strain is accommodated by a dislocation flux. This results in tangled networks of dislocations which self-organize into stable cell structures. These structures have a number of remarkable properties not least of which is that they exhibit scaling. We examine the idea that the evolution of these dislocation cells is dominated by random fluctuations; that as a result of plasticity there is a great deal of noise, and pattern formation arises from this complexity. We construct a simple model of the evolution of cell orientations. Instead of focusing on the fluctuations of the boundaries by considering dislocation mechanisms, we consider the fluctuations in the orientations of the cells. This reposes the problem in terms of a random walk in Orientation space, a diffusion problem. If all the orientations initially start with the same orientation, then there is a unit concentration of orientations at one point in Orientation Space. As a result of the fluctuations imposed by the strain state, these orientation diffuse out into Orientation Space. This is a direct analogue of the thin film problem in metallurgy. We quantitatively compare the results with the experiment data. This work was performed in part at Sandia National Laboratories under United States Department of Energy contract DE-AC04-94AL85000.

9:30 AM

Phase-Field Modeling of the Precipitation Process During a Coherent Hexagonal to Orthorhombic Transformation in Ti-Al-Nb System: *Youhai Wen*[1]; Yunzhi Wang[2]; Long-Qing Chen[1]; [1]Penn State University, Mats. Sci. Eng., 119 Steidle Bldg., University Park, PA 16802 USA; [2]Ohio State University, Mats. Sci. Eng., Columbus, OH 43210 USA

A phase-field model is utilized to simulate the precipitation process during a coherent hexagonal to orthorhombic transformation in Ti-Al-Nb system. Two alloys with different Nb content are employed to study its influence on the microstructure. In the high Nb alloy, the morphology of the precipitates is mainly rectangle/square and their mutual arrangement manifests some regular patterns. In the low Nb alloy, the dominant morphology for the precipitate is long band and their mutual arrangement also leads to some unique patterns. We have also investigated the spontaneous precipitation process where the precipitation can actually proceed without invoking any compositional separation. Our simulation demonstrate a tweed microstructure at the very beginning of the precipitation process and the final microstructure contains all those particular patterns such as star pattern, swastika pattern, fan pattern, etc., which is typical of a diffusion less structural transformation during a coherent hexagonal to orthorhombic transformation. Our simulations manifest that these regular patterns especially the star pattern can hardly appear as long as the precipitation process plays a major role during the microstructure evolution.

9:50 AM

Morphological Evolution and Coarsening Kinetics of Coherent Precipitates Using Diffuse-Interface Phase Field Model: *Venugopalan Vaithyanathan*[1]; Long Qing Chen[1]; [1]Pennsylvania State University, Dept. of Mats. Sci. and Eng., University Park, PA 16802 USA

The morphological evolution of coherent precipitates are strongly influenced by the elastic strain energy generated due to their lattice mismatch with the matrix. It is still unclear whether the conventional Ostwald ripening law is still applicable for the coarsening of these precipitates. This study focuses on establishing the coarsening kinetics of coherent precipitates in the presence of strain energy, in a matrix with negative elastic anisotropy, using diffuse-interface phase field model. A precipitate volume fraction of 20% is chosen for the study and the simulation is carried out in both two and three dimensions. The elastic anisotropy results in a cuboidal (rectangular in 2D) precipitate morphology due to preferential growth along <100> or elastically soft directions and, also precipitate alignment in these directions. The average particle size is extracted by two methods, (i) half the edge length and (ii) equivalent radius of circle with same area, as a function of time from which coarsening exponent is calculated. Particle size distributions are obtained to verify scaling behavior at late stages. Also variation in aspect ratio (ratio of longest edge length of cuboid to the shortest edge length) of precipitates is obtained as a function of time. The kinetics from the two dimensional and three dimensional simulation are compared with each other and also with the experimental results of γ' coarsening in Ni-Al binary alloys.

