MONDAY AM

ALUMINUM REDUCTION TECHNOLOGY: Smelter Technology

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Georges J. Kipouros, Dal Tech, Dalhousie University, NS B3J2X4 Canada; Mark P. Taylor, Comalco Aluminium, Ltd., Brisbane, Queensland 4001 Australia

Monday AM Room: 6F
March 1, 1999 Location: Convention Center

Session Chair: Geoff P. Bearne, Comalco, Comalco Research and Technical Support, Melbourne, Victoria 3074 Australia

8:00 AM - SPECIAL LMD PRESENTATION

A REVIEW OF PAST ALUMINUM INERT ANODE RESEARCH LEADING TO A PLAN FOR FUTURE INVESTIGATION: *Dr. H. Wayne Hayden*[1]; Dr. Greg Barthold[2]; Rodney Hanneman[3]; [1]Oak Ridge National Labs, Bldg. 4500S, Rm C47, Oak Ridge, TN 37831; [2]ASME, 1828 L. Street NW, Suite 906, Washington, DC 20036; [3]3801 Old Gun Road West, Midlothian, VA 23113

ASME has been asked by the Department of Energy's Office of Industrial Technology to assess past work on the development of an inert anode for aluminum electrolytic reduction, and to suggest a path for future research. ASME's Center for Research and Technology Development is undertaking this task by forming a Technical Working Group of recognized independent experts in the field of aluminum reduction for the purpose of evaluating literature and patents and reviewing presentations by private investigators. Their work is in the beginning stages of review and assessment of both public and private information. A peer reviewed document is expected by early spring '99 for presentation to DOE. This presentation at TMS will describe progress to date. The final report, when finished, will be made available to the public.

8:30 AM

AP21: A HIGH PERFORMANCE, HIGH PRODUCTIVITY AND LOW CAPITAL COST NEW CELL TECHNOLOGY: *P. Homsi*[1]; *J. Bos*[1]; P. Herd[1]; [1]Aluminium Pechiney, LRF - BP114, Saint-Jean-de-Maurienne, Cedex 73303 France

Based on the broad experience gained from the current generations of AP cell technology, the well known AP18 cell has been re-engineered to create a new advanced cell operating at about 210 kA and called AP21. A range of numerical models, fitted to experimental results has been used. The new design benefits from the latest improvements in lining materials and design, anode and pin dimensions and sidewall ventilation. Four test cells have been operating since September 1997 at the Saint-Jean-de-Maurienne smelter. Results are well in line with calculation predictions. Technical performances are presented and discussed. The main features of the new cell are a reduction of almost 15% in capital cost per ton of installed capacity and improvements in operating costs. The new cell technology is available for greenfield projects as well as for retrofitting of current AP18 potlines.

9:10 AM

PRODUCTIVITY INCREASE AT SORAL SMELTER: *T. Johansen*[1]; H. P. Lange[1]; R. von Kaenel[2]; [1]Sor-Norge Aluminium A/S, Elektrolysen, P.O. Box 85, Husnes N-5460 Norway; [2]Alusuisse Technology & Management Ltd., Technology Center Chippis, CH-3965, Chippis, Switzerland

The Sør-Norge Aluminium AIS(Søral) smelter was started in 1985 with Alusuisse 100 kA pre-baked anodes technology. In 1989 it was decided to retrofit the plant as the operation had not changed since start-up. Goals for the modernization were initially rather modest. The current intensity should be increased from 115kA, the current efficiency from 90 to 92% and energy consumption should be decreased from 15.3 to 14.7 kWh/kg Al. These goals should be reached by introducing a new concept for cathode shell and lining, a minimum magnetic compensation, and new process control and point feeding. These changes explored new potentials. Today Søral is operating both pot lines at 126.5 kA with a current efficiency higher than 93.5%. Ten test cells have been operating for more than 17 months at 140 kA and in cooperation with the Alusuisse Technology Centre Chippis, Søral is exploring the possibilities to operate the cells at 150 kA. This paper presents the developments and methodology in details, shows the results and possible future developments.

9:35 AM

NORDULAR FAST-TRACK ALUMINIUM MODULAR SMELTER NORDURAL IN ICELAND: *J. Lombard*[1]; [1]VAW Aluminium-Technologie GmbH, P.O. Box 101554, 41415 Neuss Germany

Significant savings in capital expenditures for greenfield smelter can be achieved by reducing the time needed from ground breaking to the first hot metal. The construction of the Nordural Smelter in Iceland is an example of such a fast-track smelter project with an outstanding construction period of only 14 months. The process technology for this smelter project was delivered by VAW Aluminium-Technologie GmbH, Germany. Nordural hf, a subsidiary of Columbia Ventures Corporation (CVC), has been designed and approved for a name plate capacity of 180,000 t/a in three phases. Phase 1 of this state-of-the-art smelter consists of 120 VAW CA180 pots, a side-by-side prebake reduction cell operating at 180,000 amps. The pots are equipped with center hoppers for alumina and aluminum fluoride with crust breakers and point feeders incorporated in the hoppers. The cells are controlled by the VAW ELAS control system. Additionally there is an anode rodding shop, casthouse, fume treatment plant, alumina handling equipment, including harbour off-loading equipment.

