

Technical Program

TUESDAY AM

DISCONTINUOUSLY REINFORCED ALUMI-NUM-COMPOSITES: PRESENT & FUTURE: Secondary Processing and Performance of Discontinuously Reinforced Aluminum

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Warren H. Hunt, Aluminum Consultants Group, Inc., Murrysville, PA 15668 USA

Tuesday AM Room: Dublin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

8:30 AM INVITED PAPER

DESIGN OF MICROSTRUCTURES FOR OPTIMUM FRACTURE TOUGHNESS: *D. L. Davidson*¹; ¹Southwest Research Institute, San Antonio, TX USA

The fracture toughness of particulate reinforced composites are below those desired for useful engineering materials. Models that account for the effects of microstructure have been developed and used to optimize fracture toughness. Constraint of deformation by particles is identified as an important influence of microstructure on fracture, and is incorporated in the models. The purpose of the models is to focus processing efforts on those issues that can enhance fracture toughness. Finally, the issue of using shearable particles to enhance fracture toughness is addressed.

9:00 AM

THERMAL FATIGUE OF ALUMINUM-BASED MMCS STUDIED BY MECHANICAL SPECTROSCOPY: *E. Carreno-Morelli*¹; R. Schaller¹; ¹Ecole Polytechnique Federale de Lausanne, Institut de Genie Atomique, PHB-Ecublens, Lausanne CH-1015 Switzerland

The mechanical behaviour of aluminium based composites reinforced with short Al₂O₃ SAFFIL fibres has been investigated by mechanical loss and elastic shear modulus measurements during thermal cycling. A mechanical loss maximum which is not present in the unreinforced alloys has been observed during cooling near 120K. It originates in the relaxation of thermal stresses at the interfaces due to the differential thermal expansion between matrix and reinforcement. The maximum height increases with the volumetric fibre content. In addition, if the matrix strength is increased by the appropriated choice of alloy and thermal treatment, the maximum diminishes and shifts to lower temperatures. No damage accumulation at the interfaces has been detected during long period thermal cycling in the range 100-500K. A description of the damping behaviour is made in terms of the development of microplastic zones which surround the fibres. Such zones, in which dissipation takes place, would be beneficial for increasing the life time of MMC components in service conditions.

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MATRIX EFFECTS ON DEFORMATION AND FRACTURE OF DISCONTINUOUSLY REINFORCED ALUMINUM (DRA) COMPOSITES: *I. Dutta*¹; T. R. McNelley¹; ¹Naval Postgraduate School, Monterey, CA USA

This talk will present a review of the impact of matrix microstructure and flow behavior on deformation and fracture of DRA. Results of experimental studies delineating how the matrix grain structure and grain boundary character, dislocation sub-structure and precipitate state can influence both ambient and high temperature flow properties will be discussed. Additionally, the roles of these parameters on fracture toughness will also be addressed. In particular, it has been found that a number of unconventional heat treatments resulting in unusual precipitate states may be advantageously utilized to produce significant changes in both flow and fracture properties of DRA. The microstructural changes associated with these heat treatments will be discussed, and their impact on the mechanical properties of DRA will be presented.

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EXTRUSION MODELING OF DISCONTINUOUSLY REINFORCED ALUMINUM COMPOSITES: *Marvin McKimpson*¹; T. Loftin²; ¹Michigan Technology University, Institute of Materials Processing, Houghton, MI 49931 USA; ²DWA Aluminum Composites, 21130 Superior Street, Chatsworth, CA 91311 USA

A major factor limiting the introduction of wrought discontinuously reinforced aluminum composites into new applications is the high cost of developing and demonstrating the secondary processing required for these applications. More extensive use of numerical metal forming simulation tools has excellent potential for reducing this cost. Accordingly, a program has been initiated to utilize DEFORM, a commercial finite element simulation code, to model hot extrusion of 180 mm dia. 6092/SiC/17.5p rounds into simple shapes. The primary objective of this work is to develop an improved understanding of the factors controlling process parameter selection for extrusion of these commercial composites. The program involves developing appropriate high temperature flow data on the 6092/SiC/17.5p material, constructing an appropriate geometric and thermal model for the extrusion press being used, and then combining the two to simulate the extrusion operation. The status of this program, as well as recent research results, will be reviewed.

11:00 AM

MILLING AND DRILLING OF GrA-Ni : A Graphitic-SiC MMC: V. Songmene¹; M. Balazinski²; R. Maranzana¹; ¹Industrial Research and Development Institute, P.O. Box 518 L4R 4L3 Canada; ²Ecole Polytechnique de Montreal, C.P. 6079, Centreville, Montreal QC H3C 3A7 Canada

A new MMC consisting of an aluminum matrix reinforced with SiC and nickel-coated graphite particles, GrA-Ni, was developed two years ago. This composite has a low weight/volume ratio, and higher wear resistance, properties that make it suitable for brake rotors and liners as replacement for grey cast iron. The addition of nickel-coated graphite particles to SiC particulate in the aluminum matrix has improved the machinability of the SiC-reinforced MMC. But the tool performance is still poor compared to that obtainable on high silicon content aluminum alloys. The expansion of the use of MMCs in engineering applications requires more machining data on strategies to cost effectively cut these composites. GrA-Ni is relatively new and there is a very limited quantity of published data on its machinability. The paper investigates machining strategies to cost effectively rough GrA-Ni with carbide tools. A design of experiment type of approach was first used to compare the machining behavior of

GrA-Ni and existing aluminum SiC-reinforced MMCs at different cutting conditions. Several machining parameters (speed, feed, depths of cut) were used to machine the GrA-Ni composite and their impact on tool wear, tool life, volume of metal removed and unit machining cost were analyzed.

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COMMERCIAL DEVELOPMENT OF DRAS FOR PERFORMANCE AND LOW COST: *J. C. Withers¹; S. M. Pickard¹; ¹MER Corporation, Tucson, AZ USA*

Commercial applications of DRA for thermal management, structural applications and sporting goods are reviewed with case studies of MERs activities in these areas. Microstructure and property interrelationships for specific material applications with best selection of reinforcement phase and matrix alloy by performance and cost criteria are discussed.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Synthesis, Processing, And Experimentation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Flow & Fracture Committee, Mechanical Metallurgy Committee

Program Organizers: Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Marc A. Meyers, IMM, University of California, San Diego, CA 92093 USA; Naresh Thadhani, Georgia Inst of Tech, Sch of Matls Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Tuesday AM Room: Sydney
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA; Naresh N. Thadhani, Georgia Institute of Technology, Materials Science and Engineering, Atlanta, GA 30332-0245 USA

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REACTION SYNTHESIS OF SHOCK-DENSIFIED TITANIUM-BASED INTERMETALLIC AND CERAMIC COMPOUNDS: *Shantanu Ashok Namjoshi¹; Naresh N. Thadhani¹; ¹Georgia Institute of Technology, Materials Science and Engineering, 778 Atlantic Drive NW, Atlanta, GA 30332-0245 USA*

Shock densification was used to obtain dense unreacted compacts of Ti-Si (Ti₅Si₃), Ti-B (TiB₂), and Ti-Al (TiAl) powders mixed in stoichiometric ratios. The compacts (~ 85-90% dense) were characterized to determine the defect densities and crystallite size reduction due to the large deformations and fracture introduced during dynamic densification process. Systematic heat treatments were carried out on these systems to determine the reaction synthesis mechanisms and their enhanced kinetics. The highly-activated and dense-packed state of these materials resulted in complete chemical reactions at much lower temperatures than that at which reactions normally initiate with the melting of one of the components. The synthesis process was dominated by defect-enhanced solid-state diffusion reactions followed by a small amount of combustion reaction. Modeling of the heat and mass transfer processes occurring during reaction synthesis of these materials, was performed to determine the balance between the rate of heat generation and dissipation, thereby obtaining the limiting conditions for the prevention of the onset of the combustion reaction. Consequently the reaction synthesized compacts had higher

hardness, and were dense and free from defects, typically associated with combustion synthesis. In this presentation, the experimental and modeling results and characteristics of the synthesized Ti-Al, Ti-Si, and Ti-B based intermetallics, ceramics, and their composites will be presented. Funded by ARO Grant Number: DAA G55-97-1-0163

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LASER-DRIVEN SHOCK LOADING DEVICE FOR TIME RESOLVED MEASUREMENT OF SHOCK INDUCED REACTION: *Tatsuhiko Aizawa¹; ¹University of Tokyo, Dept. of Metallurgy, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan*

Through the systematic studies in the recent years, it has been found that the shock induced reaction should have its intrinsic feature as compared with the shock assisted reaction, where the thermal transients during shock loading governs the whole process in reaction. Furthermore, use of mechanically alloyed precursor (MA precursor) enables us to reduce the shock pressure condition for ignition of reactions. Although the reaction process can be described through the recovery experiments for the shock induced reaction into aluminides and silicides from the starting constituent elements by varying the shock condition and the premixing parameters in preparation of MA precursor, no direct information can be obtained with respect to the shock reaction mechanism during shock loading. In the present paper, new shock loading procedure is proposed for time resolved measurement to describe the shock reaction process on the route of the laser-driven shock loading. Three points to be noticed here are: 1) Adaptive shock cell to laser-driven shock loading, 2) Investigation of shock induced reaction from the MA precursors, and 3) Time resolved measurement of shock Hugoniot by using the VISAR. Using the two types of shock cell for time resolved measurement and shock recovery experiments, we can describe the time history of particle velocity by using the VISAR and evaluate the synthesized materials by XRD and microstructure analysis. Mo - Si system is employed for a targeting material to investigate the shock ignition condition from MA precursor with different premixing level to molybdenum di-silicides.

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DYNAMIC POWDER COMPACTION BY THE ION-BEAM DRIVEN SHOCK LOADING: *Minoru Tasuchida¹; Tatsuhiko Aizawa²; Kozuhiko Horioka³; ¹University of Tokyo, Graduate School of Engineering, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan; ²University of Tokyo, Department of Metallurgy, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan; ³Tokyo Institute of Technology, Department of Energy Science, Nagatsuda 4259, Yokohama, Kanagawa 226 Japan*

New methodologies have been studied to improve the number of event actions for shock loading and to make repetitive shock compaction instead of the powder or gas guns and the explosive detonation methods. Use of powder laser becomes the first candidate to make time-resolved evaluation on the dynamic behavior during the shock loading. Since the yield of recovered sample is limited to very small amount, however, another candidate method is waited especially for shock recovery experiments. Authors have been developing the ion-driven shock loading device both for higher-velocity experiments and for shock cold and hot compactions. Different from the laser-driven shock loading devices, relatively large samples can be shock consolidated by repetitive application of high pressure pulses. The flyer can be accelerated to the targeting velocity by tuning the shock loading conditions: the carrier gas species for generation of plasmas, and the voltage of condenser bank for acceleration of plasmoids. In the present paper, our developed ion-beam driven shock loading system is briefly introduced with some comments on its capacity for acceleration and the real time measurement of flyer velocity. Two targeting material systems are employed for cold and hot compaction: 1) Rapidly quenched Al-based amorphous alloys powders, and 2) Ne-Fe-B magnetic amorphous alloys powders. Parametric study is first done to determine the adequate shock condition for successful recovery and to investigate the effect of shock pressure on the change of XRD profiles for the recovered samples. The

holding temperature is also varied to understand the effect of temperature on the compaction behavior during the shock loading.

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INVESTIGATION OF COMBINED REACTION PROCESSING AND DYNAMIC DENSIFICATION IN Mo + 2 Si POWDER MIXTURES: *Kevin S. Vandersall*¹; Naresh N. Thadhani¹; ¹Georgia Institute of Technology, Materials Science and Engineering, 778 Atlantic Drive, Atlanta, GA 30332-0245 USA

Statically pressed Mo-Si powder mixtures (~50-55% dense) were densified employing shock and magnetic implosion techniques. Reaction was observed in some of the recovered compacts and the resulting microstructure revealed differences associated with whether the reaction occurred due to shock pressure or temperature. These differences provide information on the mechanism of reaction, which can be characterized as shock induced (occurring during the high pressure shock state) or shock assisted (occurring subsequent to the shock event, but due to bulk temperature increases). The un-reacted compacts (~78-95%) were subsequently reacted by thermal treatments under controlled conditions. The influence of densification parameters including initial packing density, pressure utilized, and loading conditions (which in turn control the compact density, defect concentration, and minimization of macroscopic cracking) on the reaction behavior was investigated using x-ray diffraction (XRD) and optical and scanning electron microscopy (SEM). This paper will outline the methods applied and microstructures of MoSi₂ intermetallic formed by reaction processing under different conditions. Work supported by ARO/AASERT Grand No. DAAH04-95-1-0235.

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A CRITICAL ANALYSIS OF THE SHPB THROUGH SIMULATION AND EXPERIMENT: *David A. Gorham*¹; Xiaojun J. Wu¹; ¹The Open University, Faculty of Technology, Walton Hall, Milton Keynes, Buckinghamshire MK7 6AA UK

The paper will present results from a project in which we have examined the reliability of the compressive split Hopkinson pressure bar (SHPB) test by exploring material models in experimentally-validated simulations. The experimental work was with a 6.7 mm SHPB system, using copper specimens. For these measurements we have paid careful attention to dispersion, friction and calibration. The numerical work has been carried out with axisymmetric models in DYNA2D. Very good agreement with experiment can be obtained using a simulation model in which the input bar is truncated at the strain gauge position and is loaded at that point with an experimentally measured incident pulse. The simulation is fast enough for material parameters in an appropriate model to be adjusted by trial and error until the total force on the output face of the simulated specimen matches closely with the transmitted pulse from the experiment. This agreement with experiment has given us confidence that the detailed numerical history and mapping of stress and strain within the specimen are realistic. Such simulation alongside experimental work gives a great deal of information about the stress and strain distribution that would be impossible to obtain any other way. Using this technique we have examined the uniformity of stress, strain and strain rate over a range of specimen geometries and loading rates. This has shown that inhomogeneous deformation can be significant in areas of operation of the SHPB test that are accepted as valid. A number of examples will be presented. The close agreement between simulation and experiment means that the fitted material parameters can be regarded as the results of the test. The simulation model we have developed is fast enough, with current computer technology, to be used on a routine basis, and so a possible approach to improve the reliability of stress-strain results from the SHPB is to parallel each experiment by fitting such a simulation. An important point is that valid material characteristics can be obtained by such simulation-supported tests even when deformation is not uniform, and so provides the opportunity to obtain reliable results over a wider range of SHPB operating conditions than can be achieved at present. These possibilities will be discussed in the paper.

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ATOMISTIC FINITE DEFORMATION HIGH STRAIN RATE SIMULATIONS OF SINGLE CRYSTALS: *Mark F. Horstemeyer*¹; Mike Baskes¹; ¹Sandia National Labs, MS9405, 7011 East Ave, Livermore, CA 94550 USA

Atomistic simulations employing the Embedded Atom Method are used to determine length scale effects related to macroscopic mechanical quantities under high strain rates. Various orientations of single crystal Nickel and Copper have been deformed numerically under compression and simple shear loading up to 30% strain. The motivation for this study is to develop mathematical expressions for bridging length scales within the context of internal state variable viscoplasticity. In order to bridge scales, we believe that a systematic method of determining hardening mechanisms from a bottom-up approach is key. As such, we examine the misorientation, microtexture, and heterogeneous hardening that occurs within the grain. These simulations indicate that determining the continuum effects related to spatial length scales is feasible up to the micron level based on the recent improvements of parallel computing.

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SIMULATION OF SHEAR PLUGGING THROUGH THIN PLATES USING THE GRIM EULERIAN HYDROCODE: Philip Church¹; Ian Cullis¹; Nick Lynch¹; ¹DERA, Warheads Division, Fort Halstead, Sevenoaks, Kent TN14 7BP United Kingdom

Ballistic experiments have been performed using aluminium spheres against 10mm RHA, MARs 270, MARs 300 and titanium alloy plates to investigate the influence of plugging mechanism on material properties. The experiments have measured the threshold for plug mass and velocity as well as the recovered aluminium sphere mass over a range of velocities. Some of the experiments have been simulated using the in-house second generation Eulerian hydrocode GRIM. The calculations feature advanced material algorithms derived from interrupted tensile testing techniques and a triaxial failure model derived from notched tensile tests over a range of strain rates and temperatures. The effect of mesh resolution on the results has been investigated and understood. The simulation results illustrate the importance of the constitutive model in the shear localisation process and the subsequent plugging phenomenon. The stress triaxiality is seen as the dominant feature in controlling the onset and subsequent propagation of the crack leading to the shear plug. The simulations have demonstrated that accurate numerics coupled with accurate constitutive and fracture algorithms can successfully reproduce the observed experimental features.

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THE USE OF TRANSIENT X-RAY DIFFRACTION FOR SHOCK WAVE AND HIGH PRESSURE MATERIALS SCIENCE: *Allan A Hauer*¹; G. A. Kyrala¹; R. A. Kopp¹; D. J. Thoma¹; K. Chen¹; F. Chu¹; D. C. Wilson¹; P. Gobby¹; L. Foreman¹; J. S. Wark²; A. Loveridge²; D. H. Kalantar³; B. Remington³; J. Colvin³; M. A. Meyers⁴; J. Asay⁵; C. Hall⁵; T. Tracano⁵; ¹Los Alamos National Laboratory, Group P-24, E 526, Los Alamos, NM 87545 USA; ²Oxford University, Dept. of Physics, Oxford, Oxfordshire UK; ³Lawrence Livermore Nat. Lab., L-473, Livermore, CA 94551 USA; ⁴Univ. of Calif. San Diego, Dept. of Ames, San Diego, CA 92093 USA; ⁵Sandia Nat. Lab., Pulsed Power Sciences Dept, Albuquerque, NM 87544 USA

Transient x-ray diffraction is a powerful tool for the study of shock wave propagation and materials effects in the condensed state. Some of the phenomena that can be studied are plastic wave propagation, generation of dislocations, and the dynamics of phase transitions. By observing the angular deflection of the diffracted beam due to the passage of a shock or stress wave a real time measurement of strain can be made (with 10 ps temporal resolution) we have recently performed a series of experiments on the Trident laser at Los Alamos and the Nova laser at Livermore that illustrate the value of transient diffraction when used in laser-based experiments. For example, by

observing the diffraction from 2 or more planes simultaneously information can be obtained on plastic wave propagation. For purely elastic solid response, uniaxial compression in the direction of shock propagation would be expected. If, however, significant plastic deformation occurs a diffracted signal from planes orthogonal to the shock propagation might be expected. We have observed such a signal in experiments with LiF crystals. Similar experiments with silicon have not yet shown this behavior but exhibit other interesting phenomena such as the splitting of diffracted lines which may give temporally resolved information on phase changes. In recent work we have applied transient diffraction to the study of Beryllium. Beryllium is one of the most promising materials for the fuel containing capsule in inertial confinement fusion (ICF) target designs that may achieve ignition. Beryllium has many unusual properties such as extremely high tensile strength, and high density for such a low x-ray opacity that make it valuable as an ICF capsule material. Fabrication into a spherical capsule, however, presents a number of challenges that must be overcome through materials properties research. In addition, the performance of a Be ignition capsule in the early stages of implosion may also depend on specific Be materials properties such as grain structure or the details of melting. As part of the capsule fabrication effort, high quality single crystal Be samples have been made. We have obtained time resolved diffraction measurements using these crystals that will help to clarify the behavior of Be in laser driven implosions. Recent results from these experiments will be presented.

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TRANSIENT X-RAY DIFFRACTION OF SHOCK COMPRESSED CRYSTALS USING THE NOVA LASER: *Daniel H. Kalantar*¹; E. A. Chandler¹; J. D. Colvin¹; K. Mikaelian¹; B. A. Remington¹; S. V. Weber¹; L. G. Wiley¹; A. A. Hauer²; J. S. Wark³; *M. A. Meyers*⁴; G. Ravichandran⁵; ¹Lawrence Livermore National Lab, L-473, 7000 East Ave, Livermore, CA 94550 USA; ²Los Alamos National Lab., P.O. Box 1663, Los Alamos, NM 87545 USA; ³University of Oxford, Clarendon Laboratory, Parks Road, Oxford OX1 3PU UK; ⁴University of California, San Diego, Dept. of Applied Mechanics and Engineering Sciences, MC-0411, San Diego, CA 92093 USA; ⁵California Institute of Technology, Pasadena, CA 91125 USA

We are conducting experiments on the Nova laser to investigate the Rayleigh-Taylor instability in metal foils maintained in the solid state. In these experiments, we use a hohlraum x-ray drive to ablatively accelerate a thin Cu foil that has a preimposed modulation at the ablator-metal interface. We use x-ray backlighting to characterize the instability growth. By temporally shaping the drive history, the foil is kept below the melt temperature at peak pressures up to 3 Mbar under extreme strain rates (10^7 - 10^9). In support of this effort, we are developing dynamic diffraction as a diagnostic of material state for shock compressed samples. The crystal samples are shock compressed with an x-ray drive, and x-rays from a point backlighter are used to measure the lattice compression by diffraction. We have observed shock compression of more than 10% in 1-D in a Si crystal. In addition, the crystal is probed by simultaneous Bragg and Laue diffraction of orthogonal lattice planes [1] to study the transition from elastic to plastic compression in a shocked solid. We are also working to use dynamic Bragg diffraction as a way to verify that the metal foils used in the instability growth experiments remain solid at peak compression. We will present results from the instability growth measurements of Cu foils, and dynamic Bragg experiments using shocked Si. [1] R. R. Whitlock and J. S. Wark, Phys. Rev. B 52, 8 (1995).

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DEFORMATION LOCALIZATION OF 8090 Al-Li ALLOYS DURING DYNAMIC LOADING AT ROOM TEMPERATURE AND LIQUID NITROGEN: *Y. B. Xu*¹; W. L. Zhong²; Y. J. Chen³; Y. L. Bai²; M. A. Meyers³; ¹Chinese Academy of Sciences, Institute of Metal Research, Shengyang, P.R. China; ²Chinese Academy of Sciences, Institute of Mechanics Research, Beijing, P.R. China; ³Univer-

sity of California, San Diego, Materials Science Group, 9500 Gilman Drive, La Jolla, CA 92093-0411 USA

Shear localization at a strain rate of 10^3 s⁻¹, was studied in 8090 Al-Li alloys at 298 and 77 K in the (1)naturally-aged, (2)peak-aged, (3)under-aged, (4)over-aged conditions. The peak-aged Al-Li alloy had a higher tendency to form a shear the other three other conditions. In the peak-aged Al-Li alloy, double shear bands and kink bands were also observed. SEM revealed damage of the microstructure within the shear band. The sharp drop in the load is associated with growth and coalescence of microcracks and microvoids rather than the occurrence of shear localization, but shear localization is seen to accelerate their growth and coalescence. TEM of matrix and transition region showed an elongated structure with high dislocation densities. Structure becomes fragmented in the margin of shear band. In center, fine equiaxed grains with lower dislocation densities are observed, due to the effects of the large temperature rise and the highly localized deformation in an extremely short time. Evidence for dynamic recovery and dynamic recrystallization is found.

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Surface Energy Effects in Microstructure Formation; and Poster Talks

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223, Gaithersburg, MD 20899; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Tuesday AM
October 13, 1998

Room: International Ballroom
Location: O'Hare Hilton Hotel

Session Chair: Robert Balluffi, MIT, Cambridge, MA 02139-4307

8:30 AM INVITED PAPER

MORPHOLOGY: THERMODYNAMICS AND KINETICS: *W. Craig Carter*¹; ¹MIT, Dept. of Materials Science, 77 Massachusetts Ave, Cambridge, MA 02139-4307 USA

John Cahn's career has influenced a remarkable breadth of topics in materials science. The excitement of discovery and quality of instruction that he has provided for his colleagues cannot be overestimated. This talk will illustrate some work with Cahn which lead to the convergence of fundamental principles from his previous, and seemingly disparate, contributions to materials science: morphology, kinetics and thermodynamics. The principles behind the development of corners in a Wulff shape and the thermodynamics of a miscibility gap and the conditions for a steady-state growth shape all have the same underlying theoretical structure. The common tangent rule is related to the Wulff construction through convexification of a free energy surface. Because the choice of the metric for convexification has traditionally been different for these two topics, their connection has not been obvious. Likewise the principles be-

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hind the Gibbs-Duhem equation and the Cahn-Hoffman equations are the same; furthermore, they are equivalent to the method of characteristics which can be applied to growth shapes for particular physical models. The depth of understanding which is established by such connections will be discussed as well as applications to observed materials phenomena.

9:15 AM INVITED PAPER

TRIPLE JUNCTIONS: *Jean E. Taylor*¹; ¹Rutgers University, Mathematics, Hill Center, Busch Campus, 110 Frelinghuysen Road, Piscataway, NJ 08854 USA

It was John Cahn's interest in soap bubbles and soap films, in particular my proof that the only singularities were smooth triple junction curves and isolated tetrahedral points (as observed by Plateau a century earlier), that drew him to a lecture I gave 25 years ago and eventually led to our long collaboration. About that time he, together with Hoffman, was investigating which multiple junctions might be possible for anisotropic surface free energies. Both he and I have continued to be interested in multiple junctions and how they move. Much of our joint and separate work on this topic will be reviewed, together with observations concerning positive and negative triple junction line energies.

10:00 AM BREAK

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WETTING AND ADSORPTION TRANSITIONS IN Ga-Pb ALLOYS: *Paul Wynblatt*¹; Dominique Chatain²; ¹Carnegie Mellon University, Dept. of Mater. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; ²CMRM2-CNRS, Campus de Luminy, Case 913, F-13288 Marseille Cedex 9, Bouches du Rhone France

In 1977, Cahn predicted that a transition from incomplete to complete wetting should occur, at some temperature below the critical temperature, in binary systems displaying a liquid miscibility gap. Results will be described which establish the complete wetting of Ga-rich liquids by Pb-rich liquids, by means of Auger electron spectroscopy (AES) measurements at the surface of two-phase liquid mixtures. The measurements also demonstrate, as expected from Cahn's theory, that the thickness of the Pb layer on the Ga surface diverges as liquid-liquid coexistence is approached. Recent determinations of adsorption at single phase liquid Ga surfaces, performed by both surface energy and AES measurements, will be discussed in the light of so-called prewetting transitions also predicted by Cahn.

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BOUNDARY MOTION BY CURVATURE IN THE FERROMAGNETIC POTTS MODEL WITH TRIPLE JUNCTIONS: *Mark A. Miodownik*¹; Anthony D. Rollett²; Elizabeth A. Holm¹; ¹Sandia National Laboratories, Materials Theory & Computation, P.O. Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; ²Carnegie Mellon University, Matls Sci & Eng Dept, Pittsburgh, PA 15213-3890 USA

The ferromagnetic Potts model has been used extensively to simulate grain growth in polycrystalline materials. While excellent agreement is found in many cases, the limits of applicability of the model are not well-known. In this study, we examine the evolution of a simple polycrystalline system of known, constant driving force: columnar grains with a single crystal cap. This system contains only grain boundaries and triple junctions, and no topological changes occur during evolution. For isotropic boundary energy and uniform boundary mobility, this system evolves with dynamics equal to the continuum case, except at very small grain widths, where lattice and stochastic effects cause deviations. When boundary mobilities vary, the system finds a steady state in which the grains shrink uniformly; that is the product of mobility and curvature is constant for all the moving boundaries in the system. For anisotropic boundary energies, the system evolves correctly as well, but it becomes much more sensitive to anisotropy in the underlying lattice. Similar results are

found in three dimensions. The case of four-grain junctions is briefly discussed.

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BOUNDARY MOTION BY DISCRETIZED MEAN CURVATURE IN THE FERROMAGNETIC ISING MODEL: *Elizabeth A. Holm*¹; John W. Cahn²; *Mark A. Miodownik*¹; ¹Sandia National Laboratories, Materials Theory & Computation, P.O. Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; ²NIST, 223/A153, Gaithersburg, MD 20899 USA

The ferromagnetic Ising model is used to study coarsening of systems driven by surface energy minimization, such as magnetic domain coarsening. However, the relationship between the motion of a discretized boundary with energy determined by neighbor counting and the motion of a continuous boundary by curvature has not been elucidated. We show that for a ferroelectric Ising model with Metropolis dynamics at $T=0$, a boundary discretized on a two-dimensional lattice moves with velocity proportional to its discrete mean curvature, independent of the magnitude of the surface free energy per unit length. Simulations of isolated boundaries (i.e., shrinking circle and hairpin bicrystal) confirm this prediction. Similar results are found in three dimensions, although artificial pinning may occur for lattices of low coordination number. Boundary segments in ferromagnetic Potts models used to simulate grain growth also move by discretized mean curvature, in contrast to grain boundaries, which move by weighted mean curvature. The presence of domain junctions also influences boundary motion in such systems.

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THE EQUILIBRIUM SHAPE OF LIQUID Pb AT TRIPLE JUNCTIONS IN Al: *Heike Gabrisch*¹; ¹Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Building 72, 1, Cyclotron Road, Berkeley, CA 94720 USA

The effect of liquid metals on the properties of polycrystalline materials is well established. However, most investigations of the underlying wetting phenomena rely on indirect evidence. Here we present direct observations of the behavior of liquid Pb at grain boundaries in Al. Using in-situ transmission electron microscopy, we have determined the shape of liquid Pb inclusions in solid Al at the intersection of grain boundaries with the foil surfaces and at grain boundary triple junctions. It was found that dihedral angles along triple junctions are smaller than along grain boundaries, leading to a greater tendency for wetting. The observed shapes could be used to determine relative interfacial energies and may be understood in terms of a simple model. This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-ACO3-76SF00098.