10:10 AM

OPEN

10:30 AM Break

10:40 AM

Phase Field Modeling of Simultaneous Nucleation and Growth During Precipitation: *Chen Shen*[1]; Yunzhi Wang[1]; Jeff P. Simmons[2]; [1]Ohio State University, Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, Dayton, OH 45433 USA

Significant cost savings are realized in alloy processing by using computer models to model alloy processes, thereby reducing the amount of experimental effort necessary. While the Phase Field model has been successful in simulating realistic microstructures and their evolution under conditions of high volume fraction, elastically interacting particles, and involving ordered product phases, which make it very attractive for studying Ni-based superalloys, its current applications are limited to modeling isothermal processes under the conditions of site saturation for nucleation. Application to a real process will require that it accounts for varying processing conditions under which nucleation, growth and coarsening occur simultaneously at all stages of the microstructural evolution. This work proposes to adapt the Phase Field model to handle simultaneously nucleation, growth and coarsening during precipitation of the L12 ordered particles in Ni-Al. A stochastic process of nucleation is incorporated in the model where nuclei of critical configuration are added continuously according the nucleation rate obtained either from experiment data or from the classical nucleation theory. The effects of the interplay among the chemical driving force, reduction in interfacial energy and relaxation of elastic strain energy on the kinetics and precipitate morphology are investigated.

11:00 AM

Modeling Grain Boundary Migration in Thin Films: *David P. Field*[1]; [1]TexSEM Laboratories Inc., 392E 12300S Ste. H, Draper, UT 84020 USA

Structure evolution in thin Al and Cu films is an important consideration in the fabrication of integrated circuits. Electromigration and stress voiding are reliability concerns which have long been known to depend upon the specifics of the metal microstructure. Experiments have been performed in-situ by orientation imaging in the SEM to observe the grain growth process in narrow lines. From these experiments, it has been possible to develop some simple criterion, based upon grain boundary character, for boundary migration. These criterion have been included in a simple Monte-Carlo type model and the results have been compared with experiment and with models which do not include criteria based upon grain boundary character. It is shown that the models which include these criteria offer an improved estimation of structure evolution as compared with experiments.

11:20 AM
Microstructure Dependence of Diffusional Transport Properties: *Jingzhi Zhu*[1]; [1]Penn State University, Dept. of Mats. Sci. and Eng., 119 Steidle Bldg., University Park, PA 16803 USA
We developed a simple method to simulate the temporal diffusional mass or heat transport process through microstructures with arbitrary complexity. We use a field variable to describe a microstructure, an idea similar to the diffuse-interface phase-field approach. The mass or heat diffusion through a given microstructure is modeled by a diffusion equation with a variable coefficient, which can be efficiently solved by an accurate semi-implicit spectral method. The effective diffusion coefficient for any given microstructure can then be extracted either from the temporal evolution of a diffusion profile through the microstructure or from the steay state diffusion. Examples of microstructures to be discussed include single-phase grain structures with significantly different grain boundary and bulk diffusion coefficients, and two-phase microstructures with different diffusion coefficient in each phase. For latter case, the effect of volume fraction and microstructure connectivity on the effective diffusion coefficient will be examined. Results will be compared with existing analytical theories and computer simulations.

11:40 AM
Vacancy Generation By the Motion of Jogged Screw Dislocations: *W. J. Liu*[1]; E. Essadiqi[1]; [1]CANMET, MTL, 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

Vacancies in materials, both thermal and mechanically generated, may play important roles in microstructural evolution. In the present paper, the vacancy generation during deformation is related to the non-uniform distribution of jogs associated with moving screw dislocations. A mathematical model for calculating the vacancy generation rate will be presented. The importance of the model in the development of other microstructural evolution models will also be discussed.

12:00 PM
Diffusion-Controlled Growth Rates of a Compound Layer Between Parallel Thin Films: Huifang Zhang[1]; *Harris Wong*[1]; [1]Louisiana State University, Mech. Eng. Dept., Baton Rouge, LA 70803 USA

Diffusion controlled growth of a compound phase A_nB ($n \geq 1$) between two parallel thin films of material A and B occurs during electronic materials processing and during synthesis of high-temperature materials using multilayer films. Previous models of the growth rates do not solve the diffusion equation, and thus do not utilize fully the predictive capability. In this talk, we describe a self-similar solution of the diffusion equation with the nonlinear Kirkendall effect included. The nonlinear partial differential equation is converted into a nonlinear ordinary differential equation with a free boundary and solved by a shooting method. Due to similarity, the solution holds for a range of length scales from nano to micrometers. It is found that the growth rates of the interfaces bounding the compound phase are completely determined once the equilibrium concentrations at the interfaces and the intrinsic diffusivities of A and B in A_nB are specified. An asymptotic analytic solution valid for small differences in the equilibrium interfacial concentrations is determined and agrees with the numerical results in the appropriate limit. Implications of this complete solution will be discussed.