10:00 AM

BUSBARS OPTIMIZATION USING CELL STABILITY CRITERIA AND ITS IMPACT ON CELL PERFORMANCE: *J. P. Antille*[1]; R. von Kaenel[1]; [1]Alusuisse Technology & Management, Ltd., Technology Center Chippis, Chippis CH - 3965 Switzerland

It is desirable to operate an aluminum reduction cell at the highest possible level of current: the higher the current, the more aluminum per unit time. Various factors limit the maximum current, among them the need to maintain an acceptable heat balance, which requires reducing the anode-cathode distance as the current is increased. This tends to make the cell less stable: it becomes noisy, leading to reduced current efficiency and increased energy consumption. The busbar configuration has a major effect on cell stability. A powerful mathematical model for predicting cell stability has been developed, and is in use for optimizing

existing busbar systems. Applications at industrial scale are presented and the resulting performance improvements discussed.

10:25 AM BREAK

10:45 AM
MODERNIZATION OF S 172 - M2 PREBAKED POTS: *V. V. Geinze*[1]; V. V. Bersteniov[1]; L. K. Krylov[1]; V. J. Orlov[1]; F. Zannini[2]; H. O. Bohner[2]; J. Ifju[2]; S. Deb[3]; [1]OAO SaAZ, Sajanogorsk, Chakassia 662703; [2]Techmo Car S.P.A., Via R. Colpi, Limena 15-17 - 3501 Italy; [3]Hart Ltd - 13A New Road, 700027 Calcutta, India

AO Saianskij Aluminievyi Zavod (SaAZ) and Techmo Car/Techmo Engineering SpA (Techmo) have decided to implement a modernization project aimed at improving performance and ecology of the potline No 2 comprising S-172 M2 type cells arranged in two rows in each potroom. Application of some elements of an up-to-date technology like magnetic compensation, point feeding, process control and technology based on high acidic bath on 10 end-to-end 172 kA prebaked cells without shutting them down provides an example on the ways of possible retrofitting approaches. This paper describes the work done in the course of 30-month preparation and pretest phase, as well as the results obtained and approved during the 1-month control test period.

11:10 AM
DESIGN OF FEEDING BUSBAR SYSTEM: *F. M. El-Dawi*[1]; S. A. Mohamed[1]; U. H. Seha[1]; A. I. Hassanien[1]; [1]The Aluminium Co. of Egypt, Research and Design, Nage-Hammadi Egypt

The annual production of the Aluminium Company of Egypt is increased by adding a new pot line with prebaked anode 200 kA End to End cells. Electrical, thermal and economic calculations are accomplished. Different design alternatives are calculated and compared. The best design was the one which has lower cost and maintains certain requirements as safety. A retrofitting process requires modification of existing busbar system. This paper explains the calculation and design of the two busbar systems by computers.

11:35 AM
STARVING OF SODERBERG CELLS FROM ANODE PASTE BEFORE REPLACEMENT TO PREBAKED ANODE CELLS: *M. M. Ali*[1]; Z.M. Ramadan[1]; F. M. El-Dawi[1]; [1]The Aluminium Company of Egypt (Egyptalum), Nage-Hammadi Egypt

In the last two decades Egyptalum has made an extensive program towards converting its technology from Soderberg to Prebaked anode cells. During the period of conversion, the feeding of anode paste to the anode casing was stopped for a number of days according to a special program for stud replacement. The performance of selected cells in potline number 5 has been studied before the shutdown proceeding the cell development. The maximum non-charging days reached to 37 days without any adverse affect on cell performance. The energy consumption has been reduced and the economic benefit of this study has been calculated for one potline. Also the chemical and physical analyses of the anode layers have been compared with the raw materials specification using an in-production process for economic reuse.

AUTOMOTIVE ALLOYS III: Session I - Overview

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh Das, ARCO Aluminum Company, PO Box 32860, Louisville, KY 40232

Monday AM Room: 3
March 1, 1999 Location: Convention Center

Session Chair: Subodh K Das, ARCO Aluminum Inc., Louisville, KY 40232 USA

8:30 AM INVITED PAPER
IS THE ALUMINUM CAR A FANTASY?: *Firoze Katrak*[1]; [1]Charles River Associates, Inc., 200 Clarendon St., T-33, Boston, MA 02116 USA

Over the last two decades, aluminum usage in cars has grown significantly, but mostly in cast forms. The bulk of these applications is in transmission, engine block, and suspension type components. Use of aluminum in body-in-white (BIW) has been limited and sporadic, and steel continues to dominate in BIW applications. The use of polymers has grown only in the interior, with some small successes in exterior applications. Will usage of aluminum and polymers plateau, or will it break through and grow rapidly in the next century? How will the battle among steel companies, polymer companies, and aluminum companies affect this outcome? The paper will discuss key issues related to the future of aluminum, steel, and polymers in the automotive industry.