10:40 AM

TRIPLE JUNCTIONS IN ANISOTROPIC MATERIALS: *Alexander H. King*¹; ¹State University of New York, Materials Science & Engineering, Nicolls Road, Stony Brook, NY 11794-2275 USA

The measurement of dihedral angles at triple junctions is a time-honored method of determining interfacial energies, but is only used under the assumption of isotropic interfacial energy. The Cahn-Hoffmann capillarity vector provides a mechanism for extending the analysis to anisotropic cases. We demonstrate some applications of this approach in this presentation, concentrating upon cases in which it is known that at least one boundary plane lies in an energy-minimizing orientation. It is shown that the dihedral angles are not uniquely defined for these cases, and that they can instead fall anywhere within specified "allowed ranges." This allows for a variability of dihedral angle that can be used to minimize interfacial curvature, and thus the driving force for migration. These effects are all demonstrated through TEM observations Acknowledgement: This work is supported by NSF Grant # DMR 9530314

10:45 AM

INTERATOMIC POTENTIAL AND ATOMIC STRUCTURE OF A MODEL CERAMIC/METAL INTERFACE: {222}MgO/Cu: *Roy Benedek*¹; *David N. Seidman*²; L. H. Yang³; L. H. Yang³; L. H. Yang³; A. Alavi⁴; ¹Argonne National Laboratory, Building 223, Argonne, IL 60439 USA; ²Northwestern University, Materials Science and Engineering Dept., 2225 North Campus Drive, Evanston, IL 60208-3108 USA; ³Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; ⁴University of Belfast

Ab initio local-density-functional-theory (LDFT) calculations (with the plane-wave-pseudopotential method) and classical molecular statics simulations are applied to {222}MgO/Cu, a model interface that has been the subject of extensive experimental observation. This interface is polar, has a misfit of about 1/6, and is invariably oxygen terminated. Relatively small-cell (less than approximately 30 atoms) LDFT total-energy calculations are performed for coherent and semicoherent {222}MgO/Cu interfaces, as a function of interface spacing and translation state, to elucidate the interface potential. The results suggest a simple short-range interface interatomic potential that includes an attractive contribution and a repulsive Born-Mayer contribution. Molecular statics calculations with this potential predict a misfit dislocation network with trigonal symmetry and no standoff dislocations. This potential, however, is able to address atomic relaxation only on the metallic (Cu) side of the interface. LDFT calculations for large supercells that include misfit (with up to 360 atoms) enable the validity of the dimple interface potential to be assessed. *Supported by the U.S. Department of Energy.

10:50 AM

MICROSTRUCTURAL STABILITY AND SEGREGATION IN Al-Sc-Mg ALLOYS: *Dmitry Gorelikov*¹; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

Dilute Al-Sc alloys have excellent mechanical properties at room temperature and a high yield-strength at temperatures up to 673 K, due to the presence of elastically hard coherent and unsharable Al₃Sc precipitates that can be obtained at a high number density [R.W. Hyland, Jr. Metall. Trans. A 23A, 1947 (1992)]. These Al₃Sc precipitates remain coherent to a diameter of 20-25 nm, since the lattice parameter mismatch is only about 1%. The addition of Mg prevents recrystallization of Al-Sc alloys up to the solidus temperature. Precipitation hardening of an Al-0.2 wt.% Sc-Mg alloy is studied by atom-probe field-ion and transmission electron microscopies. The effects of segregation of Mg to {100} Al₃Sc/Al interfaces on the coarsening of coherent and semicoherent Al₃Sc precipitates is studied. The Gibbsian interfacial excess of solute at the {100} Al₃Sc/Al interface is studied as a function of ageing temperature, time, and precipitate diameter. *This research is supported by the National Science Foundation/DMR.

10:55 AM

LOCAL STRESSES AT INTERFACES AND THEIR EFFECTS ON SEGREGATION: *Olof C. Hellman*¹; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

Solute segregation to interfaces is often driven by stress effects; a solute atom's placement at or near a grain boundary (GB) can relieve both the stress associated with a solute atom in the host crystal structure and the local stress intrinsic to the grain boundary. Frequently, the GB structure and its local state of stress is simple enough that a one-dimensional model of segregation behavior suffices for explaining the effect of stress on segregation. This has been fortunate, because in the past, experimental examination of segregation to GBs was only possible on a one-dimensional level. Many GBs, however, are expected to have very complex structures for which a one-dimensional treatment does not suffice. Now that experimental techniques, such as three-dimensional atom-probe microscopy, are providing richer experimental data, our theoretical treatment of local stress at interfaces needs to be expanded to examine stress distributions in three-dimensions near interfaces, and to resolve each compo-

nent of the local stress. We are investigating the interrelation of the effects of atomic structure, local strain and impurity segregation in a series of crystallographically distinct GBs. We use an embedded-atom method (EAM) type potential for modeling atomic interactions, and Monte Carlo and energy minimization techniques for calculating atomic positions and solute segregation. We attempt to draw general conclusions about segregation at interfaces by correlating segregation behavior with each of the components of the local stress tensor. *This research is supported by the National Science Foundation/DMR.

11:00 AM

ATOMIC-SCALE STUDIES OF EARLY-STAGE DECOMPOSITION MICROSTRUCTURES IN SYSTEMS WITH A LARGE TRANSFORMATIONAL MISFIT: *Dieter Isheim*¹; *Didier Blavette*²; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA; ²Universite de Rouen, Laboratoire de Microscopie Ionique, URA CNRS 808, Faculte des Sciences et Techniques, F-76821, Mont-Saint-Aignan, Cedex France

Elastic interactions are known to cause an alignment of coherent precipitates with a large misfit with the matrix, in elastically anisotropic matrices. A periodic arrangement of precipitates can also be produced by a spinodal decomposition process. Simply observing the spatial arrangement of precipitates may not be sufficient to reveal the actual operating mechanism. In order to obtain additional insight, atomic scale analyses of the precipitate's compositions, as well as the chemical sharpness of their interfaces, during the early stages of the decomposition is necessary. This contribution presents the results of a study of the decomposition of an Fe-20 at.% Mo alloy, employing conventional and high resolution electron microscopies, as well as atom-probe field-ion microscopy including three-dimensional atomic tomography. *Research supported by the National Science Foundation/Division of Materials Research and the Deutsche Forschungsgemeinschaft.

11:05 AM

EXPLORING GRAIN-BOUNDARY PHASE SPACE ON AN ATOMIC SCALE: SIMULATIONS AND EXPERIMENTS: *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

The concept of grain boundary (GB) phase space (with the 5 macroscopic geometric variables serving as state variables leading to a local phase rule for GBs) represents a basic thermodynamic framework for understanding GB segregation [J.W. Cahn, J. Phys. (Paris) 43, C6-199 (1982)]. We employ Monte Carlo techniques (Metropolis algorithm and overlapping distributions MC) to explore systematically the 8-dimensional GB phase space for both twist and tilt boundaries in dilute single-phase binary f.c.c. alloys; the atomic interactions are described by Baskes-Daw-Foiles embedded-atom method (EAM) potentials. We first use lattice statics calculations to determine the lowest energy GB structures and then MC simulation to calculate the solute-atom distribution and Gibbsian interfacial excess at elevated temperatures for each GB structure studied. Accurate segregation free energies for atomic sites in GBs are calculated via the ODMC technique and segregation entropies are determined and found to be a linear function of the segregation internal energies for the same GB sites. The effects of both the 5 macroscopic and 3 microscopic degrees of freedom (DOFs) are studied and it is demonstrated that the Gibbsian interfacial excess is a function of these DOFs. Also a GB's atomic structure determines the partitioning of segregating solute atoms between the cores of dislocations and in the elastic stress fields of GB dislocations. We demonstrate that none of the geometric criteria frequently suggested in the literature is capable of predicting the propensity for GB segregation, much less the magnitude. Experimental segregation 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 for a

specific GB. *This research is supported by the National Science Foundation/DMR.

11:10 AM

FIRST-PRINCIPLES STUDY OF ORDERING AND PRECIPITATION IN ALUMINUM ALLOYS: *Christopher M. Wolverton*¹; Alex Zunger¹; ¹NREL, 1617 Cole Blvd., Golden, CO 80401 USA

We demonstrate a first-principles theoretical approach for studying the ordering and precipitation in aluminum alloys. The method involves mapping atomically-relaxed first-principles total energies onto an effective Hamiltonian. The effective Hamiltonian is composed of coherency strain and chemical energy terms. By combining this Hamiltonian (which is comprised of both real- and reciprocal-space terms) with a mixed basis Monte Carlo algorithm, we are able to study the thermodynamic and structural properties of Al-rich alloys, both at high and low temperatures. The method has been applied to three Al-rich alloy systems which makes for an interesting comparison, due to the different relative contributions of strain and chemical energies: Al-Mg, Al-Cu, and Al-Ni. We find that anomalously soft coherency strain for certain preferred orientations leads to the observed precipitate shape [e.g., (001)-planar in Al-Cu and Al-Ni and more spherical in Al-Mg]. Furthermore, we demonstrate the importance of anharmonic contributions to the coherency strain for large size-mismatched alloys.

11:15 AM

COHERENCY STRAIN AND VEGARD'S LAW: *Jong K. Lee*¹; ¹Michigan Technological University, Dept. of Metallurgical and Materials Engineering, 1400 Townsend Drive, Houghton, MI 49931-1295 USA

Using the Discrete Atom Method (DAM) to examine the elastic strain energy associated with arbitrarily-shaped, elastically-inhomogeneous, coherent precipitates, both miscibility gap and lattice parameter are investigated as a function of solute composition in a binary model fcc system. The solubility limit and lattice parameter depend not only on the misfit strain but also on the solvent-solute bond length. For a given positive misfit strain, as the solvent-solute bond length approaches that of the matrix phase, the solubility limit increases and the lattice parameter shows a negative deviation from Vegard's law. To take advantage of the interphase boundary area, the minor phase tends to form a network of isolated precipitates. Vegard's law is observed when the solvent-solute bond length is equal to the average of the matrix and the precipitate phase, and a positive deviation when the bond length moves toward that of the precipitate phase. Some features of coherent phase equilibria will be compared between the DAM predictions and the previous results of Cahn and other investigators based on continuum elasticity.

11:20 AM

EQUILIBRIUM OF COHERENT PHASES IN A FILM: *Alexander L Roytburd*¹; *John W Cahn*²; ¹University of Maryland, Materials and Nuclear Engineering, College Park, MD 20742 USA; ²NIST, Materials Science and Engineering, Bld.223, Gaithersburg, MD 20899 USA

The elastic energy of two coherent phases in a film can be reduced to a minimum if the phases are arranged as two layers and the film is bent. This fact results in the appearance of a metastable two-phase equilibrium for a single-component system and in the modification of phase equilibria for a two-component system. The coherent phase diagrams for a bending film are presented and they are compared to the phase diagrams for a constrained film.

11:25 AM

MODELING KINETICS OF PHASE GROWTH WITH NON-EQUILIBRIUM INTERFACES: Brian Richard Hinderliter¹; *William C. Johnson*¹; ¹University of Virginia, Material Science and Engineering, Thornton Hall, Charlottesville, VA 22903-2442 USA

We model the kinetics of multiple phase growth via diffusion in a stressed binary alloy in which the interfaces are not in local thermo-

dynamic equilibrium. The numerical model employed uses a fine grid overlay in the region of the interface that tracks the interface, while using a coarser grid over the remainder of the material. The interface is assumed to be sharp, planar and coherent. Elastic deformation can result from misfit strain or applied stresses. The numerical model is compared to analytic solution in the limit of equilibrium boundary conditions for a binary regular solution. This work was supported by the National Science Foundation under Grant DMR-9496133.

11:30 AM POSTER VIEWING

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Mechanisms of Fatigue Crack Initiation and Propagation of Conventional Alloys

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; Daniel Eylon, Univ. of Dayton, Graduate Materials Engineering, Dayton, OH 45469-0240; J. P. Gallagher, University of Dayton, UDRI-Structural Integrity, Dayton, OH 45469-0120 USA; G. Lutjering, TU Hamburg-Harburg, Hamburg 21093 Germany

Tuesday AM Room: Athens/Berlin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: A. W. Thompson; J. Petit

8:30 AM KEYNOTE

DWELL AND ENVIRONMENTAL ASPECTS OF FATIGUE IN ALPHA/BETA TITANIUM ALLOYS: *W. J. Evans*¹; *M. R. Bache*¹; ¹University of Wales Swansea, Interdisciplinary Research Centre, Department of Materials Engineering, Singleton Park, Swansea SA2 8PP U.K.

In 1973, the Rolls Royce RB-2121 engine experienced several in-service catastrophic fan disc failures which had significant repercussions throughout the titanium industry. It became clear after major research efforts both in Europe and the United States that a number of factors were involved. Several of the problems such as inadequate work during forging, poor microstructural control and inherent defects were removed relatively quickly through changes in material – the beta processed IMI 685 was replaced with Ti-Ti-6Al-4V – improved melt practice and tighter control over working and heat treatment procedures. Other issues, such as a sensitivity to dwell under fatigue loading, have continued up to the present day and have even been implicated in recent in-service problems. The objective of the paper is to review the dwell sensitivity situation with a view to highlighting the mechanisms and arriving at a consensus understanding of the phenomenon and its implications. Over the intervening years many factors have been implicated in dwell fatigue including hydrogen and other interstitials, weak microstructural links, time dependent deformation, stress redistribution and the degree of biaxiality. The paper will explore the published work on these issues and will bring to the front unpublished research at Swansea and within the UK. This work encompasses mean stress effects, HCF/LCF interactions, tension/torsion fatigue response, 'internal' and external environments and the application of EBSD in the assessment of fatigue characteristics. It will be shown that the processes involved have important implications for fatigue crack initiation and short crack growth in general. Furthermore, the paper will explore the 'dwell' effect beyond the usual low temperature regime in which it is generally placed.

The implications for forging and microstructural control are assessed and the impact on component lifing philosophy discussed.

9:00 AM

EFFECT OF MICROSTRUCTURE ON THE DWELL FATIGUE BEHAVIOR OF Ti-6242: *Andrew P Woodfield*¹; Mark D Gorman¹; John A Sutliff²; ¹GE Aircraft Engines, MPED, 1 Neumann Way, M-89, Cincinnati, OH 45215 USA; ²GE Corporate Research and Development, 1 Research Circle, Schenectady, NY 12309 USA

This work describes the relationship between microstructure, and dwell low cycle fatigue life and fracture morphology in alpha-beta processed Ti-6242. It has been found that aligned alpha colonies that form during a beta quench in the billet cycle can survive during subsequent alpha-beta billet, forge and heat treat processing. The surviving aligned alpha colonies in forgings often cannot be recognized using conventional metallographic procedures and a new experimental technique for observing these features has been developed. This technique uses electron back scattered patterns (EBSP) generated from a sample in a scanning electron microscope and combines crystallographic information from these EBSPs to form an image of the microstructure based on crystallographic orientation of the basal pole. A method for quantifying the microstructure in terms of aligned alpha colony/alpha grain parameters has been developed and a model constructed which relates the microstructural parameters to dwell fatigue life.

9:20 AM

LOW CYCLE DWELL TIME FATIGUE IN Ti-6242: *R. Faber*¹; M. E. Kassner¹; Y. Kosaka²; B. Bristow¹; S. H. Reichman²; J. A. Hall²; ¹Oregon State University, Dept. of Mech. Engr., 204 Rogers Hall, Corvallis, OR 97331-6001 USA; ²Oremet Titanium, P.O. Box 580 530 34 Ave. SW, Albany, OR 97321

Ambient temperature low cycle dwell time and conventional low cycle fatigue tests were performed on Ti-6.0Al-2.0Sn-4.0Zr-2.0Mo-0.1Si (Ti-6242). Specimens were solution annealed at various temperatures below the beta transus to control the volume fraction of primary alpha. The influence of the changes in primary alpha phase on low cycle dwell time fatigue life (DCF) were determined and compared to the conventional low cycle fatigue (LCF) properties of the alloy. A dwell significantly decreased the number of cycles to failure. Increasing primary alpha associated with lower solution temperatures significantly increased susceptibility to low cycle dwell time fatigue although this effect diminished with decreasing stress. It is believed that the susceptibility to dwell fatigue may be associated with ambient temperature time dependent plasticity or (creep).

9:40 AM

CHARACTERISTICS OF FATIGUE SOFTENING AND HARDENING IN TITANIUM ALLOYS: *Fumio Morito*¹; Junji Takahashi¹; Seiichi Muneki¹; Toshio Kainuma¹; ¹National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Details of fatigue softening behavior have been reported for the materials such as aluminum, copper, silver and iron. However fatigue properties in titanium alloys have scarcely been examined in connection with the characteristics of crystal structure and microstructure. We studied the fatigue behavior in the rolled and the recrystallized state of pure titanium, Ti-6Al-4V and Ti-3Al-8V-6Cr-4Mo-4Zr under the stress ratio(R) between 0.1 and -1. In pure titanium and Ti-3Al-8V-6Cr-4Mo-4Zr, the maximum stress of fatigue fracture in the rolled state was higher than that in the recrystallized one. In Ti-6Al-4V, however, the maximum stress of fatigue fracture under R=0.1 or -1 was nearly same irrespective of the rolled state or the recrystallized one. The maximum stress of fatigue fracture was higher under R=0.1 than that under R=-1. Fatigue softening was recognized in pure titanium and Ti-3Al-8V-6Cr-4Mo-4Zr. Fatigue hardening was very small in Ti-3Al-8V-6Cr-4Mo-4Zr. But the fatigue softening or hardening in Ti-6Al-4V was hardly recognized due to a small difference of the strength between the recrystallized state and the rolled one. Layered and elongated substructure due to fatigue deformation was observed in

pure titanium. But such a substructure was difficult to observe in Ti-6Al-4V and Ti-3Al-8V-6Cr-4Mo-4Zr.

10:00 AM

FATIGUE STRENGTH OF TITANIUM WELDS-DEFECT SENSITIVITY: *Stig Berge*¹; ¹Norwegian University of Science and Technology, Department of Marine Structures, Trondheim 7034 Norway

Compared to steel, titanium has a very good strength/weight ratio, low modulus of elasticity, and excellent corrosion resistance. Titanium is therefore well suited as a material for risers for offshore oil and gas production. Risers are subjected to significant fatigue loading. Whereas the fatigue properties of base material have been extensively studied, very little data is available on fatigue strength and defect sensitivity of heavy section titanium fusion welds. Fatigue data are presented for TIG welded plates and pipe coupons of two different medium strength alloys, with a discussion on defect sensitivity.

10:20 AM BREAK

10:40 AM INVITED PAPER

NUCLEATION AND PROPAGATION OF FATIGUE CRACKS IN B-TITANIUM ALLOYS: *J. O. Peters*¹; G. Lutjering¹; ¹Technische Universität Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany

The influence of the microstructure on the fatigue properties of the two β -titanium alloys β -CEZ (developed by CEZUS, France) and VT 22 (Russia) was investigated. For the β -CEZ alloy a comparison between lamellar microstructures (β -processed) and bi-modal microstructures ($\alpha+\beta$ processed) at a yield stress level of 1200 MPa was performed: Bi-modal microstructures showed a higher ductility, higher LCF and HCF strength level and a higher resistance against microcrack propagation whereas lamellar microstructures showed a higher resistance against macrocrack propagation and a higher fracture toughness. These findings could be explained on the basis of β -grain size and α -plate dimensions. In the second part of this work a comparison between the bi-modal condition of the β -CEZ alloy and the VT 22 alloy was made. For this comparison the $\alpha+\beta$ processed VT 22 alloy was heat treated to the same yield stress level of 1200 MPa. The differences in mechanical properties will be discussed in terms of microstructural differences between the two alloys.

11:00 AM

MICROSTRUCTURAL ASPECTS OF FATIGUE CRACK INITIATION AND GROWTH IN Ti-10V-2Fe-3Al: *P. S. Shankar*¹; K. S. Ravichandran¹; ¹The University of Utah, Department of Metallurgical Engineering, 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The influence of microstructure on fatigue crack initiation and growth response of the beta titanium alloy Ti-10V-2Fe-3Al was investigated. Three beta-heat-treated microstructures with nearly the same tensile strength levels were employed. One was unaged, one was $a\delta$ aged and the other was $w\delta$ -aged after furnace cooling from above the beta transus. Fatigue crack nucleation studies were performed using electropolished specimens under a stress ratio R of 0.1. Fatigue crack growth behavior at $R=0.1$ was determined using compact tension specimens. Fractography was employed to identify sites of crack nucleation and the mechanism of crack growth. Replication studies were performed to identify the relative importance of crack nucleation versus crack growth of fatigue life. The effects of microstructure on fatigue resistance are rationalized in terms of deformation characteristics, size and location of fatigue crack initiation site and the intrinsic resistance to fatigue crack growth.

11:20 AM

EFFECT OF AGING ON THE DEPENDENCE OF FATIGUE CRACK GROWTH BEHAVIOR ON MEAN STRESS (STRESS

RATIO) IN THE BETA TITANIUM ALLOY: Ti-10V-2Fe-3Al: S. K. Jha¹; K. S. Ravichandran¹; ¹The University of Utah, Department of Metallurgical Engineering, 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

Effect of mean stress (stress ratio, R) on fatigue crack growth (FCG) behavior of β - aged and ω - aged microstructures of the beta titanium alloy Ti-10V-2Fe-3Al was investigated. While the mean stress had a negligible effect on the FCG behavior of the β - aged microstructure, a strong effect was observed for the ω - aged microstructure. Crack closure levels for the β - aged microstructure were found to be very low compared to the ω - aged microstructure. Transmission and scanning electron microscopic studies of microstructures and fracture surfaces were performed to gain insight into the deformation characteristics and crack propagation mechanisms in these microstructures. The results are rationalized in terms of the effect of aging on slip and crack closure.

11:40 AM

EFFECTS OF ALPHA CASE ON FATIGUE STRENGTHS IN TITANIUM ALLOYS: H. Fukui¹; H. Iizumi¹; K. Minakawa²; ¹NKK America, 1 Kokan-cho, Fukuyama, Hiroshima 721-8510 Japan; ²NKK America, 450 Park Avenue, New York, NY 10022 USA

Formation of alpha case occurs during hot working, even during superplastic forming which is generally performed in argon gas. When Ti-6Al-4V is superplastically formed at approximately 900°C, alpha case of 50-100 μ m in thickness typically forms at the surface of the alloy. Since alpha case significantly degrades fatigue properties of titanium alloys, this oxygen contamination must be removed by chemical milling which is an expensive and rather environmentally unfriendly process. In recent years, considerable attention has been given to titanium alloys with lower superplastic forming temperatures, because these alloys should be able to reduce the degree of alpha case formation. It was reported that little alpha case (less than 5 μ m in thickness) was observed in Ti-4.5Al-2Mo-2Fe after superplastic forming at 775°C. However, oxygen enrichment took place in the region approximately 30 μ m in depth from the surface. It is not clear whether or not such very thin alpha case or the oxygen enriched region affects the fatigue strength of titanium alloys. The effect of alpha case on bending fatigue strength (R = -1) is being examined for Ti-4.5Al-2Mo-2Fe and Ti-6Al-4V in the present study. Specimens are carefully heat treated in argon gas at various temperatures in order to develop a variety of thickness in α case and the oxygen enriched region at the specimen surface. Fatigue test results will be discussed in terms of thickness of alpha case and depth of oxygen enriched region.

12:00 PM

CHARACTERIZING THE CRACKING BEHAVIOR OF HARD ALPHA DEFECTS IN ROTOR GRADE TiTi-6Al-4V ALLOY: P. C. McKeighan¹; R. C. McClung¹; A. E. Nicholls¹; L. Perocchi¹; ¹Southwest Research Institute, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

Improved methods for life management of aircraft turbine engine rotors are being developed with an initial focus on fatigue cracking at hard alpha defects in titanium (FAA Grant 95-G-041). Specimens manufactured from Ti-6-4 and containing small artificial hard alpha defects (with diffusion zones) at surface or subsurface locations were tested both statically and under fatigue loading. The goal was to determine the stresses required to nucleate and grow cracks in the hard alpha core, the surrounding diffusion zone, and the matrix material. The onset and development of cracking was detected during the tests using visual and nonvisual (potential drop, acoustic emission, and ultrasonic) techniques, along with post-test destructive sectioning. Results indicate large differences in cracking behavior depending upon defect size, defect position, and loading type.

HOT DEFORMATION OF ALUMINUM ALLOYS: Constitutive Modeling, Dislocations and Grain Boundaries

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division, Metal Processing & Fabrication of Met. Soc., Non-Ferrous Metals, Shaping and Forming

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

Tuesday AM

Room: Orchard

October 13, 1998

Location: O'Hare Hilton Hotel

Session Chairs: Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Jurgen Hirsch, VAW Aluminium AG, R&D, Bonn 53014 Germany

8:30 AM INVITED PAPER

MODELLING PLASTIC DEFORMATION OF ALUMINIUM ALLOYS: E. Nes¹; K. Marthinsen²; ¹Norwegian University of Science and Technology, Department of Metallurgy, Trondheim N-7034 Norway; ²SINTEF Materials Technology, Trondheim N-7034 Norway

A new approach to the modelling of work hardening during plastic deformation of metals has recently been proposed (E. Nes, Modelling work hardening and stress saturation in FCC metals, *Progr. Mater. Sci.*, in press; K. Marthinsen and E. Nes., A general model for metal plasticity, *Mater.Sci.Engineering. A234-236*, 1095, 1997). This model is based on a microstructural concept comprising three elements, the cell/subgrain size, d , the cell interior dislocation density, ρ_i , and the cell boundary dislocation density or the sub-boundary misorientation ρ_b or f . The model is further based on a statistical approach to the storage of dislocations and the model provides a solution to the basic "dislocation-book-keeping problem" by defining a differential equation which regulates the storage of dislocations into (i) a cell interior dislocation network, (ii) increases in boundary misorientation and (iii) the creation of new cell boundaries. By combining the solution for the dislocation storage problem with models for dynamic recovery of network dislocations and sub-boundary structures, a general internal variable solution is obtained. Recently the model has been further developed by including (i): the aspect of kinetics in the calculation of work hardening, (ii): the dynamic recovery aspect associated with the thermal stability of the sub-boundary structure, (iii): the grain aspect, i.e. polycrystalline metals as well as single crystals can be handled, and (iv): the alloying aspect associated with the interaction between migrating dislocations (and/or sub-boundaries) and both precipitate particles and atoms in solid solution. The result is a complete work hardening model and associated computer code capable of providing the stress strain behaviour for a given metal or alloy composition under any combination of constant strain rate and temperature. The model has been applied to the problems of work hardening of aluminium alloys. It is demonstrated that the model predictions, in terms of microstructure evolution and associated strengthening, are in good agreement with experimental observations.

9:00 AM INVITED PAPER

CONSTITUTIVE RELATIONSHIPS FOR MODELLING HOT ROLLING OF ALUMINUM-MAGNESIUM ALLOYS: C. M. Sellars¹; Q. Zhu¹; ¹The University of Sheffield, IMMPEUS, Institute of Microstructural and Mechanical Process Engineering, Mappin Street, Sheffield, S1 1 3JD UK

During industrial hot working operations, the deformation history of material is complex in terms both of changes in strain rate and changes in strain path. These changes can be accurately predicted as a function of position in the working stock by Finite Element Modelling, but their consequences on microstructural evolution are little understood. Most laboratory studies have been carried out at constant strain rate, with simple strain paths. Current research on Al-Mg alloys has employed systematic changes in strain rate and in strain path driving hot deformation. The as - deformed microstructures have been characterised in terms of internal dislocation, density, subgrain size and subgrain boundary misorientation. These have been used as internal state variables in modelling, in which the interrelationships between the evolution equations have been considered. The effect of the variations of deformation conditions on the subsequent static recrystallisation kinetics and the recrystallised grain size have also been measured and attempts have been made to incorporate the relationships in the microstructural evolution models.

9:30 AM INVITED PAPER

MULTISCALE MODELING OF THE PLASTICITY OF FCC CRYSTALS: *L. P. Kubin*¹; ¹LEM, CNRS-ONERA, 29 Av. de la Division Leclerc, BP 72, Chatillon Cedex 92322 France

The numerical modeling of crystal plasticity involves a combination of three approaches: microscopic (atomistic scale), mesoscopic (scale of the microstructure) and macroscopic (continuum modeling). The present state of the art is discussed in the case of fcc crystals in the domain of dislocation glide and at small strains. The connection between micro- and mesoscopic aspects is performed in series. The output expected from atomistic studies is essentially concerned with properties stemming from the core structure of dislocations. For instance, very promising results have been obtained in the past two years on cross-slip properties and the structure of dislocation junctions and locks. At the mesoscopic scale, the main issues are the formation of organised dislocation microstructures in monotonic and cyclic deformation and the structure of the corresponding internal stresses. Examples will be given of the output of a 3-D, mesoscopic, numerical model. Finally, the connection between meso- and macroscopic scales has to be performed in parallel by coupling a meso simulation with a FE code. Such connection between the discrete and continuum approaches of plasticity is now possible. It poses a few interesting questions that are discussed.