Processing and Properties of Structural Nanomaterials V

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Powder Materials Committee, Alloy Phases Committee, Materials Processing & Manufacturing Division, ASM-MSCTS, Materials & Processing Committee
Program Organizers: Leon L. Shaw, University of Connecticut, Department of Metallurgy & Materials Engineering, Storrs, CT 06269-3136 USA; Lawrence T. Kabacoff, Office of Naval Research, Arlington, VA 22217 USA; Carl C. Koch, North Carolina State University, Department of Materials Science & Engineering, Raleigh, NC 27695 USA

Thursday AM
November 4, 1999
Room: Rookwood
Location: Omni Netherland Plaza Hotel

Session Chairs: B. H. Kear, Rutgers University, Center for Nanomaterials Research, Piscataway, NJ 08855 USA; Fereshtch Ebrahimi, University of Florida, Dept. of Mats. Sci. and Eng., Gainesville, FL USA

8:30 AM Invited
Thermal Stability of Nanocrystalline Copper: Grain Growth Versus Strain Release: L. Lu[1]; L. B. Wang[1]; *K. Lu*[1]; B. Z. Ding[1]; [1]Institute of Metal Research, CAS, State Key Lab. for RSA, 72 Wenhua Rd., Shenyang 110015 ROC

Nanocrystalline (nc) materials are thermodynamically metastable and under thermal activation, they will transfer to more stable state by releasing the excess energy stored in the numerous grain boundaries as well as the nanocrystallites. Grain growth and strain release are normally observed processes responsible for thermal annealing. In order to understand the underlying mechanism for the two processes, thermal stability of nc Cu samples with different amounts of microstrain but the same grain size was investigated. The nc Cu sample with a high purity and a high density was made by means of electrodeposition, in which the microstrain is negligible. Cold-rolling of the as-deposited nc Cu was used to introduce microstrain in the nc samples of which the grain size keeps unchanged. Measurement results shown that the grain growth kinetics and strain release process are sensitive to the level of microstrain in the nc samples. With an increase of the microstrain, the grain growth shifts to higher temperatures, while the strain release temperature is depressed. The activation energies for the grain growth and strain release processes were determined and their correlation was analyzed.

9:00 AM Invited
Nanostructured Materials in Multicomponent Alloy Systems: *Jürgen H. Eckert*[1]; [1]IFW Dresden, Institute für Metallische Werkstoffe, P.O. Box 270016, Dresden 01171 Germany

The discovery of ultra-high strength, good ductility and excellent wear resistance for nanophase multicomponent alloys prepared by solid state processing utilizing mechanical alloying techniques with or without subsequent annealing or by devitrification of easy glass forming alloys has stimulated considerable interest in these materials with very uniform microstructure and interesting properties. This contribution reports on the formation and the properties of mechanically alloyed, rapidly quenched and slowly cooled Zr-, Al, and Mg-based multicomponent alloys exhibiting amorphous/nanophase or nano-quasicrystalline microstructures. In addition, results for amorphous alloys containing insoluble nanoscale metallic particles or oxides for mechanical strengthening will be presented. Various experimental methods such as x-ray diffraction, transmission electron microscopy, thermal analysis, viscosity measurements and mechanical testing were applied to investigate the

microstructure and the properties of the different alloys. Partial devitrification of initially fully amorphous samples results in bulk ultra-fine quasicrystalline or crystalline precipitates with high stability against grain growth. This will be discussed with respect to the thermodynamics and kinetics of nucleation and growth. Alternatively, nanoscale composite materials were directly prepared by mechanical alloying and subsequent powder consolidation. Mechanical properties for different strain rates and temperatures will be presented and potential applications of these new materials will be critically assessed.

9:30 AM
Glass Formation and Primary Nanocrystallization in Al-Base Metallic Glasses: *Robert I. Wu*[1]; Gerhard Wilde[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Mats. Sci. and Eng., 1509 Univ. Ave., Madison, WI 53706 USA