9:00 AM INVITED PAPER
OVERVIEW OF DOE'S PROGRAMS ON ALUMINUM AND MAGNESIUM FOR AUTOMOTIVE APPLICATIONS: *Joe Carpenter*[1]; [1]U.S. Department of Energy, EE-32, 1000 Independence Ave., S.W., Washington, D.C. 20585 USA

This will be an update and review of the DOE programs on aluminum and magnesium for automotive (including heavy duty) applications. While the main programs focused on automotive applications are in the Office of Transportation Technologies (OTT), there are contributory efforts in the DOE Office of Industrial Technologies and the Office of Energy Research. The OTT efforts are programmatically divided into Automotive Materials and Heavy Vehicle Materials, but both have efforts on materials for the body/chassis and the powertrain, and there is considerable synergism among the efforts. The bulk of the efforts are on castings, sheet and alloys with lesser efforts on metal matrix composites. The overriding theme of all the efforts is cost reductions.

9:30 AM INVITED PAPER
ALCAR — A MODEL FOR HORIZONTAL R&D CONSORTIUM: *Wayne Hayden*[1]; [1]Oak Ridge National Laboratory, Metals & Ceramic Division, P.O. Box 2008, Oak Ridge, TN 37831-6152 USA

ALCAR consortium was created to develop a low cost, non-heat treatable automotive body sheet aluminum alloy. This paper will discuss the management aspects of organizing and running a horizontal consortia for competing companies to cooperate in conducting pre-competitive research and development involving US Department of Energy, National Laboratories, Universities and Industrial consultants.

10:00 AM BREAK

10:30 AM INVITED PAPER
INNOVATIVE ALUMINUM APPLICATIONS FOR AUTOMOTIVE USE IN EUROPE: *Dietrich G. Altenpohl*[1]; [1]Rainstrasse 368, Feldmeilen CH-8706 Switzerland

Based on selected examples, a review is presented on recent and ongoing innovations within some aluminum and Automotive Industries: By the development of components and subsystems from Aluminum Alloys and thereby substituting iron and steel in achieving less weight but also better properties mainly regarding LCA.

11:00 AM
EFFECT OF THERMOMECHANICAL PROCESSING AND SURFACE TREATMENT ON FATIGUE PERFORMANCE OF HIGH-STRENGTH MAGNESIUM ALLOYS: *M. Hilpert*[1]; [1]Technical University of Brandenburg at Cottbus, Chair of Materials Technology, P.O. Box 101344, Cottburg 03013 Germany

The high-strength magnesium alloy AZ 80 was received as extruded bar stock. Material to be deformed by hot rolling, 1-dimensional and 3-dimensional pressing or swagging was first solution heat treated at T = 405ŸC for 1 hour. The forming temperature was constant at T = 400ŸC. For the various forming procedures, the total deformation degree was roughly constant at f = 1. After deformation, the crystallographic texture was determined. Tensile tests were performed using initial strain rates of e = 8.4 - 10-3 s-1. Fatigue tests were conducted on electrolytically polished hour-glass shaped specimens in rotating beam loading (R

= -1) in air. In addition, mechanical surface treatments were used to improve the fatigue performance of the various conditions. Examples of fatigue life improvements will be shown for shot peening and roller-burnishing and will be interpreted in terms of the resistances to fatigue crack nucleation and microcrack growth as affected by surface roughness, work hardening and residual compressive stresses.

11:20 AM

CONDUCTIVE HEAT RESISTANCE SEAM WELDING OF ALUMINUM: *Lawrence Robert Lehman*[1]; [1]Edison Welding Institute, Resistance & Solid State Welding, 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

As consumer demand for more fuel efficient vehicles increases, as well as increased government regulation of fleet fuel economy, the use of aluminum for automotive components is becoming more attractive. As part of this effort, aluminum tailored blanks containing continuous joints have become of interest. To date, the processes used to fabricate these joints have been expensive. These processes have included laser welding, GTA welding, and electron beam welding. Recently, a new process has been developed for continuously joining aluminum alloy sheet in a butt joint configuration. The process utilizes resistance heating of steel cover sheets, with subsequent conductive heating of aluminum. Preliminary weld trials have demonstrated the process is capable of joining similar and dis-similar automotive gauge aluminum alloys at process speeds up to 150-in./min.