10:00 AM INVITED PAPER

CONSTITUTIVE MODELING OF A 5182 ALUMINUM AS A FUNCTION OF STRAIN RATE AND TEMPERATURE: *S. R. Chen*¹; *M. G. Stout*¹; *U. F. Kocks*¹; *S. R. MacEwen*²; *A. J. Beaudoin*³; ¹Los Alamos National Laboratory, MST-8: Structure/Property Relations, Material Science and Technology Division, Mail Stop G755, Los Alamos, NM 87545 USA; ²Alcan International; ³University of Illinois at Urbana-Champaign

We have measured the stress/strain response of a 5182 aluminum alloys as a function of strain rate and temperature. We have separated the response into two categories, when the material displays a yield drop and when it does not. The yield drop was only observed if the yield stress was below 70MPa. In this case the work-hardening curve was for practical purposes flat. Within this regime the deformation has been labeled "Class A" behavior. It occurs by continuous motion of dislocations accompanied by diffusion of solute. It is furthered shown that a constitutive relation such as is appropriate to describe deformation in this temperature/strain-rate regime where the solute drag mechanism dominates. In this expression QD is the activation enthalpy for self diffusion of Mg in aluminum, which is 131 kJ/mol. In the high-stress regime, where the yield stress is above 80MPa, there is positive work hardening associated with flow stress behavior of the 5182 alloy. The yield stress was nearly constant; however, the hardening and saturation flow stress increases with decreasing temperature and increasing strain rate. In this regime the deformation is dominated by dislocation accumulation and dynamic recovery. We have found that the Mechanical Threshold Strength (MTS) model accurately describes the constitutive response as a function of tem-

perature and strain rate. We have observed that at room temperature dynamic strain aging is the dominate mechanism and the 5182 exhibits negative strain-rate sensitivity. Sponsored by: Basic Energy Sciences, Division of Materials Sciences, US Department of Energy.

10:30 AM INVITED PAPER

FLOW STRESSES AND RHEOLOGICAL BEHAVIOUR OF MODEL Al-Mg ALLOYS DURING HOT PLANE STRAIN COMPRESSION: *J. H. Driver*¹; *F. Basson*¹; ¹Ecole des Mines de Saint Etienne, Department of Microstructures and Processing, 158 Cours Flauriel, Saint-Etienne, Cedex 2 42023 France

The final properties of non-heat-treatable aluminum alloys depend to a great extent on the thermomechanical conditions of the hot deformation process. Therefore, constitutive equations relating the flow stress of a material with a known microstructure to the strain, strain rate and deformation temperature are essential for modeling and controlling the thermomechanical processing operations. Four model Al-Mg alloys (AA1070 with 0, 1, 3 and 5 wt.% Mg) have been deformed in plane strain compression up to a strain of unity using a hot channel die device. Deformation temperatures were between 20 and 500C and strain rates between 10⁻³ and 10 s⁻¹. Low strain rate tests were used to derive the rheological parameters from the isothermal stress-strain curves. Different approaches have been used for the work hardening analysis; though satisfactory results are obtained with a combination of linear and power law hardening, the best agreement is given by a modified Kocks-Mecking analysis of the work hardening rate. The accuracy of the parameters determination is discussed.

11:00 AM INVITED PAPER

GRAIN BOUNDARY PROPERTIES FOR MICROSTRUCTURAL EVOLUTION AT HIGH TEMPERATURES: *A. D. Rollett*¹; *B. L. Adams*¹; *D. Kinderlehrer*¹; *W. W. Mullins*¹; *S. Ta'asan*¹; ¹Carnegie Mellon University, Materials Science & Engineering, Rm 3327, Wean Hall, 5000 Forbes Avenue, Pittsburgh, PA 15213-3890 USA

Detailed descriptions of the properties of grain boundaries as a function of misorientation (and inclination) would be of great value in many systems and applications. This is particularly relevant to the challenge of understanding microstructural evolution via grain growth and recrystallization at high temperatures. We describe a new approach to extracting grain boundary excess free energy and (curvature driven) boundary mobility as a function of crystallographic type. The method depends on measuring very large numbers of triple junction (TJ) configurations in order to obtain statistically valid samples of relationships between energies (or mobilities) of every combination of type. The method provides relative energies (mobilities) over the entire fundamental zone and therefore requires calibration with absolute measurements of at least one reference type that is obtained independently. The extraction of boundary energies assumes local equilibrium at each TJ thus permitting the application of Herring's relations. The method is illustrated by an application to the case of a material with a strict fiber texture for which the boundary type can be simplified to a one-parameter description. The extraction of mobilities assumes that boundary velocity is proportional to energy and curvature but does not require equilibrium at each TJ. As for boundary energies the set of relative mobilities must be calibrated by the measurement of the absolute mobility at least one boundary type. Preliminary results of serial sectioning experiments on magnesia, copper and aluminum polycrystalline samples are described.

11:30 AM INVITED PAPER

GRAIN BOUNDARY EVOLUTION DURING PROCESSING AND RECRYSTALLIZATION OF SUPERPLASTIC ALUMINUM ALLOYS: *T. R. McNelley*¹; *M. E. McMahon*¹; *M. T. Perez-Prado*²; ¹Naval Postgraduate School, Department of Mechanical Engineering, 700 Dyer Road, Monterey, CA 93943-5146 USA; ²Centro Nacional de Investigaciones Metalurgical (CENIM), Avda, De Gregorio del Amo 8, Madrid Spain

Microtexture and grain boundary misorientation distributions have been studied in several superplastic aluminum alloys by means of computer-aided electron backscatter diffraction (EBSD) analysis methods. Two distinct transformation paths from the deformation microstructure of as-processed material may enable superplastic response. In alloys such as Supral 2004, the development of a superplastically enabled microstructure occurs by a continuous reaction, i.e. in a homogenous manner, during elevated temperature annealing and superplastic straining of the material. Bimodal grain boundary misorientation distributions are frequently observed in association with such reactions; such distributions may be interpreted by models combining misorientations between symmetric variants of texture components with misorientations reflecting dislocation reactions during processing. Alternatively, in aluminum alloys such as 5083 and 7475 superplastic microstructures develop by recrystallization processes involving the heterogeneous formation and subsequent growth of grains by the migration of high-angle grain boundaries. The new grains typically form within the deformation zones surrounding coarse precipitate particles and the associated grain orientations tend to be random. Correspondingly, the grain boundary misorientations tend toward random. The evolution of these different misorientation distributions during elevated temperature straining will also be described in terms of slip and grain boundary sliding.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Unusual Intermetallics Site Occupancy and Modelling

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: Ian Baker, Dartmouth College, Thayer School of Engineering, Hanover, NH 03755 USA; Easo P. George, Oak Ridge National Lab., Metals & Ceramics Division, Oak Ridge, TN 37831-6093 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

Tuesday AM Room: Madrid
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: D. Mikkola, Michigan Technological University, Department of Metallurgical and Materials Engineering, Houghton, MI 49931-1295 USA; M. J. Kaufman, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611 USA

8:30 AM

SOLUTE EFFECTS IN EXOTIC INTERMETALLICS: David P. Pope¹; Yoshisato Kimura¹; David E. Luzzi¹; Alexander Goldberg²; ¹University of Pennsylvania, Materials Science and Engineering, 3231 Walnut St., Philadelphia, PA 19104 USA; ²Cercon, Howmet-Cercast Group, 201 Cercon Dr., Hillsboro, TX 76645 USA

This talk will emphasize the effects of substitutional solutes on the mechanical properties of cubic Laves phases, with special emphasis on twinning in HfV2 alloyed with Nb. Binary HfV2 is very brittle, showing plastic flow only at quite high homologous temperatures, but alloying with Nb greatly increases the deformability at low temperatures. The deformation at low temperatures is almost totally the result of twinning in the Laves phase. Even more interesting is the fact that as the temperature increases, a region is reached where neither twinning nor dislocation slip occurs. At still higher temperatures dislocation slip is observed. In an effort to increase the deformability of this alloy, we have considered various mechanisms

that could give rise to the increased deformability. This paper will concentrate on the mechanisms we have concluded are most likely to be responsible for the ease of twinning in Nb-alloyed HfV2.

9:00 AM

SITE OCCUPANCIES AND LONG RANGE ORDERING PARAMETERS USING THE ORDERING TIE-LINE METHOD: H. L. Fraser¹; I. P. Jones²; R. Banerjee¹; J. Nan²; S. Banerjee³; R. J. Grylls¹; ¹The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; ²The University of Birmingham, School of Metallurgy and Materials, Birmingham, B15 2TT United Kingdom; ³Bhabha Atomic, Research Center, Bombay, 400 085 India

Complex ordered intermetallic alloys often have compositions which are far from those corresponding to stoichiometry and also exhibit ordering schemes which result in the alloys being not fully ordered. To understand the properties of such materials and also to be able to supply an accurate physical description of these materials to those involved in computational efforts, it is important that the site occupancies and degree of long range order be characterized accurately and precisely. A relatively new technique for determining these quantities, the ordering tie line method, will be described and then applied to Nb and Ti based ordered intermetallics. Computational efforts aimed at determining these same quantities by solution thermodynamics have been undertaken, and the computational and experimental results will be compared and contrasted.

9:30 AM

APPLICATIONS OF ALCHEMI TO ORDERED INTERMETALLIC ALLOYS: Ian M. Anderson¹; L. M. Pike¹; A. J. Duncan¹; J. Bentley¹; ¹Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831-6376 USA

The usefulness of ALCHEMI (atom location by channeling enhanced microanalysis) in the development of intermetallic alloys is best seen in the context of systematic studies, where trends in site occupancies can be correlated with other alloy properties. For example, an ALCHEMI study of 3d-transition elements (Me) from Ti to Cu Fe₃₀Al₄₅Me₅ yields site distributions that cannot be understood only in terms of site preferences but must also take into account the site-equilibration mechanism, thus demonstrating the relative importance of thermodynamic and kinetic factors in alloy processing. Another ALCHEMI study of alloys with compositions across the entire B2 phase field in the system Ni-Fe-Al suggests the role of Fe as a buffer element, while helping to explain the variations in mechanical properties with the composition of these ternary alloys. Other applications of ALCHEMI to L1₂-ordered gamma prime particles, L1₀-ordered TiAl, and C15-ordered Laves phase intermetallics will also be presented.

10:00 AM

SOLUTE EFFECTS ON DEFORMATION AND FRACTURE OF Ni₃Si: T. Takasugi¹; ¹Tohoku University, Institute for Materials Research, Katahira 2-1-1, Aoba-ku, Sendai, 980-77 Japan

Nickel silicide based on L12-type Ni₃Si is considered to be utilized as high temperature structural materials because of low density, high strength at elevated temperatures, and superior corrosion and oxidation resistance. In this talk, the effects of Ti element as substitutional, and boron, carbon and hydrogen as interstitial on the deformation and fracture behavior of Ni₃Si are reviewed, based on the results of single crystals and polycrystals. It is mentioned that Ti addition (at atomic pct) to Ni₃Si has the effect of strengthening the matrix, shifting the positive temperature dependence of the strength toward higher temperature and also improving the ambient tensile ductility via enhancing intergranular cohesion. Absorbed or residual hydrogen significantly reduces the intergranular cohesion, resulting in the intergranular fracture. On the other hand, the doping of boron and carbon (in ppm level) to Ni₃Si has the effects of strengthening the matrix, and dramatically improving the ambient tensile ductility via preventing the intergranular fracture due to hydrogen.

10:30 AM

PROGRESS IN THE MODELING OF ORDERED INTERMETALLIC ALLOY MICROSTRUCTURES: *Guillermo Bozzolo*¹; Ronald D. Noebe²; Frank S. Honey²; ¹Ohio Aerospace Institute, 22800 Cedar Point Road, Cleveland, OH 44142 USA; ²NASA Lewis Research Center, Materials Division, 21000 Brookpark Road, Cleveland, OH 44135 USA

A detailed understanding of the structure of multicomponent systems relies heavily on the behavior of the individual component elements and their interaction with each other. Basic problems, like the precise site substitution scheme of specific alloying elements and their dependence with concentration and the number of other additions are necessary in order to predict and understand the effect of various alloying schemes on the physical properties of a material, its response to various temperature treatments, and the resulting mechanical properties. It is only recently that theoretical methods can provide useful insight in this area, as most current techniques suffer from strong limitations mainly in the type of elements considered as well as the crystallographic structure of the phases that form. The Bozzolo-Ferrante-Smith (BFS) method for alloys was designed to overcome these limitations, with the intent of providing a useful tool for the theoretical prediction of fundamental properties and structure of such complex systems. After a brief description of the method and its range of applications, we concentrate on the use of BFS for the determination of site substitution schemes for individual as well as collective alloying additions to intermetallic systems, mainly NiAl, as well as the resulting behavior with respect to solubility limits and second phase formation, and the concentration dependence of the lattice parameter. In addition, we show the results of large scale numerical simulations based on BFS for calculation of the energetics of these systems, providing useful insight on the temperature dependence of these properties. Attention is also given to the AB₂A₃BC two phase alloys and the influence of alloying additions on the crystallographic misfit between these phases, including results on their partitioning behavior.

11:00 AM

STRAIN RATE SENSITIVITY OF Cr-STABILIZED CUBIC TITANIUM TRIALUMINIDES: *Steven J. Miller*¹; Thomas D. Wood¹; Donald E. Mikkola¹; ¹Michigan Technological University, Department of Metallurgical and Materials Engineering, Houghton, MI 49931-1295 USA

Negative strain rate sensitivities and serrated flow have been commonly observed with the various titanium trialuminides in the range 400-700K. These effects have usually been attributed to dynamic strain aging, but no specific solutes have been identified. In this study, the effects of selected site substitutions of solutes on the strain rate sensitivity of a Cr-stabilized trialuminide have cast doubt on the dynamic strain aging mechanism. Detailed studies of the mechanical properties of several trialuminides as a function of temperature and strain rate will be presented, and then compared to the behavior of other phases with the same structure that have been tested in the same manner. Alternate means for understanding the occurrence of negative strain rate sensitivities with only certain of these alloys will be discussed. The support of NSF (DMR-9400507) is gratefully acknowledged.

11:30 AM

EFFECTS OF TERNARY ALLOYING ON THE STRENGTH AND DUCTILITY OF Al₂Ti INTERMETALLIC COMPOUND: *J. C. Ma*¹; M. J. Lukitsch¹; J. E. Benci¹; ¹Wayne State University, Materials Science and Engineering, Detroit, MI 48202 USA

As-cast ternary-alloyed Al₂Ti + X compositions were produced by repeated arc melting of the appropriate masses of high purity Ti, Al and one of the following ternary elements: Si, V, Cr, Mn, Fe, Ni, Cu, Mo or W. Each alloy nominally contains 2 at.% of the ternary element while the Al:Ti ratio was maintained at 2 to 1. The microstructures were characterized by SEM/EDS. The compressive yield strength of each alloy was between 300YC and 1000YC. The compressive plastic strain-to-failure was also measured for each composition

at room temperature and 500YC. All ternary alloying elements studied except V increase the 300YC yield strength of Al₂Ti. Si has the greatest strengthening effect at this temperature. All ternary alloys with the exception of the Si-containing alloy have lower strengths at 1000YC than binary Al₂Ti. The transition from higher strength at lower temperatures to lower strength at higher temperatures generally occurs around 800YC. Binary Al₂Ti exhibits 0.2% plastic strain-to-failure at room temperature. Cr-, Si- and W-alloyed Al₂Ti exhibit 0.4 to 0.6% plastic strains-to-failure while Fe-alloyed Al₂Ti fails after accumulating 1.0% plastic strain. Three of the other ternary alloys exhibit no measurable plastic strain-to-failure at room temperature while two show only marginally improved or the same strain-to-failure as the binary alloy. At 500YC, binary Al₂Ti exhibits 1.5% plastic strain-to-failure. With the exception of Mn, all of the ternary additions increase the plastic strain-to-failure at this temperature compared to the binary alloy. The Fe-containing alloy exhibits 10% plastic strain-to-failure at 500YC, the most of any of the ternary alloys studied.

MECHANICAL PERFORMANCE OF LAMINATED COMPOSITES: Structure-Property Correlations

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: Donald Lesuer, Lawrence Livermore Natl. Lab., Livermore, CA 94551 USA; Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712

Tuesday AM

October 13, 1998

Room: Paris C

Location: O'Hare Hilton Hotel

Session Chairs: Eric Taleff, University of Texas at Austin, Dept. of Aerospace Engineering and Engineering Mechanics, Austin, TX 78712 USA; Desiderio Kovar, University of Texas at Austin, Department of Mechanical Engineering, Austin, TX 78712 USA

8:30 AM INVITED PAPER

INFLUENCE OF INTERFACIAL FRACTURE RESISTANCE ON THE MECHANICAL BEHAVIOUR OF LAYERED CERAMICS: *Desiderio Kovar*¹; M. D. Thouless²; ¹University of Texas at Austin, Department of Mechanical Engineering, Texas Materials Institute, Austin, TX 78712 USA; ²University of Michigan, Mechanical Engineering and Applied Mechanics Department, Ann Arbor, MI 48109-2136 USA

Recently it has been shown that high strength, high energy absorption, and graceful failure can be achieved using low-cost layered ceramics with weak interfaces. In this study, multilayered ceramic composites consisting of layers of Si₃N₄ separated by a weak interphase containing BN+Si₃N₄ were prepared and tested. The composition of the interphase was varied to alter the interfacial fracture resistance. Measurements revealed that the interfacial fracture resistance could be varied from 30 J/m² to greater than 90 J/m² by the addition of Si₃N₄ to the interphase. The influence of interfacial fracture resistance on mechanical behavior was determined using flexural tests. It was found that energy absorption was maximized at low values of interfacial fracture resistance where widespread delamination cracking was observed within the weak interphase.

9:00 AM

IMPACT OF INTERFACIAL ELECTRONIC BAND STRUCTURE EFFECTS ON MULTILAYER STABILITY: *T. W. Barbee*¹; ¹Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

TUESDAY AM

It has been experimentally observed that copper wets many transition metals and transition metal compounds. This wetting is unexpected as the copper - transition metal binary alloys exhibit terminal solid solubilities and miscibility gaps in most cases. It has recently been experimentally observed that there is an electron transfer at the interfaces in copper - transition metal multilayer nano-laminates that results in a decrease in the d band occupancy of the copper at the interfaces and an increase in the d band occupancy of the transition metals at the interfaces. Calculations indicate that the first two monolayers in each material at an interface are involved in the electron transfer. This electron transfer results in the copper being more strongly bonded to the transition metal than to itself, an effect that may represent an increase in the bonding energy of 25 % of that characteristic of copper on copper without alloying. In this study the thermal stability of copper transition metal multilayers has been studied. Specific results for multilayers of copper-niobium, copper-vanadium, copper-vanadium carbide and copper-chromium carbide are presented and discussed in terms of the interfacial electron transfer and its effect on interface stability.

9:20 AM

MECHANICAL PROPERTIES/MICROSTRUCTURE RELATIONSHIP IN ELECTRODEPOSITED Cu/Ag LAMINATED NANOCOMPOSITES: *Qing Zhai*¹; Fereshteh Ebrahimi¹; ¹University of Florida, Dept. of Materials Science & Engineering, Gainesville, FL 32611-6400 USA

It has been widely reported that the laminated composites made up of two "soft" metals may display very high strength when the microstructural scale is in the nanometer range. The Cu-Ag laminated nano-composites studied in this paper are produced by electrodeposition using a single-bath cyanide solution. The effects of the copper layer thickness and low temperature annealing on the strengthening and the fracture behavior of these composites were investigated. Tensile testing was conducted to investigate the mechanical properties of the specimens. TEM and X-ray diffraction were employed to analyze the microstructure. SEM and AFM were used to study the fracture surfaces. The mechanical property-microstructure correlation and the explanation of the fracture behavior of these composites are presented.

9:40 AM

POTENTIAL OF MULTILAYER NANO-LAMINATES FOR TRIBOLOGICAL APPLICATIONS: *Tai D. Nguyen*¹; Troy W. Barbee¹; ¹Lawrence Livermore National Laboratory, Chemistry and Materials Science Department, 1L-350, Livermore, CA 94551 USA

Nano-laminate multilayers exhibit enhanced mechanical properties and thus are potential candidates for wear resistant coating applications. In this experiment, we study the mechanical and tribological properties of multilayer nano-laminates having bilayer thickness ranging from two to hundreds of nanometers prepared by magnetron sputtering. The microstructure was characterized using x-ray diffraction and transmission electron microscopy techniques. The effects of thermal annealing on the structure stability and performance are reported. Tribological results obtained from a pin-on-disc geometry apparatus, and residual stress and hardness are presented for both as-deposited and annealed samples. Correlations between the microstructure and mechanical and tribological performance of the multilayer laminates are discussed.

10:00 AM BREAK

10:20 AM

Nb/Nb₅Si₃ MICROLAMINATES FOR HIGH-TEMPERATURE STRUCTURAL APPLICATIONS: A. J. Gavens¹; D. Van Heerden¹; C. H. Shang¹; T. Foecke²; *T. P. Weihs*¹; ¹The Johns Hopkins University, Materials Science & Engineering Department, Baltimore, MD 21218 USA; ²National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

Microlaminate foils with alternating layers of Nb and Nb₅Si₃ hold considerable promise as a new structural material for high-temperature aerospace applications. The Nb layers enhance fracture toughness at low temperatures, the Nb₅Si₃ layers provide creep strength at elevated temperatures, and both phases are chemically stable to temperatures above 1600°C. In this paper we describe mechanical characterizations of Nb/Nb₅Si₃ microlaminate foils that were sputter deposited near room temperature and then thermally processed at 1200°C. The free-standing foils are 50µm thick and contain many Nb and Nb₅Si₃ layers that individually measure 0.2µm, 1.0µm, or 5.0µm in thickness. The Nb phase is polycrystalline and equiaxed with a strong {110} texture while the Nb₅Si₃ phase is polycrystalline and equiaxed but random in orientation. The room temperature mechanical properties of the microlaminates were examined by performing tension tests on free-standing specimens. The measured fracture strengths range from 500 to 1000 MPa, and the fracture surfaces show clear signs of ductility within the Nb layers and brittle fracture within the silicide layers. Residual thermal stresses were also quantified for the specimens using asymmetric X-ray diffraction experiments. The results demonstrate that large (>100 MPa) residual stresses exist in the as-processed foils with the silicide layers being in residual compression. Variations in strength and ductility will be reported as a function of residual stress, layer thickness, and volume fraction of Nb.

10:40 AM

INFLUENCE OF PROCESSING VARIABLES ON MECHANICAL PROPERTIES OF 6090/SiC/25P//6013 LAMINATES: *C. K. Syn*¹; D. R. Lesuer¹; J. D. Rigney²; R. W. Bush³; ¹Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; ²General Electric Aircraft Engines, M/D M85, 1 Newman Way, Cincinnati, OH 45215-6301 USA; ³Alcoa Technical Center, Aluminum Co. of America, 100 Technical Drive, Alcoa Center, PA 15069 USA

The influence of bonding process and subsequent heat treatment on the mechanical properties of Al 6090/SiC/25p // Al 6013 laminates has been studied. A key processing variable in this study was the bonding time which was varied from 2 seconds to 2 hours. The variations in processing had significant influence on the interfaces between layers and the tendency for local delamination during subsequent testing. The mechanical properties were characterized by measuring fracture toughness in the crack arrester orientation (using straight notched three-point bend bar specimens); fracture toughness in the direction of the interfaces (using chevron notched short bar specimens); tensile properties in the parallel and normal directions to the constituent layers; and interface shear strength using lap-shear type of tests. It was shown that the bonding time was less significant than the heat treatment in affecting the tensile strength, toughness, and interfacial characteristics. Processing details and inter-relationships between the measured properties and failure behavior will be presented.

11:00 AM

PROCESSING AND CHARACTERIZATION OF MICROSCALE Ni-Nb MULTILAYER COMPOSITES PRODUCED BY COLD ROLLING: *F. H. Kavaranan*¹; K. S. Ravichandran¹; ¹University of Utah, Department of Metallurgical Engineering, 412 WBB, Salt Lake City, UT 84112 USA

An attempt was made to fabricate microscale Ni-Nb multilayer composites by diffusion bonding of alternately stacked high purity sheets followed by cold rolling. The general deformation behavior of these multilayers and the evolution of microstructure and hardness were studied. Hardness measurements were made as a function of nominal reduction during rolling. X-ray diffraction was used to study the possibility of formation of intermetallic compounds after heavy reduction. The development of {110} Ni preferred orientation and {100} Nb preferred orientation was observed in the multilayers. As the thickness of the laminate decreased, the microhardness of the laminate increased. For average thicknesses below 2 mm, a strong increase in microhardness was observed. Microstructural features and strengthening due to bilayer refinement were analyzed.

11:20 AM

PROCESSING, STRUCTURE AND PROPERTIES OF ALUMINUM-ALUMINIDE LAYERED COMPOSITES: *D. E. Alman*¹; ¹Albany Research Center, U.S. Department of Energy, 1450 Queen Avenue, S. W. Albany, OR 97321 USA

Laminate composites comprising of alternating aluminum and aluminide layers were produced by reactive bonding Al foils with either Ni, or Mg foils. Al foils (0.250 mm thick) were bonded to the thinner Ni foils (e.g., 0.025mm thick) by hot-pressing at 600°C and 20 MPa for 120 minutes. This produced a composite microstructure consisting of alternating Al and NiAl layers, with a variety of phases at the interface. This structure could be driven to equilibrium (e.g., alternating Al and Al₃Ni layers) by subsequent vacuum heat-treatment. For Mg-Al laminates, hot-pressing was performed at 400°C for 120 minutes. This produced a composite consisting of alternating Al, Mg₂Al₃, Mg₁₇Al₁₂, and Mg layers. Presented will be the microstructure, as well as, the tensile properties of these composite.

11:40 AM

DAMPING IN LAMINATED METAL COMPOSITES: *Donald R. Lesuer*¹; *Brian Bonner*¹; *Chol K. Syn*¹; *Albert E. Brown*¹; ¹Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

Laminated metal composites (LMCs) can improve the damping response of structural materials through the activation of damping mechanisms associated with planar interfaces and abrupt changes in elastic constants on going from one layer to another. These damping mechanisms are additive to the ones active in the individual layers from which the LMC is composed. In many cases, the LMC can have higher damping capacity than the component materials. In this presentation we will describe damping studies conducted at low frequencies (2-40 Hz) and ultrasonic frequencies (2-30 MHz). Results at low frequency have been obtained on ultrahigh carbon steel (UHCS) / brass and Ti-6Al-4V / Al-Si-Fe and results at high frequency have been obtained on Al / Al-SiC, UHCS / brass, Mg-9%Li / Al-SiC and UHCS / Ti-6Al-4V. The influence of layer thickness and frequency have been studied. Possible mechanisms of damping in LMCs will be discussed.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Fiber Reinforced Composites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: *Bhaskar S. Majumdar*, UES, Inc., Dayton, OH 45432-1894 USA; *S. Krishnamurthy*, Allied Signal, Inc. Phoenix AZ 85034 USA; *Daniel B. Miracle*, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Tuesday AM Room: Paris A
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: *Daniel B. Miracle*, Air Force Research Laboratories, Wright Patterson Air Force Base, OH USA

8:30 AM INVITED PAPER

HIGH ENERGY ABSORPTION COMPOSITES AND THE LOCALIZATION TO DELOCALIZATION TRANSITION: *N. Sridhar*¹; *B. N. Cox*¹; *J. B. Davis*¹; *F. W. Zok*²; ¹Rockwell Science Center, Thousand Oaks, CA 91358 USA; ²University of California, Materials Department, Santa Barbara, CA 93106 USA

We have developed novel composites possessing an exceptional capacity for high energy absorption. The high energy absorption is achieved by designing a reinforcement topology that has a large den-

sity of sites for damage initiation. This promotes "lock-up" leading to a change in damage from a localized to a delocalized mode of failure. The composite hardens over large strains (~100%) following the onset of damage. We demonstrate this concept in model composite systems consisting of an epoxy matrix and steel chains. Models for the deformation response as a function of the geometrical and material parameters are developed and with the aid of these results, the composite architecture is optimised.

8:55 AM

ON THE DETERMINATION OF FIBER FAILURE IN CONTINUOUSLY REINFORCED TITANIUM MATRIX COMPOSITES DURING ELEVATED TEMPERATURE TESTING EMPLOYING MODAL ACOUSTIC EMISSION: *A. H. Rosenberger*¹; *D. A. Stubbs*²; *N. E. Ashbaugh*¹; *P. M. Schubel*²; *D. J. Buchanan*²; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson AFB, OH 45433-7817 USA; ²University of Dayton Research Institute, 1031 Irving Avenue, Dayton, OH 45419-0218 USA

Fiber failure is the dominant damage mechanism during high temperature application of titanium matrix composites that incorporates periods of sustained loading. The accurate determination of the number of fiber failures is necessary for accurate modeling of the damage progression and determining the burst margin remaining after creep exposure. A new acoustic emission technique, incorporating broad band monitoring and modal analysis, has been applied to tests of composite samples in order to more accurately quantify fiber failures during mechanical testing at elevated temperatures. Alumina wave guides have been incorporated to allow monitoring at the elevated temperatures while maintaining the fidelity of the acoustic emission event. This technique has been applied to a unidirectional continuously reinforced titanium matrix composite, SCS-6/Ti-6Al-4V. Characteristics of the fiber break signature are discussed and the method is verified using an interrupted test in which the actual number of fiber failures is compared to the modal acoustic emission results.