Aluminum-rich metallic glasses containing transition and rare earth elements have been found to yield finely mixed microstructures of Al nanocrystals in an amorphous matrix and exhibit enhanced fracture strength with several percent strain. Upon primary crystallization of the melt spun ribbons, a novel microstructure comprised of a high particle density ($> 10^{20}$ m^{-3}) of Al nanocrystals (20nm) in an amorphous matrix develops and offers exceptional strength (1200MPa) and high temperature stability (260°C). Numerical modeling based upon the size distribution of the Al nanocrystals after isothermal annealing is applied to study the nucleation kinetics in the metallic glasses. In addition to the kinetic study of the primary nanocrystallization, the glass transition temperature (T_g) has been assessed in Al-7Y-5Fe and Al-8Sm alloys. In the usual measurement, the thermal response of the primary crystallization often obscures the observation of the signal corresponds to the glass transition during thermal analysis. As a result, T_g is often assumed to be near the onset of the primary crystallization reaction (T^{Al}_x). However, it has been demonstrated by modulated calorimetry that this assumption does not apply to the metallic glasses under study. The thermal stabilization of the microstructure by the occurrence of diffusion field impingement allows for the observation of the glass transition of the remaining amorphous phase in the matrix by the modulated DSC. The reliable assessment of the glass transition temperature provides not only a fundamental basis for kinetic analysis, but also an important parameter in designing suitable annealing treatments that allow for the development of desired microstructures to yield optimized properties. The support of the ARO (DAAG55-97-1-0261) and Humboldt Foundation (GW, V-3-FLF-1052606) are gratefully acknowledged.

9:55 AM Break

10:15 AM Invited
Chemical and Structural Effects Associated with Shear Band Formation and Fracture in Al-Based Metallic Glass: *G. J. Shiflet*[1]; [1]University of Virginia, Dept. Mats. Sci. and Eng., Charlottesville, VA 22903 USA

This talk will focus on the structural and chemical changes associated with deformation in Al-based metallic glass. We have previously shown that nanocrystals form within 10nm shear bands created during bending. Connected to nanocrystal formation is diffusion of the transition metal and rear earth element. This suggests that heat generation associated with the formation of the shear band is primarily responsible with the growth of the nanocrystal. Results will be presented on the size and density of nanocrystals and the chemical changes within the shear band. Observations of the immediate fracture surface resulting from tensile tests will also be given and compared to the interior of shear bands and nanocrystals formed during isothermal heat treatment. Work supported by AFOSR.

10:45 AM Invited
Laser Processing of Nanocrystalline Thin Films and Composites: *Jay Narayan*[1]; [1]North Carolina State University, Mats. Sci. and Eng., 2142 Burlington, Campus Box 7916, Raleigh, NC 27695-7916 USA

This talk reviews our work on nanocrystalline thin films and composites using laser deposition methods. Special emphasis is placed on Ge/AlN composites where quantum confinement in Ge nanocrystals leads to a blueshift in luminescence which increases with decreasing size of Ge nanocrystals. We also summarize our work on Cu and ZrO2 nanocrystalline thin films and characteristics of deformation of these films under nanoindentation plastic deformation. Using pulsed laser deposition, we are able to control crystallite size by changing the laser deposition and substrate parameters. This technique lends itself to processing of thin film composites and manipulation of properties with atomic-level controls.

11:15 AM
Magnetic Nanocrystalline Films: *Vladimir Grigor'evich Shadrow*[1]; Anatolyi Vasil'evich Boltushkin[1]; Lyudmila Vasil'evna, Nemtsevich[1]; [1]Institute of Solid State Physics, Acad. Sci. of Belarus, P. Brovki, 17, Minsk 220072 Belarus

Growth processes, structure and properties of hard magnetic electrodeposited Co based films and Co containing alumite films have been investigated by means of EM,XRD,AFM and AGFM. A mechanism of nanocrystalline structure formation in Co based electrodeposited films is proposed, which accounts for the structure changing observed as well as magnetic properties changing. The regularities of content modulated magnetic nanophase particles in the pores during pulse deposition and mechanism of supersaturated CoCu solid solutions formation are determined. Intergranular magnetic interaction and magnetization reversal properties in the above structures are investigated through remanence and delta M curves measurements and angular variations of coercivity.

P/M: Current Research and Industrial Practices: Structure/Properties/Processing Relationships

Sponsored by: Materials Design and Manufacturing Division, Powder Materials Committee
Program Organizers: F. D. S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metal Engineering, Rapid City, SD 57701-3995 USA; Enrique V. Barrera, Rice University, Metal Engineering & Materials Science Department, Houston, TX 77251 USA; G. M. Janowski, University of Alabama, Department of Materials & Mechanical Engineering, Birmingham, AL 35294-4461 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Thursday AM	Room: Caprice 2&3
November 4, 1999	Location: Omni Netherland Plaza Hotel

Session Chairs: Gregg M. Janowski, University of Alabama at Birmingham, Mats. and Mech. Eng., Birmingham, AL 35294-4461 USA; Sheeri R. Bingert, Los Alamos National Laboratory, MS G770, Los Alamos, NM USA