11:40 AM

PRODUCING STEERING WHEEL FRAMES BY USING AN AlMgSiMn- TYPE ALLOY: *Moritz C. Wuth*[1]; [1]PETRI, Grossostheimer STR 223, Aschaffenburg D-63741 Germany

In 1997 world wide 57,000,000 vehicles were built (1). This gives an idea of the size of the current steering wheel market. There are several different steering wheel frame designs and material combinations in use. The most common designs are the completely diecasted designs from aluminum-and magnesium alloys, hybrid designs from steel and aluminum and the welded designs made from steel. Hybrid designs typically consist of spokes made from bended wire, and rims made from bended pipes. Those parts are combined to a frame as inlays in high pressure die casting as well. The main target of all producers is, of course, to improve the properties and reduce the cost of a steering wheel frame. This paper describes the development of an all-aluminum steering wheel frame made of the alloy Magsimal-593 which provides excellent mechanical and dynamical properties without any heat treatment. A brief description of the requirements for steering wheel frames is outlined, the melting and casting technique is described and experiences of two years in production are discussed.

CARBON TECHNOLOGY: Raw Material Production And Qualifying

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: C. Dreyer, Aluminium Pechiney, St. Jean De Maurienne 73303 France

Monday AM — Room: 6D
March 1, 1999 — Location: Convention Center

Session Chair: Bill Spencer, Great Lakes Carbon, Kremlin, OK 73753

8:30 AM SESSION CHAIRMAN INTRODUCTION

8:35 AM

PLANT EVALUATION OF COAL TAR/PETROLEUM PITCH ANODE BINDER: *John Thomas Baron*[1]; Joe Jacobs[2]; [1]Koppers Industries Inc., 1005 William Pitt Way, Pittsburgh, PA 15238 USA; [2]ALUMAX of South Carolina, P.O. Box 1000, Goose Creek, SC 29445 USA

Recent closings of several U.S. coke plants have supported the predictions of a growing shortage of coal tar for the production of coal tar anode binder pitch. Koppers Industries Inc. began development of petroleum enhanced coal tar pitches in the early 1990's. Through the years of development, significant improvements have been made in the quality and performance of the coal tar/petroleum pitch. This paper will present the results of full plant trial that demonstrated that the performance of the coal tar/petroleum pitch was equivalent to the coal tar pitch. No changes to the anode forming process were required.

9:00 AM

INFLUENCES ON ANODE GRADE PETROLEUM COKE QUALITY: *Eberhard Lucke*[1]; *Guenter Breuer*[1]; [1]Veba Oel AG/Ruhr Oel GmbH, Coker/Calciner, Johannastrasse 2-8, Gelsenkirchen, NRW 45899 Germany

In the RUHR OEL refinery in Horst, Germany, heavy residues are upgraded to valuable liquid products by thermal cracking in a delayed coker. The "bottom product" of the coking process is petroleum coke, which is calcined and sold to the anode manufacturing industry. This paper gives the basic information on the refinery structure and the integration of the delayed coker and calciner plant. It shows the influence of crude oil properties, coker feedstock properties, coker feed mix and process parameters (coker and calciner) on coke quality. Options of process optimisation and quality control are described. The purpose of this paper is to give a deeper understanding of the coke making process and to show potential benefits in improving the contact between coke producers and anode manufacturers.

9:25 AM

REAL DENSITY MEASUREMENT OF PETROLEUM COKE: *R. E. Gehlbach*[1]; G. S. Tittle[1]; V. A. Benoit[2]; [1]Reynolds Metals Company, Smelter Technology Laboratory, 4276 Second St., Muscle Shoals, AL 35661-1258 USA; [2]Baton Rouge Calcined Coke Plant, P.O. Box 4448, Baton Rouge, LA 70821-4448 USA

Real density (RD) of petroleum coke is frequently a quality specification, and generally considered an indication of the temperature to which a coke is calcined. Its proper measurement is sensitive to sample preparation techniques, requiring the measurement of coke volume with all porosity excluded. Helium pycnometer determinations were performed to evaluate the effects of various parameters on measured real density of several cokes. Degrees of fineness of samples meeting the -200 mesh (< 75 micron) particle size criteria of ASTM and ISO standard test procedures were evaluated using conventional and air-jet sieving and Blaine analysis. The fine microporosity (< 0.1 micron) generated by well-known high temperature thermal desulfurization is frequently not excluded from the measured sample volume. Other measurement errors may result from the effects of dedusting oils. Advantages of a vacuum treatment prior to measurement of RD are discussed.

9:50 AM

PLANT EXPERIENCE IN QUALIFYING RAW MATERIALS FOR THE CARBON PLANT: *Marilou McClung*[1]; Gerald F. Chovanec[1]; J. Anthony Ross[1]; [1]Century Aluminum, Ravenswood Primary Products, P.O. Box 98, Ravenswood, WV 26164 USA

With new environmental regulations, changing product demands and changing business climates, the sources of feed stocks to the coke calcining and pitch processing facilities are frequently changing in quality and quantity. The challenge for Carbon Plants is to produce a consistently high quality product while facing changes in the incoming raw material streams. The influence of changes in the raw materials may not occur immediately but will be seen, as butt return is recycled from the Potrooms. Changes that may occur could be seen in net carbon, return butt weight, pitch demand, and aggregate sizing among other factors. This paper outlines Century's experience in the effect of recycle butt materials on carbon performance and the number of anode cycles it may take to totally add or remove the influence of a raw material from the carbon product. This paper also includes details on the procedures used to qualify new or changed raw materials in the Ravenswood Operation's Reduction Plant. These procedures take into account the effect of recycle butt material in plant and laboratory scale testing. Practical examples are given of recent plant and laboratory scale testing of a petroleum pitch blend versus a coal tar pitch used in anode production.