9:20 AM

IN SITU OBSERVATIONS AND ACOUSTIC EMISSION ANALYSIS FOR SIGMA 1140+/Ti=6-4 SiC FIBRE REINFORCED TITANIUM COMPOSITES UNDER TENSILE TRANSVERSE LOADING: *X. Wu*¹; *H. Mori*²; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials, Birmingham B15 2TT UK; ²The University of Tokyo, Research Centre for Advanced Science and Technology, 4-6-1 Komaba, Meguro-ku, Tokyo 153 Japan

The transverse response of 8 and 21% volume fraction of Sigma 1140+ SiC fibre reinforced Ti-6-4 matrix composites has been investigated in situ in a FEG-SEM using direct observation. Damage has also been monitored in air using conventional transverse tensile tests and Acoustic Emission (AE). An advanced acoustic emission system has been used to locate the position of damage and to collect AE events during such conventional tests. In situ observations have found that debonding of matrix/fibre interfaces in such a weakly debonded type of composite starts at a very low stress and does not produce a distinct knee on a conventional stress-strain curve. The first knee observed on the stress-strain curve actually corresponds to cracking of carbon-coating layers, rather than fibre/matrix interface debonding which has been previously assumed, and the second knee corresponds to local micro-cracking of the matrix near interfaces. Many AE events were recorded during cracking of the carbon-coating and were of amplitude of up to 90 dB and of energy of up to 160 mJ. Near to the end of the test, AE signals were again received in large numbers but now with an amplitude up to 70 dB and with an energy of less than 20 mJ. This combination of in situ observations and acoustic emission analysis has allowed the development of damage, as a function of volume fraction of fibres, to be quantified.

9:45 AM INVITED PAPER

STRENGTH AND FAILURE BEHAVIOR OF STITCHED CARBON/EPOXY COMPOSITES: *Ryuta Kamiya*¹; *Tsu-Wei Chou*¹; ¹Uni-

versity of Delaware, Center for Composite Materials and Department of Mechanical Engineering, Newark, DE 19716 USA

This paper presents a study of the effect of through-the-thickness stitching fibers upon the strength and failure behavior of multi-directionally reinforced composites. The woven fabric laminae were placed in four direction, (1, +45, 90), to form a quasi-isotropic preform. The fabrics have open space between adjacent tows. These inter-yarn spaces allow easy insertion of the through-the-thickness stitching fibers without seriously damaging the in-phase fibers. Fiber volume fractions over 58% were obtained by this method. The through-the-thickness yarn sizes used in this project were 2k, 4k, and 6k. Non-stitched preforms were also designed and manufactured with the same fiber and by the same procedure for the control experiments. All preforms were infiltrated with epoxy resin by the RTM technique. In-plane tensile and compressive strength, interlaminar shear strength, and Mode I fracture toughness have been measured at three through-the-thickness fiber contents for the carbon/epoxy composites. Although the through-the-thickness stitching fibers significantly enhance the Mode I fracture toughness, they tend to degrade the in-plane tensile and compressive strength. The failure process under interlaminar shear loading the DNS tests show two distinct stages: the fiber-matrix interfacial failure followed by the breakage/debonding of the through-the-thickness fibers. The through-the-thickness fibers cause a reduction of the initial failure load in the first stage, but can enhance the final failure load in the second stage. In composites with 6k through-the-thickness fibers, the final failure load can exceed the initial failure load. SEM and optical microscopic examinations have also been conducted for observing the failure mechanisms and fracture surfaces.

10:10 AM INVITED PAPER

NOTCH-SENSITIVITY OF FIBER-REINFORCED CERAMIC MATRIX COMPOSITES: EFFECTS OF INELASTIC STRAINING AND VOLUME-DEPENDENT STRENGTH: *F. W. Zok*¹; *J. C. McNulty*¹; *G. M. Genin*²; *A. G. Evans*³; ¹University of California, Materials Department, Santa Barbara, CA 93106 USA; ²Harvard University, Division of Applied Science, Cambridge, MA 02138 USA; ³Cambridge University, Currently with Engineering Department, Cambridge, England CB2 1PZ USA

The effects of circular holes and sharp notches on the tensile strength of several fiber-reinforced ceramic composites have been investigated. The role of inelastic straining in the local re-distribution of stress has been elucidated through measurements of the local strains in the regions of high stress concentration, coupled with finite element simulations of the test geometries, using a nonlinear constitutive law appropriate to CMCs. The scale-dependence of strength has been inferred from tests performed on specimens of varying size. The utility of two failure models that incorporate both the inelastic straining and the scale-dependence have been assessed: one based on the point stress failure criterion and the other on weakest link fracture statistics. Both approaches provide a reasonably consistent description of the experimental measurements. Some of the implications and limitations associated with the failure models will be discussed.

10:35 AM BREAK

10:50 AM INVITED PAPER

INITIATION OF TRAVERSE FRACTURE IN COMPOSITES FROM NONUNIFORM MICROSTRUCTURES: *Ramesh Talreja*¹; ¹Georgia Institute of Technology, School of Aerospace Engineering, Atlanta, GA 30332 USA

In fiber reinforced composites subjected to mechanical loads the first event of failure at the microscopic level is commonly believed to be fiber/matrix debonding. However, in polymer matrix composites with a glassy polymer, e.g., epoxy, as a matrix, the transverse fracture may actually initiate in the matrix before the fiber/matrix bond fails. Recently, Asp et al. (1996) investigated this and proposed cavitation-induced brittle cracking of the matrix as a critical mechanism governed by the dilatation energy density. We have recently conducted a

study of damage initiation in composites by computationally simulating nonuniform fiber distributions in an actual (as-processed) ceramic matrix composite (Bulsara, et al, 1998). The present study investigates transverse failure in polymer matrix composites under remotely imposed uniaxial and biaxial mechanical loads in addition to the residual stresses caused by thermal cooldown. Several interesting features of the failure process are revealed, noteworthy being the fact that the transverse failure tends to occur in a narrow range of the overall strain.

11:15 AM

TRANSVERSE CREEP RESPONSE OF Ti-22Al-23Nb/SiC REINFORCED COMPOSITES: *S. Woodard*¹; *T. M. Pollock*¹; ¹Carnegie Mellon University, Carnegie, PA USA

SiC-reinforced intermetallic matrix composites potentially offer high stiffness and specific strength at elevated temperatures. In some applications for which these materials are targeted, the transverse creep strength could be performance limiting. Both the matrix creep deformation and the fiber / matrix interface play a significant role in the transverse response of these materials. The research presented will focus on the interdependence of inhomogeneous local matrix plasticity and fiber/matrix interface damage during transverse creep of SiC-unidirectionally reinforced Ti-22Al-23Nb intermetallic matrix composites. Correlations between the push-out response of individual fibers, their fiber spacing environment and the macroscopic response of two Ti-22Al-23Nb intermetallic matrix composites reinforced with different SiC fibers will be discussed.

11:40 AM

TEXTURE AND RESIDUAL STRAIN IN SiC/Ti-6-2-4-2 TITANIUM MATRIX COMPOSITES: *Partha Rangaswamy*¹; *M. A.M. Bourke*¹; *R. Von Dreele*¹; *R. Von Dreele*¹; *K. Bennett*¹; *J. A. Roberts*²; *N. Jayaraman*¹; ¹Los Alamos National Laboratory, Lujan Scattering Center, H 805, LANSCE 12, Los Alamos, NM 87545 USA; ²University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Residual strain and texture variations were measured in two Titanium matrix composites reinforced with Silicon Carbide fibers (Ti/SiC) of similar composition but fabricated by different processing routes. Each composite comprised a Ti-6242 a/b matrix alloy containing 35% by volume continuous SiC fibers. In one case, the matrix was produced by a plasma spray (PS) route, and the other by a wire drawing (WD) process. The PS and WD composites were reinforced with SCS-6 (SiC) and Trimarc (SiC) fibers, respectively. The texture in the monolithic and composite Ti matrices differed significantly, from approximately 1.1x random for the monolithic and composite PS matrices to 17x random in the monolithic and 6x random in the composite WD matrices. No significant differences in matrix residual strains between the composites prepared by the two procedures were noted. The Trimarc (WD) fibers recorded higher (1.3x) compressive strains than the SCS-6 (PS) fibers in all the measured directions. The plane-specific elastic moduli measured in load tests on the unreinforced matrices, showed little difference.

12:05 PM

MODELLING OF TRANSVERSE TENSILE BEHAVIOUR OF Ti MMCS: *W. Ding*¹; *J. Liu*¹; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

Ti MMCs unidirectionally reinforced by SiC fibres are anisotropic, and are often proposed to sustain load only in the direction of fibre alignment. However, even under such loading conditions, complex stress states may exist in the composite once a crack is introduced. Thus the aim of this paper is to quantify and model the transverse tensile behaviour of Ti MMCs. Both finite element (FE) modelling and analytical modelling based on a modified Eshelby method have been used. Attention is focused not only on the effects of matrix modulus and fibre volume fraction (Vf) but also on the effects of volume fraction of fibres with debonded interfaces (Vf,B), fibre pack-

ing distance, and fibre packing architecture (rectangular and hexagonal distributions). The modelling results have also been compared with experimental measurements carried out on Ti MMCs with different Vf and different fibre/matrix combinations and good agreement has been found. This gives the opportunity to design the distribution of SiC fibres so that an optimised combination of longitudinal and transverse properties can be achieved.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - I General, Texture & Heat Capacity

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee

Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

Tuesday AM Room: Rome
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, Iowa 50011-3020 USA

8:30 AM OPENING REMARKS

8:40 AM INVITED PAPER

PAUL BECK: THE YEARS AT ILLINOIS: *Charles A. Wert*¹; ¹University of Illinois, Materials Science and Engineering, 1304 West Green St., Urbana, IL 61801 USA

Study of recrystallization, grain growth and textures occupied Paul Beck in his "first career". That work was in full bloom when he came to Illinois in 1951 but continued for 20 more years, through some 100 papers. Gradually his attention shifted to electronic and magnetic properties of alloys. He and his students carefully measured the electronic density of states of metallic alloys and the ferromagnetic and paramagnetic properties of solid solutions and compounds. Defining features of this work were careful preparation of alloys, precise measurements and excellent interpretation remarkably aided by close interaction with the condensed-matter-physicists. The work evolved to study of spin glasses and the magnetic character of thin layer alloys. He formally retired in 1976, but continued working with enthusiasm for some 14 more years. For years, he organized the colloquia of the department which became popularly known as the 3d-4s series. He also led TMS into regular inclusion of symposia in alloy theory. His work won for him many awards from both TMS and ASM. He was elected to the National Academy of Engineering and received an Honorary Degree from the University of Illinois, an honor rarely given to a faculty member.

8:55 AM INVITED PAPER

A BRIEF RECAP OF TMS COMMITTEE ON ALLOY PHASES: *Michael V. Nevitt*¹; ¹Clemson University, Dept. of Physics and Astronomy, Clemson, SC 29634 USA

Paul Beck took the initiative with TMS/AIME leadership in the early 1960's to establish a locus of interest in the physics and chemistry of alloy phases, their occurrence, structures and properties. As a result, the Committee on Alloy Phases (CAP) was formed. The cross-cutting interests of CAP have continued to emphasize the theoretical and experimental properties governing the occurrence and stability of solid solutions and intermetallics. A major role of CAP

has been in sponsoring relevant TMS symposia, most notably the annual Hume-Rothery Symposium and Award. A few highlights in CAP's thirty-year history will be reviewed.

9:10 AM INVITED PAPER

RECRYSTALLIZATION TEXTURES IN METALS: *Paul Shewmon*¹; ¹Ohio State University, Materials Science and Engineering, 2477 Lytham Rd., Columbus, OH 43220 USA

Prof. Beck advocated the 'oriented growth' theory of texture and published the results of several classic experiments demonstrating the importance of the rapid growth of certain orientations in texture development. The contesting theory was 'oriented nucleation', put forth strongly by Buerger, among others. Developments in this field since Beck's last publications on the topic 40 years ago will be summarized. It will be concluded that there is still a controversy about the relative importance of oriented growth and oriented nucleation in texture development.

9:45 AM BREAK

10:00 AM INVITED PAPER

ON THE CONTRIBUTION OF ORIENTED NUCLEATION AND GROWTH SELECTION ON THE FORMATION OF RECRYSTALLIZATION TEXTURES: *Olaf Engler*¹; ¹Los Alamos National Laboratory, Center for Materials Science, Mail Stop K765, Los Alamos, NM 87545 USA

For several decades the changes in crystallographic textures during recrystallization have been explained in terms of one out of two rivaling theories, oriented nucleation and growth selection. Whereas in the case of oriented nucleation, it is assumed that the preferred formation of special orientations determines the final recrystallization texture, the theory of growth selection - which goes back to Beck et al. - assumes that, starting from a broad spectrum of nucleus orientations, those with the best growth conditions with respect to the deformed matrix grow fastest and, therefore, dominate the recrystallization texture. The present paper reviews the two theories of oriented nucleation and growth selection in the light of recent experimental investigations - in particular with the help of electron back scattering diffraction to determine the crystallographic orientations of small regions down to sub-micrometer size in an SEM - which have provided new insight into the mechanisms of nucleation and growth of recrystallization. From these results it can be concluded that the recrystallization textures of Al-alloys evolve by a preferred formation of some orientations at characteristic nucleation sites and a subsequent growth selection of distinct orientations out of this spectrum of nucleus orientations.

10:35 AM

TEXTURE EVOLUTION IN RECRYSTALLIZED HAFNIUM AND TITANIUM: *Rong Bai*¹; Clyde L. Briant¹; ¹Brown University, Division of Engineering, 182 Hope Street, Providence, RI 02912 USA

This paper presents a study of the evolution of annealing texture in hafnium and titanium. The texture was measured by the analysis of electron back scattering diffraction patterns in the scanning electron microscope. It was found that in hafnium the texture changed both as a function of annealing temperature and cold work. After low temperature recrystallization, the deformation axis was parallel to [11-20] or [-12-10]. After annealing at high temperatures the axis parallel to the deformation axis as [01-10]. When the samples were rolled and recrystallized a texture was developed in which the rolling plane was parallel to the basal planes of the grains. These results on hafnium will be compared with those on titanium. This work was supported by the NSF MRG No. DMR-9223683 and the NSF-Sponsored MRSEC at Brown University.

11:10 AM INVITED PAPER

LOW TEMPERATURE HEAT CAPACITY IN MATERIALS RESEARCH: *James C. Ho*¹; ¹Wichita State University, Department of Physics and National Institute for Aviation Research, 1845 Fairmount, Wichita, KS 67260 USA

Based on statistical thermodynamics, heat capacity is a most fundamental property of materials. Low temperature calorimetry has played significant roles in the development of condensed matter physics. The results can be used to help characterize and analyze a given material in terms of lattice vibration, electronic density of states, magnetic behavior and phase transitions, if any. Examples of actual applications of low temperature heat capacity to materials research will be given here. Among others, they include the systematics in transition metal alloys, microstructural effect in titanium-base superconductors, magnetic clusters in paramagnetic matrix, magnetic ordering in nano-sized ferrites, and conducting polymers.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Ceramics and Ceramic Matrix Composites

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Tuesday AM Room: Chicago
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Dr. G. R. Edwards, Colorado School of Mines, Department of Metallurgical Engineering, Golden, CO USA; Dr. Mohan Edirisinghe, Loughborough University, Institute of Polymer Technology and Materials Engineering, Loughborough, Leicestershire LE 11 3TU United Kingdom

8:30 AM INVITED PAPER

SPONTANEOUS COPPER INFILTRATION OF ALUMINA BY VAPOR-STATE OXYGEN ALLOYING: *Daniel A. Javernick*¹; Glen R. Edwards¹; ¹Colorado School of Mines, Department of Metallurgical Engineering, Golden, CO USA

Additions of oxygen to copper have been shown to promote spontaneous infiltration of porous alumina bodies. This processing technique, termed the "carrier oxide" approach, is dependent upon the formation of an immiscible liquid oxide which reacts aggressively with alumina to form CuAlO_2 at the metal-ceramic interface. The relative importance of the interfacial reaction product, CuAlO_2 , and the oxygen activity in the molten copper on the processing of these materials have been investigated by analyzing the initial contact of molten copper droplets of varying oxygen content on CuAlO_2 and Al_2O_3 surfaces. These sessile drop measurements will help determine the optimal vapor-state oxygen concentration required to infiltrate porous alumina performs without depositing brittle copper oxides into the metal matrix.

9:00 AM INVITED PAPER

MULLITE-ZIRCONIA COMPOSITES PREPARED BY PLASMA SPRAYING OF ZIRCON AND ALUMINA MIXTURES: *K. A. Khor*¹; *Y. Li*¹; ¹Nanyang Technological University, School of Mechanical & Production Engineering, Nanyang Avenue 639798 Singapore

Reaction sintering of Zircon and Alumina is an easy and inexpensive route to obtain homogeneous mullite-zirconia ceramics with enhanced mechanical properties. This paper presents the preparation of ZrO_2 reinforced mullite by plasma spraying a mixture of zircon and alumina. The dissociation of zircon into zirconia and silica in a plasma flame is well-known phenomenon. Pre-mixed powders of zircon and alumina are injected into a dc plasma jet. The plasma sprayed particles are collected in distilled water and analyzed. The results indicate that the plasma sprayed powders consist of zirconia, zircon and alumina. It was found that fine grained, even amorphous and chemically homogeneous composite powders can be obtained by ball milling and plasma spraying. Recrystallization of amorphous phases and formation of mullite occurred at about 1000°C in plasma sprayed powders. This value is more than 500°C lower than the formation of mullite in as-milled powders. The effect of power and secondary gas pressure on the spheroidization of zircon and alumina mixtures was investigated. The results showed the both of them were of importance in controlling the morphology and phase composition of the spheroidized powders.

9:30 AM INVITED PAPER

EFFECT OF HEAT TREATMENT ON THERMAL CONDUCTIVITY AND MICROSTRUCTURE OF ALUMINUM NITRIDE: *Y. Baik*¹; *M. Entezaran*¹; *H. Vali*¹; *R. A. L. Drew*¹; ¹McGill University, 3610 University Street, Montreal H3A 2B2 Canada

Aluminum nitride (AlN) ceramics are in high demand for applications involving substrate materials for high speed, high capacity, and complex integrated circuits requiring high thermal conductivity. However, there is a challenge in obtaining high thermal conductivity values for polycrystalline AlN substrates compared to that of single crystal AlN which has a thermal conductivity of 320 W/m K. A study of the post-sintering heat-treatment of AlN in various reducing atmospheres was performed to determine the influence on thermal conductivity. It was found that the composition of the secondary phase and temperature of the heat treatment are the most influential factors controlling the thermal conductivity values. Two different mechanisms for the reducing reactions were proposed. The results indicate that such a heat treatment can improve the thermal conductivity of AlN up to nearly 200 W/mK. This is due further oxygen removal from both the AlN lattice and secondary grain boundary phase, simultaneously.

10:10 AM INVITED PAPER

BORON SUBOXIDE SYNTHESIS, CONSOLIDATION AND SUBSEQUENT METAL INFILTRATION: *Oguz Kayhan*¹; *Osman T. Inal*¹; ¹New Mexico Tech, Materials and Metallurgical Engineering Department, Socorro, NM 87801 USA

The intent of the program was to produce metal (aluminum) infiltrated boron suboxide ceramic drag cutters and drill bits that have improved hardness and fracture toughness values over those of commercially available ceramic Cutters. Synthesis of boron suboxide was performed by reactive sintering of crystalline boron and zinc oxide powders at 1450°C for 12 hours in an argon atmosphere. After sintering, Vickers microhardness testing was performed on randomly selected, synthesized samples and an average hardness value of 37 GPa was obtained. For further characterization, optical microscopy, SEM, and XRD analyses were performed on the powder material synthesized. After characterization, consolidation of the powder was performed. Two different routes of consolidation were carried out, explosive consolidation and hot pressing. Through both routes, satisfactory levels of compaction were obtained. Following consolidation, the compacted bodies were given a high temperature sintering treatment for full densification, at 1800°C in vacuum with no pressure applied. Aluminum infiltration was performed into these sintered bodies, in an argon atmosphere, for 10 hours, in the temperature range 1100-1250°C, again with no pressure applied. During infiltration, optimization of the infiltration temperature was also achieved. After every step (consolidation, densification sintering and infiltration steps), the samples were characterized by XRD, SEM, Vickers microhardness and density tests. Three-point bend tests were

also performed on all samples, in order to determine the subsequent fracture toughness and flexural strength values of boron suboxide after each step. The characterized infiltrated samples were finally tested at Sandia National laboratory for penetration and drag force evaluation. The presentation will detail our efforts.

10:40 AM INVITED PAPER

SOLID FREEFORM FABRICATION OF CERAMICS: *M. J. Edirisinghe*¹; ¹Loughborough University, Institute of Polymer Technology and Materials Engineering, Loughborough, Leicestershire LE11 3TU United Kingdom

Solid freeform fabrication (SFF) of ceramics has generated a great deal of interest as it offers highly automated, rapid desktop forming of small ceramic parts without molds. In SFF, 3-D computer images of parts generated with CAD software is sliced to produce 2D images and then used to control a fabrication unit which manufacturers it layer by layer. SFF methods applicable to ceramics will be briefly reviewed. The presentation will focus on continuous ink-jet printing which is a novel SFF method where a ceramic suspension is pressurized and delivered through a fine nozzle and deposited on a substrate according to a pattern generated by a computer. Research into this technique will be elucidated. The development of ceramic suspension for this process, i.e., materials and properties required will be analyzed. The use of suspensions to print monolayers and multilayers of ceramic will be illustrated with several examples. The future potential of the process to micro-engineer components while tailoring the microstructure will be elucidated.

11:10 AM INVITED PAPER

FABRICATION AND PROPERTIES OF DENSELY SINTERED TITANIUM DIBORIDE CERAMICS: *Jun-ichi Matsushita*¹; ¹Tokai University, 1117 Kitakaname, Hiratsuka 259-1292 Japan

Fabrication and properties of pressureless sintered TiB₂ containing metal and carbon for sintering aids were investigated. The powders were blended in the required ratio and wet-mixed in ethyl alcohol for 24 h using a plastic container and nylon balls. After drying, the mixture was formed with a press under 30MPa, and finally formed under CIP at 300 MPa. Pressureless sintering was conducted at 1673 to 2173K for 1 h in an argon atmosphere. Cr-C and Ni-C play an important role in the densification of TiB₂. Sintered TiB₂ containing about 7 wt% Cr(Ni) and 1 wt% C exhibited a relative density of 99(99)%, bending strength of 400(450) MPa and Vickers hardness of 22(17) GPa. The microstructure of TiB₂ sintered containing Cr(Ni) and C was found to retard the grain growth and the fracture surface of sintered body showed both transgranular and intergranular fracture modes. XRD analysis detected CrB(Ni₃B) and TiC phases besides in addition to TiB₂ in the sintered body.

11:40 AM

CERAMIC-INTERMETALLIC COMPOSITES PRODUCED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING: *J. G. Cabanas-Moreno*¹; *G. Garcia-Pacheco*¹; *O. Delgado-Gutierrez*¹; *R. Marrtinez-Sanchez*¹; *M. Umemoto*²; ¹Instituto Politecnico Nacional, ESIQIE, Apdo. Postal 75-373, Mexico D.F. 07300 Mexico; ²Toyohashi University of Technology, Tempakucho, Toyohashi 441 Japan

Nano- and micro-composites of intermetallic (Co₃Ti, AlCo₂Ti) and ceramic (Ti(C₁N), Al₂O₃) phases have been produced by spark plasma sintering (SPS) of powders resulting from the mechanical alloying of Al-Co-Ti elemental powder mixtures under argon/air atmospheres. The mechanically alloyed powders consisted of mixtures of nanocrystalline and amorphous phases which, during sintering, transformed into complex dispersions of the above mentioned phases. For Al contents lower than about 30 at % in the original powder mixtures, the use of SPS to densify the alloyed powders resulted in compacts of low porosity (1 - 2 %) and nano- or microcrystalline structures, showing microhardness values as high as ~1700kg/mm²; in these cases, the matrix of the composites was mainly formed by the Co₃Ti intermetallic phase, although the largest volume fraction cor-

responded to the Ti(C₁N) phase. The AlCo₂Ti and Al₂O₃ phases were both present in small volume fractions. Details of the processing and microstructural characterization of sintered compacts, as well as the results of attempts to characterize their fracture behavior, will be also presented. Work supported by CONACYT and IPN in Mexico and through an AIEJ scholarship in Japan.

12:00 PM INVITED PAPER

A NEW UNIDIRECTIONALLY SOLIDIFIED CERAMIC EUTECTIC WITH HIGH STRENGTH AT HIGH TEMPERATURES: *Yoshiharu Waku*¹; ¹UBE Industries, Ltd., Ube Research Laboratory, 1978-5 Kogushi, Ube City, Yamaguchi Prefecture Japan

New unidirectionally solidified eutectic composites such as Al₂O₃/Er₃Al₅O₁₂ or GdAlO₃ has recently been fabricated by controlling accurately the unidirectional solidification. The eutectic composite has a new microstructure, in which continuous networks of single-crystal Al₂O₃ and single crystal oxide compounds (Er₃Al₅O₁₂ or GdAlO₃) interpenetrate without grain boundaries. The eutectic composite fabricated is thermally stable and has the following properties: 1) the flexural strength at room temperature can be maintained up to just below the melting point of about 2040-2150 K, 2) the compression creep strength at 1873°K and a strain rate of 10⁻⁴/sec is about 7 times higher than that of sintered composites of the same composition (in case of Al₂O₃/Er₃Al₅O₁₂), 3) it shows neither weight gain or grain growth even upto heating at 1973 K in air atmosphere for 500 hours (case of Al₂O₃/Er₃Al₅O₁₂), 4) it shows substantial plastic deformation at 1873°K with a flexural yield stress of about 690 MPa and it is found that the plastic deformation occurred by dislocation motion in each phase (in case of Al₂O₃/GdAlO₃).

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CONTROLLED COMBUSTION SYNTHESIS IN THE TiH₂-B SYSTEM FOR PRODUCTION OF TiB-TiB₂-Ti CMC'S: *Sedat Ozbilen*¹; ¹Gazi University, Metallurgy Education Department, Education Department, Teknik Okullar, Ankara Turkey

Controlled Combustion Synthesis (CS) was carried out in the TiH₂-B system. Samples with different TiH₂ levels (sample #1: 82wt%TiH₂-18wt%B, sample #2:41wt%Ti-18wt%B, sample #3: 82wt%Ti-18wt%B) were studied to investigate the influence of the surface condition of the diluents (Ti in this study) on the level of exothermicity of the self-propagating reactions. Homogeneously mixed and then compressed pellets of samples #1-#3 (green compacts) were combustion synthesized in controlled fashion and in thermo explosion mode under vacuum. Thermal analysis under vacuum by DTA was carried out on the green compacts. SRD and SEM investigation were used for the examination of pressed and as-reacted pellets. It was observed that the exothermicity of the CS reactions can be increased when the amount of the activated, fresh surfaces of diluent Ti created by TiH₂ decomposition during heating of the samples under vacuum is increased as in sample #1 [having the highest TiH₂ content (82wt%) among the samples studied] thus promoting better kinetics conditions (i.e., faster CS reactions) for chemical reactions.

12:50 PM

MODIFIED OXALATE METHOD FOR THE SYNTHESIS OF PURE BARIUM TITANATE CERAMICS: *R. Ramanathan*¹; *V. Kamaraj*¹; ¹Anna University, Division of Ceramic Technology, Department of Chemical Engineering, Chennai 600 025 India

Barium titanate is an important electroceramic material with wide industrial applications such as capacitors and positive thermal coefficient thermistors. Since its discovery in 1943, processing of barium titanate has been an active field of research. In this paper a simple modified oxalate method is adopted for the preparation of barium titanate. Barium chloride dihydrate and titanium tetrachloride are used as starting materials. The solutions of the starting materials are mixed with the oxalic acid solution. After continuous stirring the solution is allowed to precipitate. The precipitate is filtered and then dried at 70°C. The dried powders are calcined at 1000°C and sintered

at 1300°C. The powder characterization studies like TGA, DTA, and SRD are done.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Processing

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Tuesday AM Room: London
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: T. E. Mitchell, Los Alamos National Laboratory, Center for Materials Science, Los Alamos, NM 87545 USA; Rajiv Mishra, University of California, Department of Chemical Engineering and Materials Science, Davis, CA 95616 USA

8:30 AM OPENING REMARKS

8:35 AM KEYNOTE

A TECHNICAL COST FRAMEWORK FOR HIGH TEMPERATURE MANUFACTURING OF SMALL COMPONENTS AND DEVICES: *A. G. Evans*¹; *J. W. Hutchinson*¹; *T. J. Lu*²; ¹Harvard University, Applied Sciences Department, Cambridge, MA 02138 USA; ²University of Cambridge, Cambridge CB2 1PZ UK

One goal of manufacturing process development is the discovery of a method for processing a material into components or devices that attain explicit property and performance specifications at minimum cost. These specifications are usually established by the system level design strategy. Establishing a methodology that links these aspects in a systematic manner is elusive. One approach that provides focus and may lead to a tractable methodology embraces a technical cost framework (TCF). This framework interrelates parameters from design and manufacturing, through a structure that accepts output from both process simulations and from design calculations. It is simple and direct. The challenge is to establish a modeling environment that either calculates or measures the input parameters with acceptable certainty. Cost simulations are used to illustrate the approach and the potential for its further development. When a high temperature step is involved, it often dominates the non-material contributions to the cost. Various methods commonly used for heat treatment are examined, in order to facilitate a logical decision about the preferred approach in any new manufacturing initiative. The philosophy evolves around the expectation that the market for the products is substantial enough to allow the furnace to operate at maximum capacity, with allowance for down-time upon maintenance and repair. Moreover, the mix of product geometries and the properties are considered to be sufficiently similar that property development occurs in essentially the same way for all products, such that yields can be controlled and characterized.