8:30 AM Keynote
Finite Plastic Flow of Granular Materials: *Sia Nemat-Nasser*[1]; [1]University of California at San Diego, Dept. of Appl. Mech. and Eng. Sci., Ctr. of Excellence for Adv. Mats., La Jolla, CA 92093-0416 USA

Fundamentals of finite-deformation elastoplastic flow of densely packed granular materials which carry the applied loads through contact friction, are discussed, integrating the material behavior at three distinct length scales, i.e., the micro-scale, the meso-scale,

and the macro-scale. Considering the contact forces, contact normals, and branches (which are vectors connecting the centroids of two adjacent contacting granules), at the micro-scale, expressions for the overall stress tensor, and the tractions transmitted across interior macroscopic planes, are developed. Macroscopic parameters which characterize the microstructure and its evolution in the course of deformation, are identified in terms of fabric tensors and the distribution density functions of unit branch vectors and contact normals, leading to explicit relations between the stress and fabric tensors. At the meso-scale, the deformation is assumed to consist of dilatant simple shearing over several interacting sliding planes. Resistance to such simple shearing is provided by friction due to rolling and sliding at the micro-scale, and restructuring through redistribution of the contact normals, i.e., the change in the fabric. An explicit expression is obtained for the critical value of the resistive shear stress, by integrating the corresponding contact forces over a typical sliding plane. Finally, physically-based continuum constitutive relations are obtained, based on the double sliding-plane model which includes dilatancy (densification), pressure sensitivity, and, most importantly, the anisotropy due to the fabric structure which enters into the resulting continuum constitutive relations through a back stress. References Nemat-Nasser, S., "On Behavior of Granular Materials in Simple Shear," Soils and Foundations, Vol. 20 (1980), 59-73. Christoffersen, J., M.M. Mehrabadi, and S. Nemat-Nasser, "A Micromechanical Description of Granular Material Behavior," Journal of Applied Mechanics, Vol. 48 (1981), 339-334. Oda, M., S. Nemat-Nasser and M.M. Mehrabadi, "A Statistical Study of Fabric in a Random Assembly of Spherical Granules," International Journal for Numerical and Analytical Methods in Geomechanics, Vol. 6 (1982), 77-94. Balendran, B. and S. Nemat-Nasser, "Double Sliding Model for Cyclic Deformation of Granular Materials, Including Dilatancy Effects," Journal of Mechanics and Physics of Solids, Vol. 41, No. 3 (1993), 573-612.

9:00 AM Invited

Fundamental Aspects in Shock Consolidation of Metal Powders: Dr. Karl Staudhammer[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., MST-6, MS G770, Los Alamos, NM 87545 USA

Shock consolidation has been widely investigated but little used in manufacturing processes. The potential arenas of application are numerous, however, for a number of reasons its employment is very limited. In part, because the process is not well understood and has inherent in it many variables. At the present time, no general accepted theory is available to account for the process mechanics, threshold conditions including very rapid reaction kinetics. Of utmost concern in the use of shock energies for compaction and consolidation is the appropriate deposition of energy, which involves a very rapid and intense shock wave. This paper will describe the many variables as applied to powder consolidation. Many of these variables are interdependent parameters such as shock pressure, strain, strain rate, temperature, shock velocity, powder size, powder distribution, powder morphology, etc. These variables must be assessed as an accumulative sum. Many descriptions of these variables and their effects have been previously presented in various forms for particular systems. However, a conceptual description in temperature-time space as applied to powders has been quit useful in understanding many of the consolidation successes and failures.

9:30 AM Invited

Hot Shock-Consolidation of Coated Powders: L. A. Japaridze[1]; *A. B. Peikrishvili*[1]; F. D. S. Marquis[2]; [1]Institute of Mining Mechanics, Acad. of Sci. of GA, Tbilisi, GA USA; [2]South Dakota School of Mines and Technology, College of Mats. Sci. and Eng., Rapid City, SD 57701 USA

The utilization of coated powders in hot shock compaction has some advantages such as uniformity of bonding, homogeneity of microstructure, higher density, improved strength and sometimes unusual microstructures. In this investigation elemental powders (Ni, Al, Fe and graphite) and refractory powders (W, WC) coated by Cu, Ni and Al have been studied, in the temperature range 400-

Index

L

M

N

O

P

Q

R

S

T

U

V

W

X

Y

Z