10:15 AM BREAK

10:35 AM

WAYS TO IMPROVE ANODE PERFORMANCE IN VS LINES: *Alexandre Gomes*[1]; [1]Aratu, Alcan Aluminio Do Brazil, Caixa Postal 7391, Pituba, Sslvador 41-810-000 Brasil

If we take into account the importance of the VS anode performance on the overall cell results, little effort is generally directed into trying to understand the interactions among the various anode operational parameters, including raw materials quality, and their exact influence on cell performance. At Alcan's Aratu plant we were no exception to that and, over the years, changes in raw materials quality as well as operational moves carried out to improve productivity and costs resulted in serious deterioration of cell performance. These problems made us look more deeply into the VS anode operation as a whole, which gave us a reasonably good understanding of the aforementioned interactions. In this paper we propose mechanisms that explain some of the various types of anode problems and, as a consequence, where should the main anode operational parameters and raw materials quality be to assure a good overall cell performance.

11:00 AM

ANALYTICAL METHOD FOR DETERMINING PITCH AND GRANULOMETRY CONTENT OF ANODE PASTES: *Stanley E. Christopherson*[1]; Mike Toda[1]; Bruce Lorenz[2]; [1]Goldendale Aluminum Co., Lab, 85 John Day Dam Rd., Goldendale, WA 98620 USA; [2]Bruce Lorenz Consulting, 922 W. 11th, The Dalles, OR 97058 USA

The monitoring of anode paste plant efficiency in regards to coke granulometry and pitch content is of primary importance. Analyzing the final carbon product, and comparing the data with the plant instrumental settings, will give a good indication of what is occurring during the mixing process and therefore a good indicator of plant efficiency. This method was developed to give a quantitative and reproducible value of pitch content in the finished product coming out of the anode paste plant. In addition to the determination of pitch content, the coke granulometry percentages and the degree of coke grind-down in processing can be measured as well. As a result of this information, a determination of scale accuracy and granulometry adjustments can also be inferred.

CAST SHOP TECHNOLOGY: Molten Metal Processing/Degassing

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Y. Sahai, The Ohio State University, Dept. of Mats. Sci. and Eng., Columbus, OH 43210-1179 USA; James O'Donnell, Commonwealth Aluminum, Dept. of Eng., Louisville, KY 40202 USA

Monday AM Room: 6C
March 1, 1999 Location: Convention Center

Session Chair: Dr. Ed Eckert, Apogee Technology, Inc., Verona, PA 15147 USA

8:30 AM

GAS FLUXING OF MOLTEN ALUMINUM: *Geoffrey K. Sigworth*[1]; [1]GKS Engineering Services, 116 Derby St., Johnstown, PA 15905 USA

The aluminum industry is under continual pressure to improve metal quality, while at the same time reduce costs. The only way to do this is through continual process optimization. Although the gas fluxing of aluminum is a reasonably mature technology, there is still room for improvement. A detailed review and theoretical analysis is given for chemical and kinetic factors which control the metal quality after gas fluxing, and suggestions are made for ways to improve the process. Particular emphasis is placed on hydrogen removal and minimization of chlorine use. Considerations related to inclusion removal are also discussed briefly.

8:55 AM

ALPUR TS, THE SOLUTION FOR LOW COST DEGASSING AND FILTRATION: *Joop De Ridder*[1]; Jean-Claude Terrier[2]; [1]Pechiney Aluminium Engineering, Postbus 49, Vlissingen 4380 AA The Netherlands; [2]Pechiney Aluminium Engineering, Centr'Alp, B.P. 24, Voreppe 38340 France

Pechiney Nederland NV, one of the largest European manufacturers of extrusion billet supplies the European market with a broad range of alloys and billet diameters. A large program was engaged to modernize their casting and homogenising facilities while keeping the same level of flexibility. In order to minimize their operating costs while maintaining high degassing and filtration efficiencies, Pechiney Nederland elected the new Alpur TS as their preferred technology. This paper describes the main items of equipment with regard to industrial needs, including special adaptations to meet the need for high flexibility associated with high standards of operating conditions.