9:15 AM INVITED PAPER

DUCTILE MACHINABLE TERNARY CARBIDES AND NITRIDES FOR HIGH TEMPERATURE APPLICATIONS: *M. W. Barsoum*¹; ¹Drexel University, Department of Materials Engineering, Philadelphia, PA 19104 USA

Recently we identified two classes of layered hexagonal ternary carbides and nitrides; the M_3BX_2 and M_2BX phases, where M is a transition metal, B is a B-group element and X is either C or N. Three interrelated characteristics distinguish these phases from other layered materials: i) the metallic nature of the bonding; ii) the presence of an operative slip system at room temperature and its confinement to the basal planes; and, iii) absence of "strong" primary bonds between layers. This results in compounds that are machinable, relatively soft ($H_v \sim 3-6$ GPa), and good thermal and electrical conductors ($2-15 \times 10^6 \Omega^{-1}m^{-1}$). They also possess excellent ambient and high temperature mechanical properties that are decoupled from machinability, room temperature plasticity in oriented samples, excellent damage tolerance, thermal shock, oxidation and creep resistance, among others. The technological implications of having these naturally layered materials will be discussed. Work funded by DMR division of NSF and AFOSR.

9:45 AM

JOINING OF STRUCTURAL CERAMICS USING THIN SPIN-ON LAYERS VIA BOTH MICROWAVE AND CONVENTIONAL HEATING: *E. D. Case*¹; *K. Y. Lee*¹; *J. G. Lee*¹; ¹Michigan State University, Materials Science and Mechanics Department, East Lansing, MI 48824 USA

Structural ceramics having complex geometries can be difficult to process and difficult to check for processing-induced flaws. However, both the processing and quality control of such components could potentially benefit from an efficient and reliable methodology to build up components of complex shape by joining subcomponents of simpler geometry. Using spin-on layers of 150 nm to 600 nm thick, the authors have joined alumina and alumina/zirconia composites specimens using zero or small (20 - 60 gram) dead-weight loads. (This is in contrast to other relatively high loads used in joining structural ceramics, especially in terms of the microwave joining work). This paper will discuss joining work on alumina, alumina/zirconia composites, and partially stabilized zirconia specimens. Microstructural and mechanical characterization of the join integrity will also be discussed.

10:10 AM

FUNCTIONAL CERAMIC-REFRACTORY METAL LAYERED COMPOSITES BY PLASMA SPRAY FORMING: *Daniel J. Sorelet*¹; *M. F. Besser*¹; *R. L. Terpstra*¹; *I. E. Anderson*¹; ¹Ceramist, Ames Laboratory, 107 Metals Development, Ames, IA 50011 USA

Melt processing of extremely reactive, high temperature alloys, e.g., Ti-based, has created the need for advanced refractory materials. Processes such as gas atomization require melting components, e.g., tubes and crucibles, to resist attack from superheated alloys much better than conventional graphite, zirconia, or alumina. We have found that rare earth oxides resist attack by molten Ti-based alloys. However, conventional sintered microstructures of these ceramics are too sensitive to thermal shock during the transient conditions of gas atomization. Tougher, more thermal shock resistant rare earth oxide components may be fabricated by thermal spraying. The lamellar microstructure and finely distributed porosity developed during thermal spraying appears to mitigate the cracking that is typical of dense, fine grained rare earth oxide. In addition, the incorporation of refractory metal sublayers can further enhance the toughness of the composite system. Developments on spray formed melting components for atomization will be reviewed. Department of Energy-BES support (W-7405-Eng-82) is acknowledged.

10:35 AM BREAK

10:45 AM INVITED PAPER

NOVEL PROCESSING ROUTES FOR ADVANCED FUNCTIONAL CERAMICS: *S. C. Danforth*¹; *A. Safari*¹; ¹Rutgers, The State University, Department of Ceramic and Materials Engineering, Center for Ceramic Research, Piscataway, NJ 08855-0909 USA

Advanced structural ceramics require a high degree of processing perfection in order to insure functional properties such as high fracture strength, high fracture toughness and a high Weibull modulus. It is still very costly and time consuming to develop advanced structural ceramic components for new applications, due to the costs and time associated with each design iteration. To solve this difficulty, solid freeform fabrication (SFF) processes are now under development for processing ceramics such as silicon nitride, Si_3N_4 . Solid freeform fabrication processes offer significant potential for manufacturing functional quality components, free from the constraints imposed by the external geometric complexity of the part. This paper gives a detailed description of the Fused Deposition of Ceramics (FDC) process, a SFF technique developed at Rutgers University, specifically for structural ceramic applications. In the FDC process, a high solids loading of ceramic powders are mixed with a multi-component thermoplastic binder and subsequently extruded into 1.778 mm filaments, which are then driven, via computer controlled counter rotating rollers, into a hot extruder and deposited onto a z-motion platform. The extruder moves in the x-y plane, depositing material only in locations specified by the original CAD file. This paper will detail the processing and quality control steps needed to fabricate high quality FDC filament feedstock using 55 vol. % of AlliedSignal's GS-44 insitu-reinforced (ISR) Si_3N_4 . The major process steps include ball milling, surfactant coating, compounding with the binder, filament extrusion, part building by FDC, thermal binder removal and finally sintering. Issues critical to manufacturing of advanced structural ceramics by the FDC process which have high strength and high Weibull modulus will be detailed. The authors wish to acknowledge sponsorship of this research under DARPA ONR Contract Number N00014-94-C-0115.

11:15 AM

SELECTIVE AREA LASER DEPOSITION VAPOR INFILTRATION, SALDVI, OF POWDERS AS A SOLID FREEFORM FABRICATION, SFF, PROCESS: James E. Crocker¹; Shay Harrison¹; L. Sun¹; H. L. Marcus¹; L. Shaw¹; ¹Institute of Materials Science, University of Connecticut, Storrs, CT 06269-3136 USA

SALDVI is a SFF approach that uses thermal or photo decomposition of precursor gases to infiltrate layer by layer of powders to create a shape. The process will be defined in its application to ceramics and results of processing variables on the microstructures will be described. The range of possible materials and combinations of materials will be presented with the majority of the effort reported on SiC and Si_3N_4 and their composites.

11:40 AM

AMORPHOUS FIBERS BY MELT EXTRACTION: *Ena A. Aguilar*¹; Robin A.L. Drew¹; Claudia Milz²; Bilge Saruhan-Brings²; Bernd Hildmann²; ¹Department of Mining and Metallurgical Engineering, McGill University, 3610 University St., Montreal, Quebec H3A 2B2 Canada; ²DLR, Deutsche Forschungsanstalt für Luft- und Raumfahrt e.V., Porz-Wahnheide Linder Höhe, D-51147 Köln Germany

Alumina-yttria fibers were fabricated using the melt extraction technique. These fibers were completely amorphous in their as-extracted state. Two types of fibers are generally obtained depending on the extraction speed. At very low speed (<3 m/s), fine and uniform fibers of high quality are produced. However, when the wheel speed exceeds a critical velocity, Rayleigh waves are formed on the free surface of the fiber due to instability and the effects of surface tension. The average tensile strength of the extracted fibers was 1 GPa for thin, uniform diameter fibers. Differential Scanning Calorimetry (DSC) on the as-extracted fiber gave one exothermic peak for YAG and YAG/YAP eutectic compositions, and three exothermic peaks for the $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic composition. In order to characterize each phase transformation, x-ray diffraction (XRD) analysis was performed on the $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$ fibers heat-treated to each phase transformation completion temperature. For YAG and YAG/YAP eutectic compositions, the exotherm corresponds to the crystallization of amorphous phase into YAG. However, for $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic composition, first peak corresponds to the crystallization of the amorphous phase into YAG, the second peak is due to the crystallization of

the remaining amorphous phase to $\delta\text{-Al}_2\text{O}_3$, finally, the third peak corresponds to the polymorphic phase transformation from $\delta\text{-Al}_2\text{O}_3$ to $\alpha\text{-Al}_2\text{O}_3$.

12:05 PM

CONTROLLED COMBUSTION SYNTHESIS IN THE $\text{TiH}_2\text{-B}$ SYSTEM: *S. Ozbilen*¹; ¹Gazi University, Faculty of Technical Education, Metal Education Dept., Teknikokullar, Ankara Turkey

Controlled combustion synthesis was carried out in the $\text{TiH}_2\text{-B}$ system. Compositions of different TiH_2 levels were studied to investigate the effect of diluents on the level of exothermicity of the self-propagating reactions. Compressed pellets of $\text{TiH}_2\text{-B}$ mixtures were degassed and ignited in a reaction chamber. Thermal analysis by DTA was carried out on the samples. XRD and SEM investigation were used for the examination of as-reacted pellets of TiB-Ti ceramic matrix composites. Resulting microstructures and properties are discussed.

TEXTURE IN FILMS AND COATINGS: Session III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee
Program Organizer: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Tuesday AM

October 13, 1998

Room: Zurich/Tokyo

Location: O'Hare Hilton Hotel

Session Chair: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

8:30 AM INVITED PAPER

THE DEVELOPMENT OF CRYSTALLOGRAPHIC TEXTURE IN FILMS USED IN MAGNETIC RECORDING: *David E. Laughlin*¹; Yu-Nu Hsu¹; Bin Lu¹; ¹Carnegie Mellon University, Department of Materials Science and Engineering, 3307 Weon Hall, Pittsburgh, PA 15213-3890 USA

Thin magnetic films used for magnetic recording are sputter-deposited on another film usually called the underlayer. The underlayer has important functions which include the control of the magnetic layer grain size, the control of the magnetic layer crystallographic texture and the production of a solute enriched grain boundary in the magnetic film. In this talk we will review these important functions of the underlayer and discuss the way that current state of the art magnetic recording films have evolved over the past two decades. Our emphasis will be on the microstructural development of the films and how Materials Science principles have been applied to aid in the development of today's Co based alloys used in high density thin film storage media. This work has been supported over the years by the Data Storage Systems Center of CMU through NSF ECD 89-07068 and the DOE (DE-FG02-90ER45423). B. L. has been supported by a grant from INTEVAC.

9:05 AM

CHARACTERIZATION OF CRYSTALLOGRAPHIC TEXTURE IN Co-BASED MAGNETIC MEDIA : *Gaurav Khanna*¹; James Freitag¹; Bruce M. Clemens¹; ¹Stanford University, Materials Science and Engineering, 416 Escondido Mall, Bldg. 550, Stanford, CA 94035 USA

In polycrystalline Co-based magnetic media, the magnetic and recording properties are strongly influenced by the orientation tendencies of the c-axis of the hcp media grains. For example, many manufacturers use a (100)-oriented Cr underlayer to induce a nominal (11-20) fiber texture in the media layer. This places the easy c-axis in

the plane of the disk, favoring in-plane magnetization. For smooth disks, the in-plane orientation of the c-axis is random and the magnetic properties are also isotropic in the plane. However on grooved disks, the magnetic properties are anisotropic, and the orientation ratio (the ratio of circumferential to radial coercivity) can be greater than one, leading to improved recording performance. Various possible explanations for this effect have been presented, including anisotropic in-plane distribution of Co c-axis and anisotropic strain. The structural origins of the orientation ratio were explored using synchrotron-based x-ray diffraction and atomic force microscopy. Asymmetric diffraction geometry was used to avoid the differential absorption and shadowing which plagues grazing incidence scattering experiments on grooved samples. X-ray wavelengths which are strongly absorbed by the NiP underlayer were used to eliminate scattering from the Al substrate. By comparing the surface topography, as measured by AFM, to the texture x-ray rocking curves parallel and perpendicular to the grooves, it was observed that both the Cr underlayer and the Co media layer exhibit conformal texture with the Cr (100) and Co (1120) planes following the local surface normal. This produces a fanning effect which reduces the intensity of diffraction features along a direction perpendicular to the grooves. However, after accounting for this fanning effect, variations in the two-fold Co diffraction peak intensities parallel and perpendicular to the grooves seem to offer strong evidence of an anisotropic in-plane distribution of the Co c-axes, with a preference for the c-axes to lie along the groove direction. Current experiments with epitaxial Cr/Co-alloy films grown on stepped MgO (001) substrates will give quantitative values for the contribution of anisotropic strain and c-axis orientation on the magnetic orientation ratio.

9:25 AM

CRYSTALLOGRAPHIC TEXTURE OF THIN FILM MATERIALS EXHIBITING GIANT MAGNETORESISTANCE: *L. P.M. Brandao*¹; Xin Dong¹; H. Garmestani¹; K. H. Dahmen²; ¹Florida State University, Department of Mechanical Engineering and MARTECH, Tallahassee, FL, USA 32310-2175, and Departamento de Engenharia Mecânica e de Materiais, IME, Rio de Janeiro, Brazil; ²Florida State University, Department of Chemistry and MARTECH, Tallahassee, FL 32306-3006 USA

Thin films of $\text{La}_{1-x}\text{M}_x\text{MnO}_3$ (M=Ca, Sr) with different thickness have been produced on (100) oriented LaAlO_3 , yttrium stabilized zirconia (YSZ) and Si substrates by liquid delivery metal organic chemical vapor deposition. The effects of the substrate type and film thickness on the crystallographic texture of these films were investigated using x-ray technique. XRD analyses showed that for thickness of ≤ 500 Å, the films were monocrystalline and epitaxially grown on LaAlO_3 substrates and polycrystalline on YSZ. At thickness ≥ 500 Å, all the films were polycrystalline and highly textured. The magnetoresistance resistance of the films on YSZ was the highest and it was explained in terms of their texture.

9:45 AM

GROWTH PROCESSES, TEXTURE AND INTERGRANULAR MAGNETIC INTERACTION IN HARD MAGNETIC Co-W AND Co-P BASED FILMS: *Vladimir Grigor'evich Shadrow*¹; Anatoly Vasil'evich Boltushkin¹; Lyudmila Vasil'evna Nemtsevich¹; ¹Inst.Solid State Physics, Acad.Sci.of Belarus, P.Brovki,17, Minsk 220072 Belarus

Texture, microstructure and magnetic properties of hard magnetic electrodeposited Co-W, Co-P based films have been investigated by means of EM, XRD, AFM and AGFM. It is shown that the character of the microstructure formation and that of the texture growth are interconnected and accounted for by electrode reactions products adsorption at the cathode. A comparison is made with structure formation peculiarities in hard magnetic Co-P based films crystallized from the initial amorphous state and with those in Co deposited alumite films. The texture and microstructure influence on magnetic properties, in particular, intergranular magnetic interaction, evaluated from the remanence and delta M curves, is discussed as well as the irreversible susceptibility curves shape dependence on magnetic (and structural) films inhomogeneity.

10:05 AM

MICROSTRUCTURES OF PLASMA-SPRAYED AISI 304 STAINLESS STEEL COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Textures and microstructures of Plasma-sprayed AISI 304 stainless steel coatings were studied by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. Parameters of plasma-spraying for AISI 304 stainless steel are gun power, arraying distance, surface speed, voltage and powder feed rate. The Plasma-sprayed layer shows a lamella structure and is built up particle by particle. Diffraction pattern of plasma-sprayed layer is changed by plasma-spraying parameter Pole figure of plasma-sprayed layer is similar to AISI 304 stainless steel polycrystalline and is affected by plasma-spraying parameter.

10:25 AM BREAK

10:35 AM

A STUDY ON THE TEXTURE OF PLASMA-SPRAYED AISI 316 STAINLESS STEEL COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim¹; Young Woo Rheem¹; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Surface morphology and texture of the plasma-sprayed AISI 316 stainless steel coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion resistance of the various coatings are investigated by using potentiostat. The corrosion and erosion resistance of the coatings are greatly affected by the gun power. Pole figure of coating layer is similar to MSI 316 stainless steel polycrystalline, and is changed by plasma-spraying parameter. The thickness of the splats and disturbance of the unmelted particles were increased with a decreasing the gun power.

10:55 AM

SURFACE MORPHOLOGY AND TEXTURE OF PLASMA-SPRAYED Ni COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 305-764 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Surface morphology and texture of the plasma-sprayed AISI 316 stainless steel coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behaviour of the various coatings are studied by using potentiostat. Diffraction pattern of plasma-spraying coatings layer is changed by plasma-spraying parameter. Texture of coating layer is similar to Nickel polycrystalline, and is affected by plasma-spraying parameter.

11:15 AM

TEXTURES AND MICROSTRUCTURES OF AS PLASMA-SPRAYED AND RECRYSTALLIZED ALUMINIUM AND COPPER COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 305-764 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Texture and microstructures of as plasma-sprayed and recrystallized Al and Cu coatings were studied by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behavior of the various types of coatings layer are investigated by using potentiostat. Texture of a plasma-sprayed and

recrystallized Al and Cu coatings are similar to each polycrystalline, and is affected by plasma-spraying parameter. Plasma-sprayed coatings layer shows the dendrite morphology.

11:35 AM

TEXTURE MEASUREMENT OF PLASMA-SPRAYED NiCr AND NiCrAl ALLOY COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Texture and surface morphology of plasma-sprayed NiCr and NiCrAl coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behavior of the various types of coatings layer are investigated by using potentiostat. The diffraction pattern and texture of as plasma-sprayed NiCr and NiCrAl alloy coatings is changed by plasma-spraying parameters. Also, texture change and corrosion and erosion behavior of plasma-spray coatings layer are affected by plasma-spraying parameters.

Technical Program

TUESDAY PM

DISCONTINUOUSLY REINFORCED ALUMINUM-COMPOSITES: PRESENT & FUTURE: Synthesis of Discontinuously Reinforced Aluminum

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Warren H. Hunt, Aluminum Consultants Group, Inc., Murrysville, PA 15668 USA

Tuesday PM Room: Dublin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Daniel B. Miracle, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

2:00 PM INVITED PAPER

CAST DISCONTINUOUSLY REINFORCED ALUMINUM FOR OPTICAL MECHANICAL ASSEMBLIES: *Joseph M. Kunze*¹; ¹Triton Systems, Inc., 200 Turnpike Road, Chelmsford, MA 01824 USA

High volume fraction (55-75%) discontinuously reinforced aluminum (DRA) produced by pressure infiltration casting techniques have been accepted by the electronic packaging industry due to their attractive combination of improved heat dissipation and tailored coefficient of thermal expansion. With their greatly increased stiffness, these DRA's also offer a lightweight alternative for use in optical mechanical assemblies (OMA's). In the past, several barriers have existed to their acceptance in this area including prototype delivery time, unit cost, and ability to process thin sections. Under SBIR funding from the Army, Triton Systems is prototyping an OMA component to demonstrate the potential of Triton's Enhanced Pressure Infiltration Casting technology in fabricating DRA OMA's. The benefits of casting a DRA OMA with this technology include increased performance, lower cost and reduced part count obtained by integrating several parts of a complex, lightweight OMA into a single casting. This paper reviews the development of the DRA component and materials.

2:30 PM

TiB₂ - REINFORCED ALUMINUM COMPOSITES: *J. D. Ellis*¹; ¹London & Scandinavian Metallurgical Co. (LSM), Fullerton Rd., Fotherham, South Yorkshire S60 1DL England

For the past 3 years LSM has been developing TiB₂ reinforced aluminum composites in a European Community funded project. This collaborative project involved 10 other organizations from Europe comprising users of aluminum composites, universities and parts fabricators. At the outset of the project a maximum of 8 wt % TiB₂ could be incorporated into the aluminum. Now over 20 wt % can be incorporated. Strength and modulus values have increased in line with this to the level at which they are comparable with SiC and Al₂O₃ reinforced aluminum. The TiB₂ is inert in aluminum and being 1 mm in size means that the MMC is easily machined and mechanically formed. Initial applications for the Al-TiB₂ MMC are for wear resistant applications requiring mechanical processing in their manufacture and sales of tens of tonnes a year are expected by 1999. The presentation will give a detailed account of the scientific, engineering

and marketing effort that LSM has undertaken, presenting an optimistic view of the future.

3:00 PM

PROCESSING AND EVALUATION OF SiC-REINFORCED ALUMINUM ALLOY COMPOSITES : *A. B. Pandley*¹; R. Wheeler¹; B. S. Majumdar¹; D. B. Miracle²; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; ²Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLM, Wright Patterson AFB, OH 45432 USA

Discontinuously reinforced aluminum (DRA) composites usually possess superior specific stiffness and strength, wear resistance, creep resistance, and thermal resistance as compared to the unreinforced aluminum alloys. Powder metallurgy processing offers better distribution of SiC particles than the conventional casting technique. Vacuum degassing of powders constitute an important step in the processing of these materials. Although higher degassing temperature may be preferred to avoid blistering during post-processing heat treatment, it may be detrimental from an oxidation point of view. In this study, the effect of degassing temperature/time on the microstructure and tensile properties of DRAs will be evaluated. The materials based on 2009/15 vol.% SiC and 7091/15 vol.% SiC will be considered for this purpose. The role of oxides on the strength and ductility of these composites will also be discussed.

3:30 PM

IN-SITU FORMATION OF SILICON CARBIDE IN ALUMINUM ALLOYS: *R. G. Reddy*¹; D. M. Kocherginsky¹; ¹The University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487 USA

Discontinuously reinforced aluminum composites are rapidly emerging as new commercial materials for aerospace and automotive industrial applications. In this study, the effect of temperature and alloy composition on the equilibrium species distribution in the liquid Al-Si/SiC composite was calculated using Gibbs energy minimization method. The alloy (Al-30%Si) and two types of carbon sources were used for in-situ formation of a silicon carbide. The experiments were carried out for 1 to 20 hours at 1273 to 1573K using alumina crucibles. The experimental results showed that when natural gas was used as a carbon source the efficiency of SiC formation was very low and also a small amount of SiC formed was floated to the surface. When the activated carbon particles were used as a carbon source most of the carbon completely reacted and formed a silicon carbide. The SiC was formed by movement of the reaction zone from the surface to the center of the carbon particles. The silicon carbide formed in the melt retained the size and the shape of initial carbon.

4:00 PM

PROCESSING AND CHARACTERIZATION OF 6061Al-TiN PARTICULATE REINFORCED METAL MATRIX COMPOSITES PRODUCED BY MELT STIR CASTING: *J. Vijay Kumar*¹; ¹Indian Institute of Science, Department of Metallurgy, Bangalore 560 012

The present paper describes the processing and characterization of a novel metal matrix composite system, 6061 Al-10TiN. There are no significant reports existing in literature on this system. The composite has been prepared using the meltstir casting route and subsequently cast billet is extruded. The present study aims at mechanical property evaluation, age hardening behaviour, sliding wear behaviour and thermal characterization. Three types of composites with varying volume fraction of reinforcement have been prepared, viz: 6061Al-5vol%TiN(78micron), 6061Al-10vol%(78micron) and 6061Al-10vol%(38micron). The unreinforced alloy also has been

processed under identical conditions to compare the results with the composites. The initial microstructural characterization has showed some interesting results. In the 38micron TiN containing composite, the microstructure reveals the extensive formation of a second phase around the TiN particles. However it is not the case with the 78micron particulate reinforced composite. Detailed SEM and XRD results indicate that the formation of the second phase is due to the conversion of the TiN particles into TiO₂ and other off stoichiometric nitrides. We are in the process of correlating the microstructure with the mechanical properties. The composites are also being tested for their room temperature and high temperature tensile and compressive strengths. We are also studying the wear behaviour of the composite under dry sliding condition. The results of all these will be presented.

4:30 PM

LOW COST ALUMINUM DROSS REINFORCED METAL MATRIX COMPOSITES: *Kevorkijan M. Varuzan*¹; ¹Head Of The Project Consortium, MMCs, Lackova 139, Limbus, Slovenia 2341

In the casting-house, aluminum dross represents a permanent source of waste generation, especially on the end of secondary dross processing, when a fine, low metallic dust is generated by tumbling. In the present work, this finely powdered by-product was employed as a low cost reinforcement in discontinuously reinforced aluminum matrix composites. By using the fine (less than 50mm) fraction of non-metallic particles generated during aluminum dross treatment and the part of in-house recovered aluminum alloy (A 380), low cost aluminum dross metal matrix composites (AIDMMCs) were routinely produced by the conventional squeeze casting and pressure infiltration techniques. The tensile strength and hardness properties of up to 20 vol. % fine dross particles in AIDMMC were examined. Some preliminary dry sliding wear testing was also carried out using a pin-on-ring wear testing machine. The initial results suggest that the fine fraction of aluminum dross particles is promising filler for wear resistant MMCs.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Behavior II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Flow & Fracture Committee, Mechanical Metallurgy Committee

Program Organizers: Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Marc A. Meyers, IMM, University of California, San Diego, CA 92093 USA; Naresh Thadhani, Georgia Inst of Tech, Sch of Mats Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Tuesday PM Room: Sydney
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Kenneth S. Vecchio, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA; Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA

2:00 PM

PLASMONS AT SHOCK FRONTS: *John J. Gilman*¹; ¹University of California at Los Angeles, Materials Science and Engineering, 6532 Boelter Hall, UCLA, Los Angeles, CA 90095-1595 USA

In dense solids, plasmons are collective analogs of molecular polarization oscillations. Thus they occur in all solids. The oscillation frequency, ω_p , is given by: $(4\pi nq/m)$ where n = electron density,

q = electron's charge, and m = its mass. Experimental frequencies (from EELS) equal: (cB) where B = bulk modulus, and c is a constant given by simple theory. Thus EELS can be used to determine local elastic stiffnesses. In addition, for the simple metals, $\omega_p = \mu S_{4/5}$ where S is the specific surface energy and μ is a constant. In semiconductors, the plasmon energy equals about 10X the band gap. Plasmas are transparent to optical wavelengths shorter than λ_p (1550 Å for Li, e.g.), so plasmon diagnosis could be useful for studying in situ shock propagation. Since the damping in the electron gas is very small, and a shock front is an intense collective excitation, it can readily cause resonant plasmon excitation.

2:20 PM

FLOW STRESS OF COMMERCIAL PURE TITANIUM OVER A BROAD RANGE OF TEMPERATURE AND STRAIN RATE: *Sia Nemat-Nasser*¹; W. G. Guo¹; J. B. Isaacs¹; ¹Center of Excellence for Advanced Materials, Department of Applied Mechanics and Engineering Sciences, University of California, San Diego, La Jolla, CA 92093-0416 USA

The flow stress of a commercially pure titanium (CP-Ti) is evaluated experimentally over the temperature range of 77K to 1000K and the strain-rate range of 0.001/s to 8,000/s. Experiments show that the material exhibits a three-stage hardening behavior in the temperature range of 400K to 800K, depending on the strain rate. Interrupted tests are performed to examine the resulting microstructural features by optical microscopy. Deformation twinning is seen to play an important role in the plastic response of CP-Ti. The density of the deformation twins increase with decreasing temperature and increasing strain rate and strain. Although the deformation mechanisms in CP-Ti include slip by dislocation movements and deformation twinning, the dislocation is the main mode of plastic flow for CP-Ti. To develop a reasonable constitutive model for the flow stress of CP-Ti, the effect of deformation twinning must be included

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DYNAMIC RESPONSE OF XD Ti-46Al: *William Frederick Cost*¹; Mustafa Guden²; I.W. Rick Hall²; Ernest S.C. Chin¹; Justin Combs²; ¹U.S. Army Research Laboratory, Weapons Materials Division, ATTN: AMSRL-WM-ME, Aberdeen Proving Ground, MD 21005 USA; ²University of Delaware, Materials Science Department, Spencer Laboratory, Newark, DE 19716 USA

An earlier ballistic study of 5%, 10%, and 15% TiB₂ reinforced XD TiAl composites showed promising dynamic behavior due to high strain-rate sensitivity. Subsequent investigation was initiated to characterize the quasi-static and high strain-rate deformation of 1 vol.% TiB₂ XD Ti-46Al in a variety of heat treated conditions to better understand the relationship between microstructure and rate-dependent phenomena. Quasi-static and Split Hopkinson Pressure Bar compression experiments were performed to characterize and compare the stress-strain response of this material. Subsequent microscopic analyses were performed to identify the dynamic flow mechanisms exhibited in this material. Details of this study will be presented along with comparisons to earlier high strain-rate results from higher reinforcement loaded XD materials tested at ambient and elevated temperatures.

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TESTING WITH THE TORSIONAL SPLIT HOPKINSON BAR AT STRAIN RATES ABOVE 10,000 1/S: *Amos Gilat*¹; Ching-Shan Cheng¹; ¹Ohio State University, Applied Mechanics, 155 W. Woodruff Ave, Columbus, OH 43210 USA

The torsional split Hopkinson bar technique is used for testing materials at strain rates above 10,000 1/s. This strain rate, which is an order of magnitude higher than the typical with this technique, is obtained by using a specimen having a very short gage length. Results from tests on steel, aluminum, and titanium show that there is a significant increase in rate sensitivity as the strain rate increases from the range of 1,000's to the range of 10,000's 1/s. The observed increase in strain rate sensitivity is in line with results obtained from

testing with the pressure-shear plate impact technique at strain rates of the order of 100,000 1/s. In addition, the results show that the formation of adiabatic shear bands is sensitive to the strain rate. Microscopic examination of tested specimens shows that the deforming specimen gage is typical of the microstructure of the bulk material. A numerical analysis of the stresses in the specimen, and a comparison of results from tests with specimens of various lengths at the same strain rate, shows that even though some error is introduced when a very short specimen is used, the increase in strain rate sensitivity represents the true material response.