9:20 AM

UPGRADING A TWO STAGE METAL TREATMENT SYSTEM TO A DUAL PROCESS MOLTEN TREATMENT SYSTEM AT COMMONWEALTH ALUMINUM UTILIZING APOGEE TECHNOLOGY'S REVROT SYSTEM: C. William Sanderson[1]; Joe Tessandori[1]; George Ducsay[2]; [1]Commonwealth Aluminum, Lewisport, KY 42351-0480; [2]Apogee Technology, Inc., Verona, PA 15147

The customer-driven qualtiy and productivity mandates of today's aluminum industry requires that the successful aluminum producer maintain a program of absolute vigilance to emerging, and potentially enabling, leap frog technology. Commonwealth Aluminum is committed to such a program, and recently evaluated various commercial processes to replace a conventional unidirectional two stage in-line metal treatment system for can sheet products at the Lewisport Operations. The objectives were demonstrable improvements in a) separation of suspended solids (inclusions) via flotation, b) hydrogen removal, and c) operator friendliness and safety. Apogee Technology's REVROT process was purchased for plant trials. The system uses a unique bi-directional phase contactor to achieve very high input power density levels. Increases in process gas bubble dispersion and vortex free liquid phase mixing can therefore result in dramatically improved performance. Following the implementation of Apogee prescribed modifications to internal baffling, a two stage, retro-fit REVROT drive system, utilizing different impellers, was operated and compared to baseline performance from the conventional system. The operating experience is summarized in this paper and comparison of critical process parameters are presented.

9:45 AM

HYDROGEN REMOVAL FROM ALUMINIUM IN CO-CURRENT AND COUNTER-CURRENT BUBBLE COLUMNS: *Martin Syvertsen*[1]; Frede Frisvold[2]; Thorvald Abel Engh[1]; Didrik S. Voss[3]; [1]Norwegian University of Science and Technology, Dept. of Metall., Alfred Getz vei 2b, Trondheim N-7034 Norway; [2]SINTEF, Mats. Tech., Alfred Getz vei 2b, Trondheim N-7034 Norway; [3]Elkem Aluminium ANS, Lista, Farsund N-4550 Norway

Bubble columns may be an interesting alternative to rotor units for removal of hydrogen from molten aluminium. This has been studied in water models removing O_2 with N_2 as purge gas. The experimental results agree well with theory, both for co-current and counter-current flow. The concept has partly been verified in industrial experiments. A gas lift pump was used to adjust the metal level in a new deep bed filter developed and tested in production runs at Elkem Aluminium ANS, Lista, Norway. The gas-lift pump also acts as a co-current bubble column degasser. Hydrogen content in the melt was measured in and out. The results are discussed based on the theory of removal in co-current bubble columns. Removal compares favourably to a rotor unit (back-mix tank) even though the pump was not primarily designed for this purpose.

10:20 AM

OPERATING EXPERIENCE WITH THE CAST HOUSE TROUGH REACTOR: *Chris English*[1]; Bruce Walker[2]; [1]Cast House Technology, Ltd., 18 Mary St., Guelph, Ontario NIG 2A7 Canada; [2]Bon L. Canada, Inc., 1850 Clements Rd., Pickering, Ontario Canada

This paper examines the performance of the Cast House Trough Reactor, which degasses with closely spaced high velocity argon jets producing small bubbles at very high bubble concentration densities. Actual degassing and particulate removal figures achieved with increased metal flow rates are compared with previously reported numbers extrapolated from lower levels of throughput. The validity of assumptions used in these extrapolations is explored and modified in the light of actual performance. Performance figures for a wide range of alloys and metal and gas flow rates are discussed. The techniques used to eliminate dross formation in the reactor and potential nozzle blockage are also demonstrated. A real-time video lasting approximately six minutes, covering start-up, shut-down, reactor draining and cleaning to make ready for the next cast, will be shown.

10:45 AM
BUBBLE SIZE AND REMOVAL RATE OF SODIUM IN IMPELLER STIRRED REFINING REACTORS: Stein Tore Johansen[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2B, Trondheim, Trøndelag 7034 Norway

At previous TMS annual meetings we have discussed the relations between bubble sizes, mass transfer and stirring power in melt processing. We also have presented experimental data for melt surface mass transfer obtained in water models and how water model data may be adopted in predicting real melt refining. In the present paper we attempt to predict sodium and hydrogen removal data obtained in the casthouse under standard operating conditions. The refining models are extended to sodium removal using reactive and non-reactive purge gases. Without resorting to constant fitting in the model we predict removal rates and compare to the casthouse experiments. The good predictions of casthouse data support the physical understanding of mass transfer and bubble size and support water models as efficient tools to understand melt refining.

CREEP BEHAVIOR OF ADVANCED MATERIALS FOR THE 21ST CENTURY: Microstructure and Mechanisms I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee, Structural Materials Division, Mechanical Metallurgy Committee, Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: Rajiv S. Mishra, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA 95616 USA; Amiya K. Mukherjee, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA 95616 USA; K. Linga Murty, North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA

Monday AM Room: 15A
March 1, 1999 Location: Convention Center

Session Chair: A.K. Mukherjee, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA 95616 USA; E.M. Taleff, University of Texas, Aerospace Engineering and Engineering Mechanics, ASE/EM, C0600, Austin, TX 78712-1085 USA

8:30 AM OPENING REMARKS

8:35 AM KEYNOTE
HIGH TEMPERATURE CREEP BEHAVIOR OF ULTRA-FINE GRAIN MATERIALS: *J. R. Weertman*[1]; [1]Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Many dispersion-strengthened ultra-fine grain (UFG) materials retain good strength levels at high temperatures. Their microstructures can be very stable, resisting grain growth or recrystallization almost up to T_m. UFG metals and alloys tend to have very low work hardening rates or even slow work softening, whether dispersion-strengthened or single phase, and thus are susceptible to inhomogeneous deformation at low strains. The dispersion-strengthened materials often appear to exhibit a threshold stress for creep. The cause(s) or even existence of a true threshold stress is not agreed upon. Above the threshold stress, creep is usually modeled as power law behavior. Various stress exponents are observed. Diffusional creep may be evident at lower stresses but frequently is much slower than predicted by classic diffusional creep equations. *Research supported by US DOE Grant DE-FG02-86ER45229

9:05 AM
VALIDATION OF A TRANSITION IN CREEP MECHANISM PREDICTED BY A NEW THEORY OF CREEP IN PRECIPITATION-STRENGTHENED ALLOYS: *Brian Dyson*[1]; [1]Imperial College of Science, Technology and Medicine, Department of Materials, Prince Consort Road, London SW7 2BP England

A large creep database on 2˜Cr1Mo steel has been taken from the literature and used to test one of the key predictions of a new theory of creep resistance in precipitation-strengthened alloys. The new theory proposes that the generally-accepted climb/glide mechanism of creep in these alloys operates macroscopically as a parallel kinetic process and leads to creep being controlled by the slower of two sequential processes: "recovery" of glide-dislocations from the precipitate dispersion; or their subsequent matrix glide-rate. The activation area for recovery-creep is shown to be proportional to the particle spacing and is smaller than that for viscous glide and thus glide-control is favoured by a combination of low stress and low particle volume fraction. Four sets of data from different nickel-base superalloys (20-60% volume fraction), including a single crystal alloy, demonstrate that creep is consistent with recovery-control at all practical stresses and temperatures. In contrast, minimum creep rate data at several temperature levels for the 2˜Cr1Mo steel (approximately 1% volume fraction of particles) have been modelled as two sequential mechanisms with glide-control at low stresses and recovery-control at high. The steel data exhibit certain characteristics of conventionally defined threshold behaviour and the new theory therefore offers an alternative (non-threshold) explanation.

9:25 AM
A CONTINUUM DAMAGE MECHANICS APPROACH TO MODELLING ANISOTROPIC CREEP IN SINGLE CRYSTAL SUPERALLOYS: *M. McLean*[1]; M. Ardakani[1]; H. Basoalto[1]; R. N. Ghosh[1]; B. A. Shollock[1]; [1]Imperial College of Science, Dept. of Mats., Technology and Medicine, Prince Consort Rd., London SW7 2BP UK

The anisotropy of creep deformation of single crystal superalloys is a complex function of crystallography, defect content, stress and temperature and it is influenced by the evolution of the precipitate microstructure. This paper will describe the development of a mechanism-based model of anisotropic creep that uses the formalism of continuum damage mechanics to account for restriction of viscous glide to specified slip systems, progressive softening due to the accumulation of mobile dislocations, the effects of pre-existing casting porosity and the development of γ' rafting. The model is shown to successfully represent extensive databases of <001> and <111> creep curves for SRR99 and CMSX4 from which the model parameters are derived. The model also allows the prediction of changes in crystal orientation and specimen cross-sectional shape with increasing creep strain. These measures provide a sensitive approach to validating the model by comparison of model calculations with experimental measurements. Using the model parameters deduced from the database a range of predictions have been made and compared with measurements; these include tensile creep for complex orientations, strain controlled deformation (both in monotonic stress-strain curves and in low cycle fatigue) and implementation of the model in finite element codes to simulate multiaxial creep behaviour in notched and hollow specimens.

9:45 AM
EFFECT OF NETWORK FORMATION ON CREEP OF PLATELET- AND WHISKER-REINFORCED CERAMICS: *David S. Wilkinson*[1]; Rosaura Ham-Su[1]; [1]McMaster University, Materials Science. & Engineering, 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

When platelets or whiskers are added to a ceramic matrix a creep-resistant network is formed. The nature of this network depends on the volume fraction and the degree of orientation. In this presentation experimental work on alumina reinforced with SiC platelets will be presented. It will be shown that the creep resistance depends on the processing route. In particular, if platelets are aligned in one direction little network interaction develops and the resulting creep rate is high. A more random network develops greater coordination and thus a higher creep resistance. The development and evolution of network stresses during creep has been followed using neutron diffraction. The results of this study have been rationalized using a model based on the viscoelastic bending of platelets. The model is equally applicable to whiskers.