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DYNAMIC RESPONSE OF NATURAL AND ENGINEERED SYNTHETIC BRITTLE/DUCTILE LAMINATE COMPOSITES: *Kenneth S. Vecchio*¹; David Harach¹; Andrew Strutt¹; ¹University of CA, San Diego, Dept. of AMES, Materials Science Group, La Jolla, CA 92093-0411 USA

The quasi-static and dynamic response of brittle/ductile laminate composites are investigated over a range of strain rates, orientations, and loading conditions. The natural brittle/ductile composites, in the form of seashells, were studied to form the basis for mimicking these microstructures and developing engineered synthetic laminate composites (biomimetics). The synthetic composites are metallic (Ti, Ni, or Fe) -intermetallic (Ti-aluminide, Ni-aluminide, or Fe-aluminide) laminate composites containing a range of layer thicknesses, phase volume fractions, layer sequencing, and numbers of layers. The mechanical responses of these synthetic composites were evaluated in order to optimize the microstructure for improved dynamic performance. Deformation behavior and damage evolution will be discussed as a means of supporting the optimization process. Ballistic performance of these natural and synthetic laminate composites will also be presented.

3:40 PM BREAK

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A SHOCK-INDUCED OMEGA TRANSFORMATION IN TANTALUM AND TANTALUM-TUNGSTEN ALLOYS: *Luke M. Hsiung*¹; ¹Lawrence Livermore National Laboratory, Materials Science and Technology, L-370, P.O. Box 808, Livermore, CA 94551-9900 USA

A shock-induced omega transformation has been observed within shock-recovered pure Ta and Ta-10W samples. Plate- or lath-like ω phase is found to coexist with the $\{112\}\langle 111\rangle$ type deformation twins. Both deformation twins and shock-induced ω phase are formed along the $\{211\}$ slip planes, and have a common $\{211\}$ habit plane with the parent β matrix. It is suggested that the $\{211\}\langle 111\rangle$ shear has played a major role in the occurrence of the omega transformation in shocked pure Ta and Ta-10W. The result of higher volume fraction of shock-induced ω phase in Ta-10W than in pure Ta suggests that the shock-induced omega transformation can be promoted by solid-solution alloying. A dislocation mechanism for the shock-induced omega transformation is proposed and discussed. This work was supported by the Joint DOD/DOE Munitions Development Program under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

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A COMPARISON OF MECHANICAL RESPONSE BETWEEN A WROUGHT AND AN INVESTMENT CAST GAMMA-TiAl ALLOYS AS A FUNCTION OF STRAIN RATE AND TEMPERATURE: *Zhe Jin*¹; George T. Gray¹; ¹Los Alamos National Laboratory, Materials Science and Technology Division, MST-8, MS G755, Los Alamos, NM 87545 USA

Mechanical responses of a wrought and an investment cast gamma-TiAl alloys at strain rates ranging from 0.001/s to 3000/s and temperatures from -196°C to 1000°C were studied in compression. Both alloys exhibited similar work hardening rates and strain rate sensitivi-

ties within the strain rates and temperatures studied. Although the temperature dependence of yield and flow stresses was similar in these two alloys, the wrought TiAl alloy showed high yield and flow stresses compared to the cast TiAl alloy. The difference in their yield and flow stresses is ascribed to the smaller grain size in the wrought TiAl alloy and the difference in texture between them. The yield and flow stresses were found to be sensitive to texture in both alloys. Mechanisms controlling mechanical responses at different strain rates and temperatures are analyzed.

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SPONTANEOUS AND FORCED SHEAR LOCALIZATION IN HIGH-STRAIN-RATE DEFORMATION OF TANTALUM: *Yuejian Chen*¹; Marc A. Meyers¹; Vitali F. Nesterenko¹; ¹University of California, San Diego, Department of Applied Mechanics and Engineering Sciences, 9500 Gilman Drive, La Jolla, CA 92093 USA

High-strain-rate shear localization was induced in tantalum by (a) lowering the deformation temperature or (b) subjecting it to high strains by dynamic deformation (up to $\epsilon_t = -0.8$) or (c) pre-shocking (at $\epsilon_{eff} = 0.22$) and then deforming it. Although at ambient temperature the deformation of tantalum is macroscopically uniform to high strains ($\epsilon_t @ -0.8$), at 77 K shear localization under the same loading condition was developed with a critical strain of $-0.2, -0.3$. This higher propensity to shear localization at low temperatures is a direct consequence of the combination of lower heat capacity and higher rate of thermal softening. At the three temperatures investigated (77, 190, and 298 K), localization occurs at strains significantly higher than the instability strains (the maxima of the adiabatic stress-strain curves for these three temperatures). The thicknesses of the forced localization regions and shear bands were found to be a function of temperature, and decreased with decreasing temperature (at the same strain) in accord with the equation proposed by Y. Bai et al. (Acta Mechanica Sinica, 2(1986), 1). Shock deformation of tantalum enhances its predisposition to subsequent shear localization, and this was demonstrated by subjecting shocked and unshocked specimens to high strain, high strain rate deformation through the collapse of a thick-walled cylinder assembly.

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DYNAMIC BEHAVIOUR OF SOME STRUCTURAL MATERIALS AT STRAIN RATES 10^2 - 10^4 S⁻¹: *Anatoly M. Bragov*¹; Andrey K. Lomunov¹; Vladimir B. Korobov¹; Andrey G. Ugodchikov¹; ¹Research Institute of Mechanics, State University of Nizhny Novgorod, 23, Gagarin Ave., Korp.6, Nizhny Novgorod, Nizhegorodskaya obl. 603600 Russia

In this lecture we suggest the results of the study of the effect of the strain rate and its history of change on the physico-mechanical properties of some metals and alloys. With the help of Kolsky method and using the Split Hopkinson Pressure Bar dynamic diagrams have been obtained for deformation under tension, compression and shear as well as limit characteristics of strength and plasticity. Along with macroscopic properties, microstructural features of the high strain rate processes have been investigated. To this end an analysis of microhardness, of residual microstresses of the second kind and of the block-type structure parameters has been accomplished. Based on the results obtained a mechanism is suggested which explains the strain-rate hardening of the materials tested. The complex of physico-mechanical properties obtained testifies to a complicated rheological behaviour of the materials tested.

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ON THE TIME-TEMPERATURE CORRESPONDENCE FOR YIELD STRESS DURING THE DYNAMIC DEFORMATION OF METALS: Alexey A. Gruzdokov¹; *Nikita F. Morozov*¹; Yuri V. Petrov¹; ¹St.Petersburg State University, Mathematics & Mechanics Faculty, Petrodvorets, Bibliotechnaya pl., 2, St.Petersburg 198904 Russia

The dynamic yield criterion applicable for both high-strain-rate and quasistatic deformation of metals is proposed. The criterion is based on the notion of incubation time previously proposed by au-

thors for use in short pulse fracture mechanics. In special cases the approach coincides with the classical critical stress and the Campbell time integral of stress criteria. The incubation time is associated with the mean dislocation move velocity. On the basis of this criterion the estimation of the time-temperature correspondence for mild steel is carried out. Good agreement between theoretical calculations and well-known experiments is observed.

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Diffuse Interface Approaches to Microstructure Evolution

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Tuesday PM Room: International Ballroom
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Paul Fife, University of Utah

2:00 PM INVITED PAPER

PHASE FIELD MODELS OF SOLIDIFICATION: *James A. Warren*¹; ¹NIST, Metallurgy Division & CTCMS, Bldg 223/B164, Gaithersburg, MD 20899 USA

Phase field models are now a popular technique for simulating the dynamics of solidifying materials. These models employ the concept of a diffuse liquid-solid interface and energy penalties for gradients in place of the more traditional interface tracking techniques. Many of the fundamental ideas embodied in current phase field models were first elucidated by John W. Cahn in 1960 (nearly twenty years before the development of current models). This talk will address the enormous variety of phenomena which are now being modeled with this method, with emphasis on directional solidification, grain boundary formation and grain growth.

2:45 PM INVITED PAPER

MODELING PRECIPITATION AND COARSENING OF ORDERED INTERMETALLICS USING SYSTEMS OF CAHN-HILLIARD AND ALLEN-CAHN EQUATIONS: *Long-Qing Chen*¹; ¹Penn State University, Materials Science and Engineering, 118 Steidle Building, University Park, PA 16802 USA

Precipitation of an ordered intermetallic phase or phases from a disordered matrix is a basic process that underlies the processing of many advanced alloys such as high-temperature superalloys and ultralight aluminum alloys. It involves a number of different processes, including atomic ordering to produce the ordered structure, compositional clustering to reach the equilibrium precipitate composition, and coarsening to reduce the total interfacial energy. The

precipitate morphology and coarsening kinetics depend on many factors such as volume fraction, temperature, and the lattice mismatch between precipitates and matrix. In this presentation, a phase-field model using systems of Cahn-Hilliard and Allen-Cahn equations will be presented for modeling the microstructural evolution during precipitation of ordered intermetallics from a disordered matrix. The dependencies of the two-phase morphology and the corresponding coarsening kinetics on the volume fraction, the lattice mismatch, and an externally applied stress will be discussed. The results will be compared with existing theories and experimental measurements. This work is supported by the Office of Naval Research and the National Science Foundation.

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3:50 PM INVITED PAPER

THE ROLE OF INTERFACES AND DOMAIN BOUNDARIES IN ORDER-DISORDER TRANSITIONS: *Annick Loiseau*¹; ¹Laboratoire d'Etudes des Microstructures, Unite Mixte Onera-Cnrs, O.N.E.R.A., B.P. 72, 29 av. de la Division Leclerc, 92322, CH=E2tillon France

In recent years substantial experimental and theoretical progress has been made in our knowledge of the thermodynamics and kinetic behaviour of domain boundaries and interfaces in order-disorder transitions. Recent advances include quantitative studies of this behaviour at equilibrium of domain walls and surfaces close to a phase transition and numerical simulations of the kinetics of ordering reactions and microstructural evolutions. The scope of the talk is to present the state of the art in this field.

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Fatigue in Titanium Intermetallics

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; Daniel Eylon, Univ. of Dayton, Graduate Materials Engineering, Dayton, OH 45469-0240; J. P. Gallagher, University of Dayton, UDRI-Structural Integrity, Dayton, OH 45469-0120 USA; G. Lutjering, TU Hamburg-Harburg, Hamburg 21093 Germany

Tuesday PM Room: Athens/Berlin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: G. Lutjering; R. O. Ritchie

2:00 PM KEYNOTE

EFFECTS OF MICROSTRUCTURE ON THE FATIGUE CRACK GROWTH RESISTANCE AND TOTAL LIFE OF GAMMA TiAl BASED ALUMINIDES: *P. Bowen*¹; *Z. W. Huang*¹; *M. Salmani*¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT UK

The influence of microstructure, lamellar colony size, and lamellar colony orientation on fatigue crack growth resistance and total life at room and elevated temperatures will be highlighted. Particular emphasis throughout the presentation will be placed on the fully lamellar and/or near (fully) lamellar microstructures. In this context detailed TEM observations of dislocation and twinning behaviour local to a fatigue crack-tip will be introduced, and micromechanisms of crack advance will be discussed with particular reference to translamellar and intralamellar failure. The importance of both aligned lamellar colonies and randomised lamellar colonies on fatigue crack growth resistance and total life will be addressed. In particular, the

problem of premature interlamellar failure under cyclic loading, and its engineering significance, will be explored. Here, the surface condition of the testpiece must also be taken into account and the influence of as-machined conditions and residual stresses produced by shot peening will be considered. The concept of stress-sampling volume correlations will then be used to rationalise the total life of gamma based aluminides under cyclic loading observed for both plane sided and notched testpieces. Such correlations will be essential and must be quantified if the performance of large scale components is to be predicted (and approved) on the basis of tests carried out on relatively small scale testpieces.

2:30 PM KEYNOTE

OPPORTUNITIES AND CHALLENGES FOR THE USE OF GAMMA TiAl ALLOYS IN HIGH PERFORMANCE TURBINE ENGINES: *James M. Larsen*¹; A. H. Rosenberger¹; K. Li¹; R. John¹; D. C. Maxwell¹; W. J. Porter¹; S. K. Plank¹; N. E. Ashbaugh¹; ¹U.S. Air Force, WL/MLLN, Bldg. 655, 22230 Tenth St., Ste 1, Wright Patterson Air Force Base, OH 45433-7817 USA

Gamma TiAl alloys are highly promising candidates for replacement of more dense nickel-base superalloys currently used in a variety of components in high performance gas turbine engines. The applications under consideration include both static and rotating components, many of which will be subjected to complex loading spectra containing combinations of low and high-cycle fatigue over a range of temperatures. To quantify opportunities and challenges to the widespread use of gamma alloys in engines, a variety of anticipated service conditions were examined, and the effects of a range of loading variables on component fatigue life were evaluated. Key life-limiting conditions were identified for a variety of microstructures. Factors addressed include effects of intrinsic material defects, service-induced damage, loading spectra, temperature, and environment. Emphasis is given to damage tolerance requirements for major, fracture critical components such as engine disks. For components that are exposed to significant levels of high cycle fatigue, such as airfoils, resistance to crack formation and threshold crack growth are crucial to achieving reliability. As an aid to interpretation of the findings, structural capabilities of gamma alloys are contrasted with properties of more conventional materials. The results of this study suggest avenues for materials development and component design that maximize the potential for the use of these materials for long-term applications under realistic engine operating conditions.

3:00 PM

EFFECTS OF MACHINING ON FATIGUE STRENGTH AND CRACK INITIATION IN Ti-47Al-2Nb-2Cr: *P. E. Jones*¹; D. Eylon²; ¹General Motors Powertrain Division, Saginaw, MI 48605-5073 USA; ²University of Dayton, Graduate Materials Engr., 300 College Park, Dayton, OH 45469-0240 USA

Conventional machining processes, such as turning and grinding, will be used to fabricate fatigue loaded components from gamma titanium aluminide alloys. These metal removal processes created a severely work hardened surface layer on Ti-47Al-2Nb-2Cr (at%) samples produced by permanent mold casting and heat treated to a near-gamma microstructure. Effects of this layer on the high cycle fatigue strength (10⁶ cycles) and fatigue initiation sites were examined above and below the ductile-to-brittle transition temperature. All samples were tested to failure under the same step loading profile. Comparisons were made between samples having the same load history. At room temperature, fatigue strength and initiation sites were equivalent for turned and electropolished surface conditions. At the anticipated service temperature, 760C, the work hardened layer quickly recrystallized. This fine recrystallized surface enhanced the fatigue crack initiation resistance of turned specimens when compared to electropolished samples which did not recrystallize during the test. Surprisingly, the severe surface deformation resulting from conventional machining did not impair the high cycle fatigue behavior of this intermetallic alloy under the conditions evaluated.

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FATIGUE STRENGTH OF GAMMA TITANIUM ALUMINIDE AFTER IMPACT DAMAGE: *C. M. Austin*¹; P. K. Wright¹; S. L. Draper²; J. M. Perriera²; B. A. Lerch²; M. V. Nathal²; M. P. Rubal³; P. S. Steif³; T. S. Harding⁴; J. W. Jones⁴; ¹G.E. Aircraft Engines, Cincinnati, OH 45140 USA; ²NASA-Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135 USA; ³Carnegie Mellon University, Department of Mechanical Engineering, Pittsburgh, PA 15213-3890 USA; ⁴University of Michigan, College of Engineering, 2300 Hayward Street, Ann Arbor, MI 48109 USA

Gamma titanium aluminide alloys have properties that are well-matched to the requirements of low pressure turbine blades, offering significant weight reduction potential. A possible exception is impact resistance: gamma blades will be much more vulnerable than superalloy blades. Since impact damage is rare in the latter parts of the turbine where gamma is to be used (barring major upstream malfunction), the actual requirement for impact resistance is difficult to define. Nevertheless, extensive work is being done to characterize the nature of impact damage and its ramifications. A variety of objectives and approaches is being pursued. One group is studying the relationships between impact events, specimen geometries, and observed deformation and cracking. Another group is studying the features controlling crack formation and subsequent growth in fatigue. Another group is directly measuring the fatigue resistance of simulated airfoils after ballistic impacts done at temperature and under tensile load. This paper will provide an overview of these programs and the engineering context in which they are being performed. The authors are grateful for support from NASA and AFOSR.

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FATIGUE LIFE PREDICTION IN IMPACT-DAMAGED GAMMA TiAl: *Trevor S. Harding*¹; J. Wayne Jones¹; Tresa M. Pollock²; Matt P. Rubal³; Paul S. Steif³; ¹University of Michigan, Materials Science and Engineering, 2105 H.H.Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA; ²Carnegie Mellon University, Materials Science and Engineering, Wean Hall, Pittsburgh, PA 15213 USA; ³Carnegie Mellon University, Mechanical Engineering, Scaife Hall, Pittsburgh, PA 15213 USA

The room temperature and elevated temperature growth of fatigue cracks induced by impact damage in gamma titanium aluminides has been investigated in terms of application to damage in low pressure turbine blades. The responses of two gamma titanium aluminide alloys, a duplex Ti-47.9Al-2.0Cr-1.9Nb alloy and nearly fully lamellar Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si alloy, were examined in this study. Damage was induced by low speed impacts and characterized in terms of critical parameters for predicting fatigue failure stress. An experimental method for determining this stress was developed. Results for tests conducted at both room temperature and 600°C will be discussed. Modeling of near-threshold growth of damage induced cracks and its effect on fatigue failure stress prediction will be described, along with an explanation of the possible effects of small crack behavior in impacted specimens.

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FATIGUE DEFORMATION AND FAILURE IN WROUGHT Ti-46Al-2Cr-3Nb-0.2W: *Y-W Kim*¹; D. M. Dimiduk¹; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA

The fatigue properties of a wrought gamma TiAl alloy in air were investigated at temperatures between 600 and 870°C in a duplex (GS~12 micron) and a fully-lamellar (FL) (GS~300 micron) microstructural forms. Tension-tension (R=0.1) fatigue experiments conducted on hourglass specimens show that both microstructures exhibit fairly flat S-N curves at 600°C, with runout fatigue strengths (FS) as high as 0.96UTS at 10⁷ cycles. The duplex material shows higher FS's, which is consistent with their tensile strengths. At and above 800°C, both materials exhibit a two-stage behavior, a gradual decrease of FS at the low cycle regime and a more rapid drop at higher cycle

regime, and the FL material exhibits higher fatigue resistance than the duplex material. The dependency of fatigue behavior on microstructure and temperature is explained using the corresponding tensile behavior with respect to respective brittle-ductile transition temperatures (BDTT). At all temperatures tested, crack-initiation appears to be related to soft grains, microstructural nonuniformity, or flaws. The rapid crack growth behavior under cyclic loading conditions and the small but apparent effect of microstructure on the propagation are discussed in connection with the da/dN curves measured on the same materials. The fracture modes at low temperatures are predominantly transgranular for both materials, but as temperature approaches BDTT, intergranular fracture and lamellar splitting become more important. This boundary weakening is similar to that observed under tensile loading conditions, and its implications will be discussed.

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FATIGUE LIFE PREDICTION FOR TiAl-BASED ALLOY: *Kwai S. Chan*¹; ¹Southwest Research Institute, Materials Engineering Department, 6220 Culebra Road, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

The prediction of fatigue life in TiAl-based alloys is considered in this paper. The fatigue characteristics of TiAl-based alloys are first summarized. Particular attention is focused on the mechanisms of fatigue crack initiation, growth, and coalescence in these alloys, followed by appropriate treatments of these individual processes. The use of conventional life prediction approaches to predict the fatigue life of TiAl-based alloys is then examined through parametric calculations. Fatigue life and crack growth approaches are both considered, either with or without using the concept of a fatigue limit or a large crack growth threshold. The roles of small fatigue cracks and current crack nucleation and coalescence in fatigue life are also addressed. These results are used to identify possible stress regimes where adequate damage tolerance characteristics can be achieved in TiAl-based alloys.

5:00 PM

ELEVATED TEMPERATURE FATIGUE CRACK GROWTH BEHAVIOUR OF A TiAl-BASED ALLOY: *Gilbert Henaff*¹; *C. Mabru*¹; *J. Petit*¹; ¹ENSMA, Laboratoire de Mécanique et Physique des Matériaux, UMR CNRS 6617, Téléport 2, BP 109, Futuroscope Cedex 86960 France

TiAl-based alloys are currently holding high promises as potential elevated-temperature structural materials. Indeed thanks to their superior combination of specific strength as well as their good oxidation and burn resistance, they outperform conventional Ti alloys and may also replace superalloys in the temperature range 550-850°C. With this prospect their damage tolerance must be assessed. However their fatigue crack growth behaviour is still ill-known. In particular a literature survey indicates the influence of temperature on the fatigue crack growth resistance is extremely controversial. The reason is that the observed behaviour actually results from a complex balance of different factors which may act in opposite ways. The present investigations is addressing this issue. The material considered is a quaternary alloy Ti-48Al-2Mn-2Nb tested in the as-cast condition. The methodology used aims in uncoupling the respective influence of temperature on the various factors involved in the growth process: intrinsic resistance, environmental deleterious influence, crack closure shielding, Testings have been carried at room and elevated temperature, in vacuum and ambient air. Some ancillary testings on intermediate atmospheres have also been performed at room temperature in order to gain deeper insights in the embrittling mechanisms. Thus the intrinsic fatigue crack growth resistance, i. e. the resistance exhibited in an inert environment and after crack closure correction, has been investigated at room and elevated temperatures (up to 850°C). It comes out that increasing the temperature induces an enhanced fatigue crack growth resistance in the mid to high growth rate regime in relationship with an improvement in fracture toughness. However the near-threshold behaviour is nearly unaffected when the temperature varies. The same remark applies for the fatigue crack

propagation in ambient air. Besides it has been shown that the magnitude of crack closure effects is nearly similar whatever the temperature and unaffected by environment. In particular, even at elevated temperature, the oxide-induced closure mechanism is not relevant in these materials. In-situ observations support a prevailing roughness-induced mechanism. As a consequence it appears that the environmental contribution in the slow growth rate regime is nearly the same at room temperature as in the range 650- 850°C. This result is discussed in terms of active species, surface reaction kinetics and possible embrittlement by hydrogen released by the dissociation of adsorbed water vapour molecules.

HOT DEFORMATION OF ALUMINUM ALLOYS: Constitutive Modeling, Texture Evolution

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

Tuesday PM

October 13, 1998

Room: Orchard

Location: O'Hare Hilton Hotel

Session Chairs: Paul Dawson, Cornell University, Sibley School of Mechanical and Aerospace Engineering, Ithaca, NY 14853 USA; Dr. Armeand J. Beaudoin, University of Illinois, Urbana-Champaign, IL USA

2:00 PM INVITED PAPER

RECRYSTALLIZATION PROCESSING OF HOT DEFORMED ALUMINUM: "WHAT IS KNOWN AND WHAT NEEDS TO BE UNDERSTOOD": *R. D. Doherty*¹; ¹Drexel University, Department of Material Engineering, Philadelphia, PA 19104 USA

Recent work on rolled or plane strain compressed commercial purity aluminum by different groups has largely solved the long standing problems of the origin of recrystallization texture and its dependence on deformation temperature, strain and initial grain structure. The key features are the apparent stability of cube oriented grains and the ability of such grains to develop low stored energies so giving a very high frequency of cube grain nucleation especially after warm deformation. A further critical feature has been identification of the importance of "orientation pinning - the strong inhibition of growth of recrystallized grains with an orientation that occurs very frequently in the deformed structure. The critical feature of this pinning is when the spacing, in the normal direction, of the texture component in the rolled material being significantly less than the final grain thickness in the fully recrystallized state. However, at least two major problems need to be understood before these initial successes can be repeated in other cases of aluminum alloy processing. The first is a means of predicting the stability of different orientations during deformation and the second task is to understand the precipitation of supersaturated solute during warm deformation of high solute, age-hardening alloys and the influence of such precipitation on recrystallization processing.

2:30 PM INVITED PAPER

MECHANISMS RESPONSIBLE FOR TEXTURE DEVELOPMENT IN A 5182 ALUMINUM ALLOY DEFORMED AT ELEVATED TEMPERATURE: *M. G. Stout*¹; *S. R. Chen*¹; *U. Fred Kocks*¹; *A. Schwartz*²;

S. R. MacEwen³; A. J. Beaudoin⁴; ¹Los Alamos National Laboratory, MST-8: Structure/Property Relations, Materials Science & Technology Division, Mail Stop G755, Los Alamos, NM 87544 USA; ²Lawrence Livermore National Laboratory, Los Alamos, NM 94550 USA; ³Alcan International Ltd., Kingston, Ontario K7L 5L9 Canada; ⁴University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA

The textures that develop in a 5182 aluminum alloy as a result of monotonic high-temperature compression have been investigated. We found that the deformation texture was a function of temperature. For compressive deformation at 200°C and below the material formed the classic (101) deformation texture, while the material develops a texture that is a combination of the classical uniaxial compression deformation texture, (101), and static recrystallization texture, (001), as a result of the deformation alone when the deformation temperature was above 400°C. Our investigation has focused on determining the mechanism responsible for the development of this unusual progression of deformation textures. In order to understand the mechanism(s) responsible for this particular development, we have performed interrupted compression experiments as well as microstructural analyses. The deformation experiments consisted of deforming to a given strain, holding at temperature for various times at zero strain rate, followed by either quenching or further deformation. In addition we have performed orientation imaging microscopy (OIM) to identify the shapes of grains with particular orientations and grain to grain orientation relationships. Finally, crystal plasticity theory was used to model whether or not deformation via different slip systems and diffusion mechanisms could produce the (101) and (001) texture components. Our conclusions are summarized as follows: The texture development is not a result of static processes, i.e. static recrystallization at the various hold times altered neither the textures nor the constitutive behavior. Using the crystal plasticity analysis, we could not predict the (001) texture component, as a result of deformation, with reasonable assumptions of active slip systems. Finally, the microscopic OIM results are consistent with these observations. Sponsored by: Basic Energy Sciences, Division of Materials Sciences, US Department of Energy.

3:00 PM INVITED PAPER

EXPERIMENTAL VALIDATION OF MICROMECHANICAL MODEL PREDICTIONS FOR COLUMNAR GRAIN POLYCRYSTALS: *R. Becker*¹; *H. Weiland*¹; ¹Alcoa Technical Center, Alcoa Center, PA 15609 USA

Polycrystal plasticity models for predicting the evolution of crystallographic texture during deformation have been available for several decades. They have been successful in explaining why certain crystallographic orientations are preferred and have provided reasonable predictions of crystallographic orientation distributions. However, quantitative comparisons of the evolution of lattice orientations in individual grains with experimental results have been less than satisfactory. This discrepancy has been attributed to essential simplifications in the models whereby each orientation is assumed to deform homogeneously and that each grain deforms independently of the influence of the specific surrounding grains. In more recent years, several research groups have incorporated the kinematics of crystal plasticity as the constitutive model in finite element codes. Simulations have been successful in predicting the development of shear bands in single crystals as well as slip patterns and lattice rotations near bicrystal boundaries. By representing the combined effects of several orientations at each integration point, process models have provided predictions of earing in cup drawing and the evolution of crystallographic texture during rolling, forging and extrusion processes. Despite these successes, finite element micromechanical models of the grain structure in a polycrystal have not been successful in simulating the details of the grain fragmentation and local lattice rotations. The reason given is that the two dimensional models are not capable of capturing the true three dimensional interactions of grains in a polycrystal. Thus, validation of micromechanical models for polycrystal deformation would require either three dimensional polycrystal models or two dimensional polycrystals. The former necessitates not only significant computational resources but full three

dimensional characterization of the polycrystal before and after deformation. The latter approach is simpler as it requires only creation of polycrystals with a columnar grain structure. Columnar grain structures have been cast for commercial purity aluminum and three binary aluminum alloys to assess the ability of micromechanical models to simulate details of the deformation of a polycrystal. The columnar grain samples have been deformed at room and elevated temperature in a channel die with the columnar direction of the grains aligned with the zero strain direction of the channel. Microstructural measurements at the surface of the specimens were obtained at several stages of deformation by orientation imaging microscopy. At the final strain, the specimens were sectioned along their mid-plane to obtain lattice orientation measurements devoid of the surface effects. The measured initial grain structure was used to define the initial state of the micromechanical models. The full specimen cross section was modeled to alleviate concerns about the effects of boundary conditions on the deformation. The slip system constitutive behavior and slip mode were altered to account for changes in mechanisms at the various temperatures. Results from both the experiments and analysis are given as well as a detailed comparison.

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COUPLED FINITE ELEMENT-MONTE CARLO SIMULATION OF MICROSTRUCTURE AND TEXTURE EVOLUTION DURING HOT DEFORMATION OF ALUMINUM ALLOYS: *B. Radhakrishnan*¹; *G. Sarma*¹; *T. Zacharia*¹; ¹Oak Ridge National Laboratory, Computer Science and Mathematics Division, Oak Ridge, TN 37831-6140 USA

A novel simulation technique for capturing the microstructure and texture evolution during hot working of fcc polycrystals is presented. The technique is based on coupling a microstructural deformation model based on crystal plasticity with a Monte Carlo simulation of recovery and recrystallization. The deformation model captures the stored energy and the crystallographic orientation distributions in the microstructure for a given incremental deformation. The Monte Carlo simulation captures the microstructural evolution by thermally assisted phenomena such as recovery and recrystallization in the corresponding time increment. The recovery model captures the evolution of the dislocation substructure and also the formation of dynamically recrystallized nuclei. A unique feature of the simulation technique is its ability to predict the orientations of the dynamically recovered/recrystallized grains, so that it is possible to follow the texture evolution as a function of deformation. The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes. *This research was sponsored by the Division of Materials Science, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. The authors acknowledge the use of the Intel Paragon XP/S 35 located in the Oak Ridge National Laboratory Center for Computational Sciences (CCS), funded by the Department of Energy's Office of Scientific Computing.

4:00 PM INVITED PAPER

POLYCRYSTAL SIMULATIONS OF TEXTURE EVOLUTION DURING DEFORMATION PROCESSING: *G. Sarma*¹; *B. Radhakrishnan*¹; *T. Zacharia*¹; ¹Oak Ridge National Laboratory, Oak Ridge, TN 37831-6140 USA

Some recent research on the hot deformation of aluminum crystals has led to the theory that at elevated temperatures, slip occurs on {110}<110> systems in addition to the usual {111}<110> systems active at lower temperatures. The effect of these additional slip systems on the texture evolution of aluminum single and polycrystals is studied using finite element simulations. The crystals are deformed in plane strain compression, and the constitutive response is modeled using crystal plasticity to track the reorientation and work hardening of the crystals. By discretizing each crystal with a large number of elements, the non-uniform deformations due to local inhomogene-

ities and interactions with neighboring crystals are modeled. The resulting textures and microstructures at different strains are examined with regard to effect of including the additional systems, initial orientation of the single crystals, and stability of the cube orientation in a polycrystal. The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes. *This research was sponsored by the Division of Materials Science, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. The research was supported in part by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge National Laboratory and the Oak Ridge Institute for Science and Education. The authors acknowledge the use of the Intel Paragon XP/S 35 located in the Oak Ridge National Laboratory Center for Computational Sciences (CCS), funded by the Department of Energy's Office of Scientific Computing.

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MODELING OF THE RECRYSTALLIZATION TEXTURES OF AL-ALLOYS AFTER HOT DEFORMATION: *O. Engler*¹; *H. E. Vatne*²;

¹Los Alamos National Laboratory, Center for Materials Science, K765, Los Alamos, NM 87545 USA; ²Hydro Aluminum a.s. R & D Materials Technology, P.O. Box 219, Sunndalsorm N-6601 Norway

The recrystallization textures of most Al-alloys can be traced down to a growth selection of grains with an approximate 40% $\langle 111 \rangle$ orientation relationship out of a limited spectrum of preferentially formed nucleus orientations. To model this behavior, a function f^{nuc} describing the probability of nucleation and a function f^{grow} representing the growth probability must be derived. The resulting recrystallization textures are then simulated by multiplying these two probability functions. The probability of nucleation is given by the distribution of potential nucleus orientations, which have been determined by local texture analysis for the most important nucleation sites in commercially deformed Al-alloys, i.e. cube-bands, grain boundaries and second phase particles. If several nucleation sites are active simultaneously, the nucleation probabilities are weighted according to their respective contributions. For that purpose, the number of nuclei forming at each nucleation site is calculated according to an approach to derive the number of nuclei forming at the respective nucleation sites, which is based on experimental investigations on the nature and particularly on the evolution of the various nucleation sites. The probability of growth of the respective grains is associated with a 40% $\langle 111 \rangle$ transformation of the rolling texture. The paper describes the model approach of recrystallization texture simulation in Al-alloys and gives examples of recrystallization textures simulated regarding a variation of different metallurgical parameters to demonstrate the predictive power of the model.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Titanium Aluminides

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: Ian Baker, Dartmouth College, Thayer School of Engineering, Hanover, NH 03755 USA; Easo P. George, Oak Ridge National Lab., Metals & Ceramics Division, Oak Ridge, TN 37831-6093 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

Tuesday PM Room: Madrid
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Y. Kim, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, Ohio 45433 USA; P. J. Maziasz, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

2:00 PM

EFFECTS OF W, Mo AND B ADDITIONS ON THE STRUCTURE AND PROPERTIES OF LAMELLAR TiAl ALLOYS: *C. T. Liu*¹; *P. J. Maziasz*¹; *D. J. Larson*¹; ¹Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

This paper summarizes our recent work on the effects of alloying additions of W, Mo, and B on the microstructure and mechanical properties of lamellar TiAl alloys based on Ti-47Al-2Cr-2Nb (at. %). The dual-phase alloys were prepared by arc melting and drop casting, followed by hot extrusion at temperatures above T_{α} . The mechanical properties of the TiAl alloys were determined by tensile testing at temperatures to 1000°C, and their microstructure and solute distribution were characterized by TEM and atom probe field ion microscopy. The mechanical properties of the lamellar TiAl alloys are sensitive to their microstructural features, which are strongly influenced by the alloy additions at levels $< 2\%$. Boron is effective in refining lamellar structures, in addition to pin the colony boundaries by borides. Tungsten tends to segregate to α_2 / γ interfaces and stabilize fine lamellar structures. Mo promotes the formation of β phases, which harden the alloys at ambient temperatures but lower the strength at elevated temperatures. The correlation between the microstructure and mechanical properties will be discussed. Research was sponsored by the Division of Materials Science, US Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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ALLOY DESIGN OF TiAl-BASED ALLOYS: *Y.-W. Kim*¹; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433 USA

Significant improvements in tensile properties in wrought gamma TiAl alloys have recently been realized possible by refining lamellar grains and spacing. Grain refinement was achieved by various means, including: adding small amounts of boron, employing novel processing routes, and conducting heat treatments in two-phase fields. Lamellar spacing was refined mainly by innovative heat-treatment cycles involving rapid cooling through, or before, and after the lamellar formation process. These refined microstructures were also found to enhance creep resistance, fatigue strength and damage tolerance at temperatures below BDTT. The improvements, however, were made often at the expense of higher-temperature deformation/fracture resistance. The investigations at Wright Laboratory have indicated that the deficiencies can be significantly alleviated by alloy modifica-

tion and/or microalloying. This talk briefs the fundamentals for designing TiAl alloys, and then discusses the importance, as well as limitations, of combining microstructure control and chemistry modification with highlights from specific examples. *UES, Inc., Dayton, OH 45432, USA

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THE INFLUENCE OF SOLID SOLUTIONS ON FLOW BEHAVIOR IN GAMMA-TiAl: *C. Woodward*¹; S. A. Kajihara²; A. Kajihara²; S. I. Rao²; D. M. Dimiduk²; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright Patterson AFB, OH 45433-6533 USA; ²UES Inc., Materials Research Division, Dayton, OH 45432

Intermetallic alloys, such as TiAl and NiAl, are an important class of structural material due to their low density and excellent strength retention at high temperature. Modifications of alloy chemistry are often used to tailor the intrinsic flow behavior of these structural materials. Relaxed structures and energies, for intrinsic and substitutional point defects are calculated using a first principles plane-wave-pseudopotential method. Calculated defect energies are used to predict the density and site preferences of solid solutions (Si, Nb, Mo, Ta and W) in gamma-TiAl. Size and modulus misfit parameters are calculated and the interaction of these defects with active deformation modes are evaluated within anisotropic elasticity theory. The derived interaction strength is then related to solid solution strengthening for these defect centers. Solid solution effects for the binary alloy are in good agreement with experimental observations and several ternary compositions are predicted to have improved flow properties at intermediate temperatures.

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YIELD POINT PHENOMENA OF TiAl-BASED ALLOYS CONTAINING BORON: *T. T. Cheng*¹; R. Botten¹; P. Bate¹; H. A. Lipsitt¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham, B15 2TT United Kingdom

It is becoming standard practice to add boron to TiAl-based alloys for refining the grain size in the as-cast microstructure. In addition to the grain refinement. Kampe et al. (S.L. Kampe, P. Sadler, L. Christodoulou and D.E. Larsen, *Met & Mat. Trans. A*, 25 (10), 2181-2197 (1994).) have reported a strengthening effect greater than that could be accounted for by particulate strengthening due to borides. In this presentation the issue has been addressed by comparing tensile data obtained from a number of paired TiAl-based alloys whose compositions are identical except for the addition of boron. A yield plateau and strain aging phenomena have been observed in all those alloys which have a duplex microstructure and contain boron. It is proposed that boron additions result in solute locking of the dislocations and the results above the interpreted in that light.

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THE EFFECT OF CARBON CONCENTRATION ON PRIMARY CREEP OF AN INVESTMENT CAST Ti-47Al-2Mn-2Nb-0.8 WT.% TiB₂ ALLOY: *D. Y. Seo*¹; T. Everard¹; T. R. Bieler¹; P. A. McQuay²; ¹Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; ²Howmet Research Corp., 1500 S. Warner St., Whitehall, MI 49461-1895 USA

Creep experiments on an investment cast XDTM alloy in the as-HIPed duplex microstructure with carbon additions exhibit significantly improved primary creep resistance compared to the same alloy without carbon additions. The carbon levels examined are 0.006, 0.04, 0.07 and 0.11 wt%. The microstructure is investigated in selected tensile creep specimens deformed to 0.5% strain at 650°C and 276 MPa, 760°C and 815°C at 138 MPa. A ten-fold increase in time to 0.5% was observed under all conditions tested with a carbon concentration between 0.04 and 0.07 wt%, with little or no additional benefit from higher concentrations. Prior studies with high levels (0.1 wt%) of interstitial oxygen and nitrogen indicated only a two-fold increase in primary creep times to 0.5% strain (MRS symp. proc. vol. 460). The higher amounts of oxygen and nitrogen interstitials

also caused a higher volume fraction of the alpha-2 phase, and increased the contribution of mechanical twinning in lamellar refinement. Primary creep investigations on low carbon alloys indicate that primary creep is divided into two stages. The initial rapid process refines lamellar spacing up to strains between 0.2 and 0.5%, and it has a low activation energy. The second primary creep process has a higher activation energy, and no lamellar refinement is associated with this process (*Metal. Mater. Trans* 29A, p. 89). The effect of carbon on these two primary creep processes will be examined using measurements of initial lamellar spacing, and the changes in lamellar spacing after 0.5% strain.

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DISLOCATION-SOLUTE EFFECTS IN Al-RICH TiAl INTERMETALLICS: *J.M.K. Wiezorek*¹; M J. Mills¹; H. L. Fraser¹; ¹The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43221 USA

Single-phase TiAl alloys with slightly Al-rich compositions in the range of up to 56at.%Al exhibit both anomalous strengthening and serrated flow at intermediate temperatures. Traditionally and in analogy to intermetallics with the L1₂-structure the anomalous strengthening has been associated with an intrinsic effect, i.e. cross-slip locking. The operation of such a mechanism requires the activity of superdislocations with dissociated core configurations. However, for TiAl a yield strength anomaly is also observed when the predominantly activated deformation systems consist of ordinary dislocations with compact cores which are not prone to undergo Kear-Wilsdorf like locking. Furthermore, serrated flow has been observed for single-phase TiAl alloys. The latter constitutes an extrinsic effect, an interaction between mobile dislocations and thus far unidentified obstacles. The present transmission electron microscopy based study elucidates the micro-mechanisms underlying the observed mechanical behavior of single-phase Ti-(52-56at.%)Al alloys. The microscopic observations are discussed in relation to the mechanical behavior of the alloys.

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CREEP DEFORMATION OF TWO-PHASE TiAl ALLOYS CONTROLLED BY THE VISCOUS GLIDE OF INTERFACIAL DISLOCATIONS: *L. M. Hsiung*¹; T. G. Nieh¹; ¹Lawrence Livermore National Laboratory, Materials Science and Technology Division, P.O. Box 808, L-370, Livermore, CA 94551-9900 USA

A linear creep behavior, i.e. creep rate is linearly proportional to the applied stress, was observed when two-phase TiAl alloys with a refined lamella microstructure (thickness of γ' lamella: 100-300 nm) were creep deformed at stresses < 400 MPa and at temperatures above 650°C. TEM examinations of the corresponding deformation substructures revealed that interface sliding by a cooperative motion of interfacial dislocations is the predominant deformation mechanism. The measured activation energy (145-160 kJ/mol) of creep was found to be close to the activation enthalpy of oxygen diffusion in TiAl. This supports the idea that the viscous glide of interfacial dislocations dragged by interstitial atoms controls creep in this regime (<400 MPa, T > 650°C). A constitutive equation for the linear creep behavior is accordingly derived.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Fiber Reinforced Composites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: Bhaskar S. Majumdar, UES, Inc., Dayton, OH 45432-1894 USA; S. Krishnamurthy, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Tuesday PM Room: Paris A
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Andy Rosenberger, Air Force Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH USA USA

2:00 PM INVITED PAPER

MODELLING AND CHARACTERISATION OF FATIGUE CRACK INITIATION AND GROWTH IN SiC FIBRE REINFORCED Ti MMCs UNDER CYLOADING: P. Bowen¹; J. Liu¹; W. Ding¹; D. Parrott¹; N. Wang¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

The initiation and growth of fatigue cracks in SiC fibre reinforced Ti MMCs differs from that in monolithic alloys. Minor defects and damage can arise from a local fibre failure and/or defects at the ends of fibres. Thus, fatigue crack initiation in the matrix of the composites can be facilitated by such defects and damage. Observations have been made both for selectively reinforced Ti MMCs containing artificially embedded fibre ends and modelling based on a 3-D finite element method shows that the local stress at the ends of fibres is critical. For fatigue crack growth, in selectively reinforced (clad) testpieces, fatigue cracks can either grow into the composite so that fibre bridging can occur or along the fibre-matrix interface thus preventing fibre bridging. To characterise this latter mode of failure has necessitated extensive experimental and modelling studies. In uniformly reinforced Ti MMCs, fibre bridging can always occur and to model the bridging effect, in-situ SEM tensile tests have been conducted to provide insight into the physical nature of bridging. A combination of modelling and in-situ SEM measurements has been used to predict accurately the transition from crack arrest to catastrophic specimen failure.

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STRESS RATIO AND TEMPERATURE EFFECTS ON CRACK PROPAGATION IN A UNIDIRECTIONALLY REINFORCED ORTHORHOMBIC TITANIUM MATRIX COMPOSITE: Reji John¹; Jay R. Jira²; ¹Air Force Research Laboratory (AFRL/MLLN), Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433 USA; ²University of Dayton Research Institute (UDRI), Dayton, OH 45469 USA

Titanium matrix composites (TMC) based upon orthorhombic "O" phase titanium aluminide and high strength Ultra SCS™ fibers are being considered for applications in rotating engine components operating at temperatures up to 650C. This presentation discusses the results of the experimental and analytical investigation of fatigue crack propagation in [0]8 Ultra SCS™ /Ti-22Al-26Nb. The tests for crack growth perpendicular and parallel to fibers were conducted at 21, 482 and 649C with stress ratios of 0.05, 0.5 and 0.8. Baseline tests were also conducted on fiberless Ti-22Al-26Nb under similar loading conditions. During some of the tests on the composite, crack

opening displacement profiles were measured to verify and/or calibrate the fiber bridging models. The ability of the shear lag models to predict bridged crack growth perpendicular to fibers in Ultra SCS™ /Ti-22Al-26Nb will be discussed. The correlation between crack growth parallel to fibers in Ultra SCS™ /Ti-22Al-26Nb and that in fiberless Ti-22Al-26Nb will also be discussed.

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ANALYSIS OF BRIDGED SURFACE CRACKS IN FIBER REINFORCED METAL MATRIX COMPOSITES: G. Bao¹; Z. Lin¹; ¹The Johns Hopkins University, Baltimore, MD 21218 USA

In this work, a systematic study of fracture of fully bridged 3D surface cracks is performed using a finite element method. The surface cracks under study include semi-circular, semi-elliptical, quarter-circular and quarter-elliptical; both finite and infinite body problems are considered. A non-dimensional loading parameter is identified which combines the applied load, the material properties of the composite, and the interfacial sliding shear stress. The mode I stress intensity factors at the crack tip and the maximum stress in the bridging fibers are calculated for surface crack specimens with different crack aspect ratio, crack depth, specimen width, and the effective applied load. Numerical curve fit solutions are developed for the crack tip stress intensity and the maximum fiber stress of fully bridged 3D surface and corner cracks based on the finite element solutions. An engineering model is further developed to predict the crack-tip stress intensity factors of partially bridged surface cracks based on the curve fit functions for fully bridged and unbridged surface cracks. The model is subsequently validated by comparing the model predictions with experimental results for fatigue crack growth of surface cracks with an initial unbridged notch.

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DAMAGE AND DEFORMATION PREDICATION OF [0] TITANIUM MATRIX COMPOSITES UNDER FATIGUE-DWELL LOADING: D. J. Buchanan¹; N. E. Ashbaugh¹; A. H. Rosenberger²; ¹Air Force Research Laboratory (AFRL/MLLN), Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; ²University of Dayton Research Institute, 1031 Irving Ave., Dayton, OH 45419-0218 USA

A one-dimensional deformation and rupture model for a unidirectional continuously reinforced titanium matrix composite, SCS-6/Ti-6Al-4V, has been extended for application to thermomechanical fatigue spectrum loading. A refined constitutive model for the Ti-6Al-4V matrix has been shown to be more accurate than the Bodner-Partom model for predicting creep and relaxation behaviors. The deformation model also incorporates a two parameter Weibull distribution to characterize the SCS-6 fiber strengths. The Weibull parameters, m and so, were deduced for an entire panel from a single test specimen. The deformation, accumulation of fiber failures and rupture life for other specimens in the panel were predicted with the deduced values of m and so. Acoustic emission data were collected during the tests and provided the history of the damage accumulation to compare with the predicted fiber failures from the model. The predictions accurately captured the creep and relaxation behavior and predicted time to failure within a factor of two.

3:40 PM BREAK

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S/N BEHAVIOUR AND FIBRE STRENGTH DEGRADATION FOR Ti-MMCs: S. Zamperini¹; P. McDonnell¹; H. Mori²; P. Bowen¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT UK; ²The University of Tokyo, Research Centre for Advanced Science and Technology, 4-6-1 Komaba, Meguro-ku, Tokyo 153 Japan

The total life of TiMMC's have been studied to determine the extent of fibre degradation due to fatigue and elevated temperature exposure. In all cases fibre strength distributions have been com-

pared with the unfatigued as-received composite. Fibre failure during the test has been monitored by means of acoustic emission. Of particular importance is to detect whether premature fibre failure occurs during the early stages of the total life of the composite and this issue will be addressed in detail. The effect of fatigue on the fibre strength distribution has been evaluated by single fibre tests. The results of the single fibre test have been analysed with Weibull weakest-link theory. As well as fibre strength degradation due to fatigue, effects of the manufacturing process of the composite on the virgin fibres and effects of the etching solution used to remove fibres from the composite have been studied carefully. Within the overall study effects of fibre volume fraction on total life has been addressed and rationalised. Further subtle effects relate to the degree of fibre strength degradation when linked to the combination of stress range and number of cycles applied to particular composites and these issues will also be discussed.

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EVALUATING DAMAGE MECHANISMS IN SCS-6/TI-15-3 CAUSED BY ELEVATED TEMPERATURE FATIGUE: *J. R. Calcaterra*¹; *S. Mall*²; ¹Department of the Air Force AFRL/VAVS, Building 63, 1901 Tenth Street, Wright-Patterson AFB, OH 45433-7605 USA; ²Department of the Air Force, AFIT/ENY Building 640, 2950 P. Street, Wright-Patterson AFB, OH 45433-7765 USA

The majority of experimental fatigue studies performed on Titanium Matrix Composite (TMC) systems have taken the specimens to complete failure. Unfortunately, this type of study provides little information as to the progression of damage mechanisms or the residual strength degradation of the material. To address this concern, an experimental test program has been conducted on SCS-6/Ti-15-3 composites with laminate orientations of [0]8, [0/90]2s, and [0/45/90]s. The main goal of this research was to determine the effect of strain-controlled, fully-reversed, elevated-temperature fatigue on the degradation of residual strength in TMCs. Specimens with varying fiber volume fractions were subjected to isothermal fatigue and residual tensile strength tests, all at 427°C. Specimens were cycled until they had reached either 50 or 75 percent of their fatigue life. After cycling, the specimens were statically tested until failure. Extensive post-test microscopic examination of the specimens provided information as to the progression of damage mechanisms during fatigue. Results indicate the laminate orientation is the primary factor controlling damage progression morphology. The results also show that fiber volume fraction has an effect on both strain-controlled fatigue life and residual strength degradation.

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EXTRINSIC AND INTRINSIC FACTORS INFLUENCING FATIGUE CRACK GROWTH BEHAVIOUR OF SiC FIBRE REINFORCED Ti MMCs: *J. Liu*¹; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

The improved fatigue crack growth resistance of Ti MMCs derives from fibre bridging, and the magnitude of this resistance is strongly affected by extrinsic and intrinsic factors. The aim of this paper is to examine the effects of extrinsic factors, and the intrinsic factor of fibre strength. The results show that under the same initial K_{max} values, small initial notches under high stress can be more damaging than large initial notches under low stress. In addition a wider specimen can always sustain higher initial K_{max} values without failure, and this can be explained by a modified bridging model. Single fibre tensile tests have been conducted on fibres extracted from composite specimens to establish the degradation of fibre strength under fatigue. The influence of fibre failure on fatigue crack growth has been monitored by a combination of acoustic emission (AE) and direct current potential drop (DCPD) techniques. The influence of reduced fibre strength in producing poorer fatigue crack growth resistance has thus been quantified in this study.

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MICROSTRUCTURAL ANALYSIS OF CVD-SiC MONOFILAMENTS FOR METAL MATRIX COMPOSITES: *K. L. Dyos*; *R. A. Shatwell*

A comparative study of commercially available and experimental monofilaments has been carried out using fractography techniques, micro-indentation, X-ray diffraction, scanning electron microscopy, wavelength dispersive analysis and transmission electron microscopy. The fracture surfaces have been examined, and their behavior correlated with their microstructures. Initiation of fracture has been identified at various interfaces. The strength has been linked to variations in the SiC microstructure, which affect the fracture toughness.

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DAMAGE MECHANISMS AND FIBER FABRIC ORIENTATION EFFECTS ON MECHANICAL BEHAVIOR OF A NEXTEL|312(BN)/BLACKGLAS| LOW COST CERAMIC COMPOSITE: *Wei Zhao*¹; *Peter K. Liaw*¹; *Ronald Belardinelli*²; *David C. Joy*³; *Charlie R. Brooks*¹; *N. Allen Yu*⁴; *Carl J. McHargue*¹; ¹University of Tennessee at Knoxville, Dept. of Materials Science and Engineering, Dougherty Engr. Bldg., 1512 Middle Dr., Knoxville, TN 37996-2200 USA; ²Northrop Grumman, 9314 W. Jefferson Blvd., Dallas, TX 75211 USA; ³University of Tennessee, Dept. of Biochemistry, Cellular and Molecular Biology, 232 Sci. & Engr. Facilities, Knoxville, TN 37996 USA; ⁴University of Tennessee, Dept. of Mechanical and Aerospace Engineering and Engineering Sciences, 323 Perkins Hall, Knoxville, TN 37996 USA

In this paper, the damage mechanisms and fiber fabric orientation effects on the mechanical behavior of a Nextel 312(BN)/Blackglas composite are investigated. The laminated composite is a newly developed advanced low cost ceramic composite. The Blackglas matrix is pyrolyzed with a crowfoot satin Nextel 312 ceramic fiber (3M product) as reinforcements. A four-point-bend testing is performed using a Material Testing System (MTS) at room and elevated temperatures. Samples are cut with fiber fabric at angles of 30, 45, 60 to the fiber longitudinal direction. A scanning electron microscopy (SEM) is employed to examine microstructures, fiber/matrix interface, and fracture surfaces. Chemical variations after oxidation are detected by a SEM energy dispersive X-ray spectroscopy (EDXS) line-scan technique. A quantification model based on a classical laminated plate theory is also provided to rationalize the experimental results. The present work is supported by National Science Foundation under a contract number, EEC-9527527, with Mary Poats as the program manager.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - II Heat Capacity & Alloy & Structure

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee

Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

Tuesday PM

Room: Rome

October 13, 1998

Location: O'Hare Hilton Hotel

Session Chair: Michael V. Nevitt, Clemson University, Dept. of Physics & Astronomy, Clemson, South Carolina 29634 USA

2:00 PM INVITED PAPER

LOW-TEMPERATURE HEAT CAPACITY AS RELATED TO MAGNETIC PHENOMENA: *Vitalij K. Pecharsky*¹; ¹Iowa State University, Ames Laboratory, 242 Spedding, Ames, IA 50011-3020 USA

The heat capacity is a fundamental thermodynamic property of solids that allows experimental measurements of various contributions to the total entropy. The experimental magnetic heat capacity and the magnetic entropy yield important information about magnetic phenomena. A study of the heat capacity in magnetic fields allows one to clarify the nature of magnetic phase transitions, even though the behavior of the magnetic heat capacity does not disclose details of the magnetic structure. Low-temperature (1.5 to 350 K) heat capacity data measured in zero magnetic fields and in fields up to 10 T for a variety of magnetic systems including ferromagnets, antiferromagnets and spin glasses show distinct differences in the observed heat capacity. The discussion will include the use of the heat capacity as a valuable tool to: 1) determine the magnetic ordering temperature; 2) to refine the magnetic phase diagram; 3) to establish whether the magnetic phase transition is of the first or the second order; and 4) to estimate whether the observed magnetic phenomenon occurs in the matrix phase or in the impurity phase. This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences under Contract No. W-7405-ENG-82.

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CONCEPTS IN THE STRUCTURAL CHEMISTRY OF STABLE AND METASTABLE INTERMETALLICS: *Bill C. Giessen*¹; ¹North-eastern University, Barnett Institute and Dept. of Chemistry, 360 Huntington Ave., 341 MU, Boston, MA 02115 USA

Through his and his students' encyclopedic studies of alloy phase diagrams, especially those of ternary transition metal systems, Paul Beck contributed early to the present immense body of knowledge on alloy phases, especially their occurrence and structural classification. We trace the path of increasing understanding from phenomenological beginnings of alloy phase classification through the systematic structural compilations, the emphasis on distance and size relationships (Pearson), "electron lattice" concepts (Schubert), the pseudopotential approach (Heine and Weaire), quasichemical considerations and structure maps (Miedema; Phillips and Rabe) to first principles calculations (Pettifor), considering metastable alloy phases as well as equilibrium ones and extrapolating to future progress in the field.

3:10 PM INVITED PAPER

TRANSITION ELEMENT ALLOYS AS IMPLANTABLE BIOMATERIALS: *Denes I. Bardos*¹; ¹Smith & Nephew, Inc., Scientific Affairs, 1450 Brooks Rd., Memphis, TN 38116 USA

All current high quality implants are fabricated from transition element alloys. Phase relationships, re crystallization, sigma phase and other intermetallics as well as other metallurgical advances pioneered by Dr. Beck enabled the author to contribute to the development of new and improved biomaterials for orthopaedic surgical implant applications. The high strength thermo-mechanically formed Co-Cr-Mo alloy is now the choice for total hip replacement (arthroplasty), Ti-Zr-Hf alloys offer far superior biocompatibility than any other alloy, other Ti alloys are the safest hypo allergenic materials. A special medical grade Fe-Cr-Ni-Mo type stainless steel has been improved for trauma applications with unprecedented clinical results. Professor Beck's legacy lives on to reduce pain and restore mobility to millions of orthopaedic patients.

3:45 PM BREAK

4:05 PM INVITED PAPER

"METALLIC" ZINTL PHASES: ARE THEY POSSIBLE?: *Gordon J. Miller*¹; ¹Iowa State University, Department of Chemistry, 3659 Gilman, Ames, IA 50011 USA

Zintl phases are semiconducting compounds that bridge intermetallics and valence compounds: they are formed from elements with (semi)metallic character yet build structures according to the octet rule. Recent experimental and theoretical efforts with ternary aluminides show that intermetallic structures exist which have no energy gap and yet show a distinct separation between bonding and antibonding states. Examples from alkaline earth/Li/Al (CaLi(x)Al(2-x)), alkaline earth/Cu/Al (AECu(5-x)Al(8+x)), and rare earth/Au/Al (LnAu(x)Al(4-x)) demonstrate how the observed compositions, structures, atomic arrangements, and properties are influenced by the electronic structure. Results from synthetic, structural, and theoretical investigations will be presented. In fact, for these types of compounds, theoretical calculations are imperative as no simple chemical bonding picture exists for their description. This work is supported by the National Science Foundation under DMR-96-27161.

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PHASE STABILITY, POINT DEFECTS, AND FRACTURE TOUGHNESS IN Nb-Cr-Fe TERNARY SYSTEMS: *J. H. Zhu*¹; *P. K. Liaw*¹; *L. M. Pike*²; *C. T. Liu*²; ¹University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; ²Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

The phase stability, point defects, and fracture resistance in ternary Nb-Cr-Fe Laves phase system are studied in this paper. Our previous work indicates that when the atomic size ratios are kept identical, the average electron concentration factor, e/a , is the dominating factor in controlling the phase stability of NbCr₂-based transition-metal Laves phases. In order to verify the e/a correlation with phase stability experimentally, addition of Fe was selected to replace Cr in the NbCr₂ Laves phase. The boundaries of the C15/C14 transitions were precisely determined and the e/a ratios corresponding to the different Laves polytypes were calculated and compared to the previous prediction. It was found that the electron concentration and phase stability correlation is obeyed in the Nb-Cr-Fe system. The changes in lattice constant, hardness, and fracture toughness were also determined as a function of the Fe content, which were discussed in light of the phase stability difference of the alloys. The point defects of the binary NbFe₂ and NbCr₂ and ternary Nb(Fe,Cr)₂ were also determined and anti-site substitution was found to be the major defect mechanism in these Laves phases.

PROCESSING OF HIGH TEMPERATURE ALLOYS: Session I

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Thomas Zogas, Carpenter Technology Corp., Reading, PA 19612-4662 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

Tuesday PM Room: Paris C
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Thomas Zogas, Carpenter Technology Corp., Reading, PA 19612-4662 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

2:00 PM

THE GROWTH-PATH METHOD FOR PREDICTION OF SPURIOUS GRAIN NUCLEATION: *Ralph E. Napolitano*¹; ¹National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD USA

Predictive techniques for the assessment of potential yield are essential for the cost-effective design and production of superalloy single-crystal castings. Such castings can be rendered defective by many types of crystal imperfections, several of which are difficult to control during processing. The tendency for the formation of these grain defects must be evaluated and minimized during the development of a casting design/process. Currently, yield maximization often involves several design cycles where castings are produced and evaluated experimentally to provide a basis for design modification. The high cost of this approach is driving the development of predictive techniques which can be used by casting designers to assess defect likelihood without the need for test castings. The growth-path technique, presented here, is a method for the quantitative assessment of the potential for spurious grain nucleation in a single-crystal casting. Ledge and platform areas of single-crystal turbine blades are especially prone to such defects. For a casting of complex geometry, the model must predict whether the undercooling in any part of the casting becomes sufficiently large to induce nucleation, before the dendritic structure growing from the grain selector reaches that point. The growth-path method uses thermal simulation results, a kinetic model for dendrite tip and branch growth, and a specified dendrite crystal orientation to determine the path by which the dendritic structure reaches any point or region in the casting. Using thermal simulation results and a nucleation model which may be based on actual experimental data, the nucleation tendency at all points along the path can be computed. The method is demonstrated for a René N5 casting and compared with experiment.

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DEVELOPMENT OF A P/M SUPERALLOY CONSOLIDATION PROCESS FOR CONVENTIONAL FORGING: *X. Pierron*¹; T. Banik¹; G. E. Maurer¹; ¹Special Metals Corporation, 4317 Middle Settlement Road, New Hartford, NY 13413-9576 USA

Recent investigation has identified hot isostatic pressing (HIP) conditions of P/M Superalloys that significantly enhance hot workability of the consolidated materials. This process incorporates HIP'ing of the powder at temperatures close to the liquidus of the alloy. It has been tested on UDIMET Alloy 720 and René 95 and has resulted in superior hot workability compared to that of conventional Superalloy powder metallurgy. Use of this method could result in reducing the cost of P/M Superalloys with the ability to produce large components with uniform microstructures and properties by subsequently using conventional forging practices other than isothermal forging. Results of the study on the effect of process parameter and composition on the hot workability of UDIMET Alloy 720 and René 95 will be presented. In addition, mechanisms leading to the enhanced workability observed in the P/M alloys consolidated by this new process will be proposed.

2:40 PM
MANUFACTURING OF IRON ALUMINIDE SHEETS BY THERMOMECHANICAL PROCESSING: *S. C. Deevi*¹; M. R. Hajaligol¹; V. K. Sikka²; C. Scorey³; ¹Philip Morris, Research and Development Center, 4201 Commerce Road, Richmond, VA 23261 USA; ²Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6083 USA; ³Ametek Specialty Metal Products Division, P.O. Box 5807, Wallingford, CT 06492 USA

An innovative combination of powder metallurgy and thermomechanical processing were used to manufacture FeAl alloy intermetallic sheets. Green sheets of FeAl were obtained by roll compaction of water-atomized FeAl powder with a polymeric binder. Roll-compacted green sheets were debindered and partially sintered prior to cold rolling through tungsten carbide rolls. Cold rolling lowered the thickness, reduced the level of porosity, and heavily work hardened the FeAl sheets. Several intermediate annealings at or above 1100 C were found to be necessary to relieve the stresses (from work hardening) prior to rolling the sheets to a final thickness of 0.20 mm. Thermomechanical processing of cold-rolled sheets allowed the commercial manufacture of FeAl intermetallic sheets without the neces-

sity of hot rolling of a cast FeAl ingot. Mechanical properties of FeAl sheets are comparable to the properties of hot-extruded FeAl alloys and far superior to those of cast iron aluminides.

3:00 PM
DESIGN OF CARPENTER'S NEW 20-TON TILT-CHAMBER VIM FURNACE FOR MELTING OF PREMIUM WROUGHT SUPER-ALLOYS: *W. E. Johnson*¹; D. D. Wegman¹; ¹Carpenter Technology Corporation, P.O. Box 14662, Reading, PA 19601 USA

A new ALD Vacuum Technologies 20-ton tilt-chamber VIM furnace was commissioned in February 1997. The addition of this largest and most advanced tilt-chamber furnace in the world has enabled Carpenter to reliably manufacture premium VIM product in excess of 100 tons/day in order to meet the demand of the superalloy industry. Significant design and operational aspects which enhance the productivity, reliability, and product quality of the furnace to be reviewed are: 1) the dual melting/casting chamber design which allows many tasks to be performed simultaneously, 2) the tilt melt chamber utilizing external cable connections for quick transfer of furnace vessels, 3) the long launder used for metal transfer and inclusion flotation/removal and complementary video camera system, 4) the 5 MW power supply with supplementary 3-phase inductive stirring capability, 5) the Saveway lining monitor system providing real time measurement of metal penetration through the refractory lining, 6) the vacuum filter system to capture dirt and extend vacuum pump life, 7) the material recharge bucket system allowing for maximization of recharge weight and minimization of required recharges within a heat. The furnace has already been qualified to supply premium wrought product to many superalloy customers with the remaining qualifications in process. Multi disciplinary teams continue to work on improving the furnace productivity, reliability, and product quality.

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ALUMINUM VAPORIZATION KINETICS DURING SKULL MELTING OF TITANIUM ALUMINIDES : *Linda L. Rishel*¹; Thomas Kelly²; Tresa M. Pollock¹; Alan Cramb¹; ¹Carnegie Mellon University, MSE Department, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; ²General Electric Aircraft Engines, One Neumann Way, Cincinnati, OH 45215 USA

Gamma titanium aluminides are currently being considered for aerospace and automotive components. Presently, gamma titanium aluminide components are cast after vacuum arc remelting or vacuum induction skull remelting. Control of melt chemistry, especially aluminum and other volatile elements, during melting in vacuum is critical to the successful implementation of these materials. In this work a series of experiments were conducted to determine the vaporization kinetics of aluminum from gamma titanium aluminides in the vacuum induction skull remelting process. Aluminum loss will be presented for different melt durations and vacuum levels. Rate controlling steps for the vaporization of aluminum from the meltbath will be discussed and a kinetic model for the vaporization of aluminum from the meltbath will be presented.

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CHARACTERIZATION OF SPRAY FORMED ALLOY 718 BILLET: Lee Shaw²; Graham Oakes²; Larry Jackman¹; *Chris O'Brien*¹; Robin Forbes Jones¹; Dick Kennedy¹; ¹Allvac-SMP, P.O. Box 5030, 2020 Ashcraft Avenue, Monroe, NC 28110-0530 USA; ²Allvac-SMP

Spray forming of superalloy billets by the Osprey Process is attractive because it offers the possibility of cost savings relative to conventional cast/wrought and powder metallurgy processing routes. Also, benefits may be derived from the unique structure that is generated by spray forming. In this presentation spray formed alloy 18 billet, 203 mm (8 inches) in diameter is characterized and compared to cast/wrought billet of the same diameter. The spray formed billet was produced from a sprayed preform 419 mm (16.5 inches) in diameter which was also characterized. Structures were investigated by macro etching, optical microscopy, and scanning electron micros-

copy. Hot workability was determined by Gleeble testing and various mechanical properties were measured. Thermal stability was also evaluated and electron beam buttons were melted to evaluate cleanliness. Several unique features were observed for the spray formed product.

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THE IMPORTANCE OF FINISHING TEMPERATURE CONTROL DURING HOT WORKING OF SUPERALLOYS: *R. B. Frank*¹; ¹Carpenter Technology Corporation, P.O. Box 14662, Reading, PA 19612-4662 USA

Because superalloys are designed for high strength at elevated temperatures, these alloys are very resistant to deformation during hot working operations. Because of this stiffness and typically narrow hot working temperature ranges, the temptation is to hot roll or forge as hot as possible to avoid reheating, surface tears, and wear of hot working equipment. However, finishing at lower temperatures within the typical hot working range of 1750-2100 F is often necessary to avoid hot tearing or to meet grain size and mechanical property requirements. The importance of finishing temperature control during hot working of several Carpenter superalloys including Pyromet alloys 718, A-286, 41 and 751 as well as corrosion-resistant alloys MP159 and Custom Age625 PLUS will be discussed.

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MODELING SPRINGBACK IN SUPERALLOY SHEET FABRICATION: *R. A. Jaramillo*¹; *B. A. Baker*¹; ¹Inco Alloys International Inc., 3200 Riverside Drive, Huntington, WV 25705-1771 USA

Precision forming of aerospace and automotive components is becoming increasingly critical as performance criteria become more demanding. It was, therefore, deemed advantageous to develop a more precise means of determining springback in superalloy sheet, particularly, because of the differing cost and tensile properties of superalloys compared to ferritic and stainless steels. A model was developed which incorporates the work-hardening behavior of a material into the springback model of Queener and De Angelis. Several work-hardening models were evaluated from which the Kock's differential equation was selected for best fit. Experimental bend tests using INCONEL alloy 625LCF were performed and compared to calculated values. Strengths and limitations of the calculation are discussed. INCONEL and 625LCF are trademarks of the Inco group of companies.

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THE ELEVATION OF THE INCIPIENT MELTING TEMPERATURE OF STELLITE 21 ALLOY THROUGH NITROGEN ALLOYING: *Wade L. Karlsen*¹; ¹Helsinki University of Technology, Laboratory of Engineering Materials, Otakaari 4, P.O. Box 4100, Espoo, FIN 02015 HUT Finland

The Co-Cr-Mo alloys such as Stellite 21 were historically used in structural components for high-temperature environments such as jet-engines. However, now more well known for its application in demanding biomedical components, it has suffered from incipient melting during high-temperature sintering heat-treatments associated with porous coating of implants. As a part of a broader study, this paper discusses how alloying a Stellite 21 with nitrogen has elevated the incipient melting temperature of the alloy from below 1240°C, to above 1260°C. Findings indicate that as the nitrogen content is increased, a Cr sub 2 N second phase is favored over the traditional Cr sub 23 C sub 6 carbide, leading to the subsequent elevation of the incipient melting temperature. Besides its significance to the biomedical component fabrication industry, this finding may also be of renewing interest to high-temperature applications of this alloy.

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PROCESSING OF HIGH TEMPERATURE ALLOYS BY LASER-ENGINEERED NET-SHAPING|: *Richard J Grylls*¹; *T J Lienert*¹; *D M Keicher*²; *H L Fraser*²; ¹The Ohio State University, Department of

Materials Science and Engineering, 2041 College Road, Columbus, Ohio 43210 U.S.A.; ²Optomec Design Company, 2701-D Pan American Freeway, NE, Albuquerque, NM 87107 U.S.A.

Laser-Engineered Net-Shaping (LENS|) is a novel process for direct fabrication of metal powders to form net-shapes direct from a CAD model. A focussed laser provides localized melting of a stream of metal powder, and the beam is rastered such that a large part can be built-up progressively. A wide range of metals have been processed in this way, from aluminum to tungsten. One advantage of the process is that melting is containerless, with surface tension providing support for the molten metal. Also, the small heat input and rapid heat extraction can give cooling rates of up to 10⁵ Ks⁻¹, such that the benefits of rapid solidification may be achieved in a bulk structure. In this presentation the application of LENS| to a variety of high-temperature alloys, such as nickel, titanium and niobium-based alloys, will be discussed. Detailed microstructural characterization has been used to investigate the effect of the processing technique on the final product. The advantages and disadvantages of this technique will be addressed. LENS| is a trademark of Sandia National Laboratories.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Metal-Matrix Composites - I

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Tuesday PM Room: Chicago
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Dr. M. Manoharan, Nanyang Technological University, Division of Materials Engineering, Singapore 639798; Dr. G. S. Bhat, University of Tennessee, Knoxville, TN

2:00 PM INVITED PAPER

A COMPARATIVE STUDY ON THE WORK HARDENING BEHAVIOUR OF AN UNDERAGED AND AN OVERAGED SiC REINFORCED ALUMINUM METAL - MATRIX COMPOSITE: *M. Gupta*¹; *M. Manoharan*²; ¹National University of Singapore, Department of Mechanical and Production Engineering 639798 Singapore; ²Nanyang Technological University, Singapore, Division of Materials Engineering, School of Applied Science 639798 Singapore

An understanding of the work hardening behaviour of particulate reinforced metal matrix composites is crucial in optimizing the parameters for deformation processing of these materials. The microstructure and mechanical properties of these composites can be altered by a suitable aging treatment. The precipitation hardening mechanisms can be changed by moving from an underaged to an overaged microstructure. In particulate reinforced composites, however, the strength and ductility, as measured by strain to fracture are determined predominantly by the effects of the reinforcement. Thus, a model for predicting the mechanical properties of particulate reinforced composites should be able to do so for all aging conditions. In this paper a modified continuum model is used to relate the work hardening behaviour of the composite to microstructural parameters. The model is also used to predict the fracture strain of the composite for an underaged and an overaged condition. The model is shown to predict the fracture strain of the composite quite accurately for both aging conditions.

2:30 PM INVITED PAPER

MIXED-MODE TOUGHNESS OF A 6061 ALUMINUM ALLOY BASED COMPOSITE: M. Manoharan¹; L. M. Tham²; R. K. Oruganti¹; H. P. Seow¹; M. Gupta²; ¹Nanyang Technological University, Division of Materials Engineering, School of Applied Science, Singapore 119260; ²National University of Singapore, Department of Mechanical and Production Engineering 119260 Singapore

The advancement of technology has increased the need for materials which have exceptional performance characteristics. In many situations where the structural integrity is important, the fracture toughness of the material is crucial in determining the suitability of a particular material. Traditionally fracture toughness has been determined under tensile or Mode I conditions. However, cracks in real materials can be subjected not just to tensile stresses but to complex stress states so that the development of suitable parameters to characterize mixed-mode crack initiation and propagation is important in the evolution of suitable design criteria. Further, observations indicate that initially flat cracks in some tough materials tend to reorient themselves to oblique planes during growth. For these materials, crack propagation can be said to occur under combined-mode conditions. This paper will outline some recent work on the mixed mode behaviour of a 6061 aluminum alloy based composite.

3:00 PM INVITED PAPER

THE ROLE OF FABRICATION METHOD ON STRENGTHENING AND TOUGHNESS OF A PARTICULATE METAL MATRIX COMPOSITE: M. J. Hadianfard¹; ¹Shiraz University, Department of Materials Science, School of University, Shiraz Iran

Discontinuously reinforced metal matrix composites (Mmes) have considered as materials for demanding conditions because of their superior stiffness, higher specific mechanical properties and higher wear resistance. The properties of (Mmes) are largely influenced by factors such as; matrix and reinforcement properties, reinforcement shape, size and volume fraction, and fabrication methods. The effects of four different fabrication methods; powder metallurgy & hot extrusion, on strengthening and toughness of a 6061 Al reinforced with SiC particles were investigated by using mechanical sand electron microscopy testing. Results show extensive effect of fabrication method on properties of the MMC. The results are discussed according to microstructural and metal/particles interfacial factors.

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BLADE FABRICATION PROCESS OF TITANIUM MATRIX COMPOSITES (TMC): T. Yamada¹; T. Tsuzuku¹; Y. Kawachi¹; K. Yasuhira¹; ¹Research Institute of Advanced Material Gas-Generator, 4-2-6 Kohnata, Bunkyo-ky, Tokyo 112 Japan

Continuous fiber reinforced titanium matrix composites (TMC) are attractive as one of the potential structural materials for aerospace applications, because of their high specific strength and stiffness. However, TMC parts are not yet put into practical use due to an enormous production cost resulting from process limitation requiring preliminary forming and elaborate tooling for consolidation. In order to reduce the production cost, superplastic TMC sheets (SiC/Ti-4.5Al-3V-2Mo-2Fe) have been developed, and deformation properties, mechanical properties and cavitation behavior have been investigated. SiC/Ti-4.5Al-3V-2Mo-2Fe composites were demonstrated to be superplastic with m-value of 0.58 and to show elongation exceeding 69% in true strain, at the temperature of 1048°K and strain rate of 5x10⁻⁵ s⁻¹. The blade-shaped model was formed successfully out of the newly developed TMC sheet by means of Argon-gas-pressure forming technique. Diffusion bonding techniques of attaching root blocks to deformed TMC sheets are also discussed.

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SUPERPLASTIC FOAMING OF Ti AND Ti-6Al-4V: Naomi G. Davis¹; David C. Dunand¹; ¹Northwestern University, Materials Science and Engineering, 2225 N. Campus Drive, Room 2036, Evanston, IL 60208 USA

Pure titanium and the titanium alloy Ti-6Al-4V were foamed by expansion of small, high-pressure, argon-filled pores. To delay cell wall busting, the foaming was carried out under conditions where the matrix exhibited transformation superplasticity. This mechanism does not depend on the grain size as in fine grained superplasticity but, instead, relies on internal stresses due to the density mismatch between the a and the b phases that occurs during the allotropic phase transformation. Elastic constants (Young's modulus, shear modulus and Poisson's ratio) were measured as a function of porosity and correlated to initial and foamed pore morphology.

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OPTIMIZATION OF ARTIFICIAL AGEING RESPONSE OF AL-SIC(P), METAL MATRIX COMPOSITE SYSTEM: D. Sood¹; G. Grewal¹; H. R. Anand²; ¹Thapar Corporate Research and Development Centre, Patiala India; ²Thaspar Institute of Engineering and Technology, Patiala India

In the present study, a systematic investigation is undertaken to parametrically model the artificial ageing characteristics of a composite system based on 2024 type aluminum alloy matrix reinforced with particulate silicon carbide. The composite system, which comprises of a series of compositions with varying volume fraction and mean particle size of the reinforcing phase, has been fabricated using a novel two-stage technique comprising of mushy-zone hot pressing followed by extrusion in a heated die. The entire study has been conducted in the framework of an analytical methodology based on statistical experiment design and two way analysis of variance with interactions effects included. Based on a rigorous analysis of the data obtained, a response surface, for the various optimal ageing parameters, is derived in the two variable design space of the volume fraction and mean particle size of reinforcing phase. Further, the ageing response of the metal matrix composite system is correlated with the evolving microstructure using optical metallography and scanning electron microscopy.

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DEVELOPMENT OF ALUMINUM-CORUNDUM METAL-MATRIX COMPOSITES: K. V. Sharma¹; ¹UVCE, Department of Mechanical Engineering, K.R. Circle, Bangalore, Karnataka 560001 India

The main objective of the paper is to characterize the aluminum-corundum particulate metal matrix composites. Addition of aluminum is expected to yield properties better than that of alloy. The mechanical properties better than that of alloy. The mechanical properties viz, ultimate tensile strength, compressive strength, wear resistance, etc. are evaluated. Aluminum corundum material will be prepared through liquid metallurgy technique using the vortex method, in which, a vortex is created in the melt through an impeller rotated at 400 RPM. The uncoated but preheated corundum particulates will be added into vortex in the melt & stirred thoroughly before pouring into molds. The specimen is tested for above properties and results presented. Work is already in progress and results are expected soon.

5:10 PM

MACHINABILITY STUDIES ON ALUMINUM-CORUNDUM METAL-MATRIX COMPOSITES: K. V. Sharma¹; ¹UVCE, Department of Mechanical Engineering, K.R. Circle, Bangalore, Karnataka 560001 India

Aluminum-Corundum composites were prepared using liquid metallurgy technique. Apart from other tests, machinability test were conducted to ascertain condition under which these composites can be machined, specifically to check the effect of particles in chip length. Earlier reports showed chip length of composite samples decrease with dispersion of ceramic particles in aluminum matrix. Al-Corundum composites were subjected to machinability tests at different cutting conditions, such as cutting speed, cutting feed and depth of cut. Fx, Fy and Fz with chip thickness and length were measured under various cutting conditions. These values were used to calculate chip thickness ratio Yc and hence shear plane angle ϕ_1 . Tangible evidence were provided using SEM Techniques.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Processing

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Tuesday PM Room: London
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Brian Derby, University of Oxford, Department of Materials, Oxford, OX1 3PH UK

2:00 PM KEYNOTE

STRONG CERAMIC MATERIALS THROUGH NANO TO MOLECULAR LEVEL DESIGN: Koichi Niihara¹; Masafumi Kusunose¹; Tohru Sekino¹; Yong-Ho Choa¹; ¹Osaka University, The Research Institute for Scientific and Industrial Research (ISIR), 8-1 Mihogaoka, Ibaraki, Osaka 567 Japan

It is now well-known that the nanocomposite concept has a great possibility to essentially improve the mechanical properties of ceramic materials even at high temperatures. For example, the room temperature strength could be increased two to five times by only 5vol% nanodispersion, and the high temperature creep resistance was also improved two to four orders by this technology. In addition to the structural ceramic based composites, strong improvement in mechanical properties was also observed in the ceramic/metal systems. Recently, we applied the nanocomposite technology to the electronic ceramics such as BaTiO₃, ZnO and PZT, intermetallic compounds and also polymers, and found that this technology will work well even in these materials field. The purpose of nanocomposite technology extent is to find new materials design concept for developing the multi-functional ceramic materials, and many fruitful results are now coming in. In the BaTiO₃/Ni nanocomposite system, for example, two to three times improvement was achieved not only in mechanical properties such as strength and toughness but also in dielectric characteristics. For the Al₂O₃/Ni, MgO/BaTiO₃ and MgO/PZT nanocomposite systems, we succeeded to incorporate the fracture and/or stress sensing function originated from ferromagnetic of Ni and ferroelectric of BaTiO₃ and PZT with improved mechanical properties. Striking finding during these researches is that the functionally graded structure control of interfaces between the nano-sized particle and matrix drastically decreases the volume fraction of second phases at which the mechanical properties are strongly improved: 5vol% for usual nanocomposites to below 0.5vol% for this new materials with the functionally graded interface structure. In the presentation, new processing for the nanocomposites and the property improvements by this technology is first reviewed and then their property improvement mechanisms will be discussed. In these discussions, special emphasis will be placed on understanding of the roles of the interface structure. Finally, the new materials design concept of ceramic materials will be proposed. These new concept was induced from the molecular level design of the matrix grain and the functionally graded interface structure between the nano-sized particle and matrix grain.

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MICROWAVE SINTERING OF CaO DOPED ZrO₂ NANOCERAMICS: S. Bhaduri¹; E. Zhou¹; ¹University of Idaho, Metallurgical and Mining Eng., Moscow, ID 83844-3024 USA

Nanocrystalline CaO doped ZrO₂ powders were synthesized using the Auto Ignition technique. The objective of the work was to densify nanocrystalline ZrO₂ using an activated sintering process such as microwave sintering so that nanocrystalline grain size is maintained while achieving densification. Zr-nitrate and Ca-nitrates were used as oxidizers and urea was used as the fuel. The as-synthesized powders were nanocrystalline in size. Compacts were pressed and sintered at 1200YC for up to half an hour. It was seen that microwave coupling was better with more CaO content. Development of phases and microstructures will be discussed.

3:05 PM

FABRICATION OF Ni-Co ALLOY DISPERSED ALUMINA COMPOSITE AND ITS PROPERTIES: Sung-Tag Oh¹; Masanobu Awano²; Mutsuo Sando²; Koichi Niihara³; ¹Fine Ceramics Research Association, Synergy Ceramics Laboratory, 1-1 Hirate-Cho, Kita-Ku, Nagoya, Aichi 462-8510 Japan; ²National Industrial Research Institute of Nagoya, 1-1 Hirate-Cho, Nagoya, Aichi 462-8510 Japan; ³Osaka University, ISIR, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

In purpose of introducing ferromagnetism into structural ceramics, ceramic based composites dispersed with nano-sized phases were studied. In this approach, Al₂O₃, which has been well examined as a structural ceramics, and Ni-4.5 wt% Co alloy as a material having ferromagnetic property were selected for the ceramic matrix and dispersions, respectively. The composites were fabricated by the hydrogen reduction and the hot-pressing of Al₂O₃ and NiO-CoO powder mixtures, in which the Ni- and Co-nitrate was used as the source of NiO and CoO. Microstructural observation revealed that the nano-sized Ni-Co alloy particles were homogeneously dispersed in the Al₂O₃ matrix. The composite with 10 wt% alloy showed enhanced fracture strength as 1070 MPa. The composite exhibited the ferromagnetic property, and the magnetic response such as coercive force and magnetostriction showed the strong dependence on the dispersoid size.

3:30 PM

PROCESSING OF CERAMIC-MATRIX NANOCOMPOSITES BY HIGH PRESSURE SINTERING: Rajiv S. Mishra¹; Amiya K. Mukherjee¹; ¹University of California, Dept. of Chemical Eng. and Materials Science, One Shields Avenue, 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 ceramic-matrix nanocomposites will be highlighted. We have been able to synthesize fully dense alumina nanocomposites with grain size as fine as 30-40 nm. The results show significantly higher hardness and toughness in nanocomposites.

3:55 PM BREAK

4:05 PM KEYNOTE

AMORPHOUS AND CRYSTALLINE POLYMER-DERIVED CERAMICS: Fritz Aldinger¹; ¹MPI fuer Metallforschung, PML, Heisenbergstrasse 5, 70569 Stuttgart, Germany

The condensation of preceramic compounds is done by solid state pyrolysis, chemical vapour deposition or chemical liquid deposition. The general idea of such process routes is that the precursor molecules contain already structural units of the residual inorganics, thus providing novel paths of controlling the composition, atomic array and microstructure of materials. For the manufacture of bulk components the production of covalent bonded inorganics on the basis of Si, B, C and N by solid state pyrolysis is of special interest. Using proper precursor polymers either amorphous or crystalline materials can be

produced. Due to the lack of grain boundaries and a low atomic diffusivity in covalent materials their amorphous states provide thermal stability, oxidation resistance and very attractive mechanical properties at rather high temperatures. The crystalline materials also create substantial interest. Depending on the type of precursor, devitrification of the amorphous state into the nanocrystalline state occurs at very high temperatures providing the basis for thermally quite stable microstructures. Another favorable feature of the process is that for the shaping of products as parts, coatings, fibers or other components economic techniques of standard polymer engineering can be applied.

4:45 PM

MICROSTRUCTURAL STABILITY AND PROCESSING IN SILICON NITRIDE COMPOSITES: *K. Rajan*¹; Pavol Sajgalik²; ¹Materials Science and Engineering Department, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA; ²Institute of Inorganic Chemistry, Slovak Academy of Sciences, Bratislava Slovak Republic

The relationship between microstructural evolution in beta silicon nitride composites and processing parameters is explored in this presentation. Of particular interest is the coupling of microchemistry and interface structure on anisotropic grain growth. The implications on developing controlled microstructures during processing by controlling interfacial chemistry is emphasized.

5:10 PM

PREPARATION, MICROSTRUCTURE AND OXIDATIVE STABILITY OF SiC-C NANO-COMPOSITES BY A POLYMER PRECURSOR ROUTE: *K. V. Moraes*¹; *K. Rajan*¹; *L. V. Interrante*²; ¹Materials Science & Engineering Department, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA; ²Rensselaer Polytechnic Institute, Department of Chemistry, Troy, NY 12180-3590 USA

SiC-C nano-composites having different proportions of carbon and SiC were prepared by co-pyrolysis of a mixed precursor system. Allyl-hydridopolycarbosilane (AHPCS) was used as the precursor for SiC and a pitch as a precursor for carbon. Microstructural development in these materials as a function of annealing temperature up to 1600°C was studied by transmission electron microscopy. The oxidation rates in air, of these SiC-C nano-composites for various combinations of SiC and carbon will also be presented and correlated to the microstructure. This system is being studied as a potential source of oxidation-resistant fiber coatings for SiCf/SiCm composites.

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COMBINED MECHANICAL ALLOYING AND COMBUSTION SYNTHESIS IN THE TiH₂-B SYSTEM: *S. Ozbilen*¹; ¹Gazi University, Faculty of Technical Education, Metal Education Dept., Teknikokullar, Ankara Turkey

Combined mechanical alloying and combustion synthesis was carried out in the TiH₂-B system to investigate the influence of particle size of reactants in the exothermicity of the self propagating reactions together with the other effects of mechanical alloying on CS technique. Mechanically alloyed and compressed pellets of TiH-B mixtures were degassed and ignited in a reaction chamber. A combination of DTA, XRD and SEM study of samples before and after MA and CS was resulted in fruitful data collection for TiB-Ti ceramic matrix composites which will be presented and discussed in detail.