10:05 AM
CREEP DESIGN ANALYSIS OF SILICON NITRIDE USING STRESS RELAXATION TESTING: *David A. Woodford*[1]; [1]Materials Performance Analysis, Inc., 1707 Garden St., Santa Barbara, CA 93101 USA

A new approach to tensile creep analysis for ceramics based on stress relaxation testing is described for sintered silicon nitride. The results are presented in terms of creep rate vs. stress covering up to five orders of magnitude in tests lasting less than one day. Tests from various initial stresses at temperatures between 1200ϒC and 1350ϒC are analyzed and compared with creep rates measured during conventional constant load testing. It is shown that a significant portion of the accumulated creep strain is anelastic and recoverable. Excellent repeatability is demonstrated, and the effects of deformation history and prior thermal exposure are described. Pseudo stress vs. time curves generated from the data show a strong rate sensitivity. The systematic rate dependance provides a new basis for design against creep in ceramics in terms of a time-dependent secant modulus analysis.

10:25 AM BREAK

10:30 AM INVITED PAPER
MODELING TIME DEPENDENT PLASTICITY WITH LOCAL INTERACTIONS: *Glenn S. Daehn*[1]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1178 USA

Models that are similar to cellular automata developed to study toppling in sand piles are applied to the study of time dependent plastic deformation. The models are based upon a large number of sites, each of which may 'slip' in a given time step based on a probability determined solely by the 'strength' of the site and the local stress. If a given site slips, the stress on it is reduced while the stress on neighboring sites is increased such that the stress averaged over the material remains constant. Such locally interacting models of deformation provide many features that are observed in experimentally in plasticity but which are difficult to obtain in closed-form models. In particular: (a) These models show a size-dependence wherein the 'strength' of a solid decreases with increasing the number of interacting elements that comprise it. (b) Changes in stress produce a rather complex transient behavior wherein flow may either be forward, backward or zero upon a stress reduction. This is very similar to experimentally-observed behavior. (c) If a distribution of site strengths (either bimodal or continuous) is considered, an extended transient in the strain-time behavior is obtained in constant stress creep. This transient is typically close to a power-law form where strain accumulates with time raised to a power which is usually less than one-third. Such forms have been observed many times in creep experiments and are usually regarded as being empirical. The development of, and assumptions in, such models will be presented and it will be argued that simple models based on local interactions can offer much insight into the phenomenology of plastic deformation.

10:55 AM INVITED PAPER
CORRELATION BETWEEN CREEP RATE AND STACKING FAULT ENERGY DURING DISLOCATION CREEP: *Eric M. Taleff*[1]; Robert W. Hayes[2]; [1]University of Texas, Aerospace Engineering and Engineering Mechanics, ASE/EM, C0600, Austin, TX 78712-1085 USA; [2]Metals Technology Inc., 19801 Nordhoff St., Northridge, CA 91324 USA

Work by several investigators has shown that the creep rate during dislocation creep is strongly affected by stacking fault energy. These classical results can now be applied to several materials using stacking fault energy data not available until recently. Making use of stacking fault energy it is possible to normalize creep rate in a manner allowing the comparison of data from many different materials. When creep rate is normalized by both stacking fault energy and temperature and is plotted against modulus-compensated stress, the data of several different materials fall onto one master curve. This master curve offers a useful means of understanding and predicting dislocation creep behavior.

11:15 AM
CREEP BEHAVIOR OF BULK AMORPHOUS AND NANOCRYSTALLINE Zr-BASE ALLOYS: *A. Leonhard*[1]; M. Heilmaier[1]; J. Eckert[1]; L. Schultz[1]; [1]Institute of Solid State and Materials Research Dresden, Dresden D-01069 Germany

Bulk amorphous Zr-base alloys are known to reveal outstanding mechanical properties at room temperature, namely a beneficial combination of very high strength, relatively low "Young" modulus, some microplasticity and high wear resistance. However, their creep properties at elevated temperatures have not been investigated so far Zr-Al-Cu-Ni alloys with a significant supercooled liquid region ($\Delta T_x = T_x - T_g$ = 90 K, T_x: onset of crystallization, T_g: glass transition temperature) were produced by die casting into a copper mould. Annealing of the amorphous samples at 693 K leads to formation of a two-phase material consisting of nanocrystalline precipitates embedded in an amorphous matrix. The microstructure was analysed by x-ray diffraction (XRD) and transmission electron microscopy (TEM) with special emphasis on the size and composition of these crystallites. Creep experiments have been carried out by constant extension rate tests in compression with cylindrical specimens of typically 3 mm diameter and 6 mm height around T_g. In contrast to dispersion strengthened materials low concentrations of nanocrystals do not significantly influence the viscous flow type creep. However, the observed thermal stability against crystallization provides a promising possibility for easy shaping of complex parts of bulk amorphous metals at temperatures well above T_g.

11:35 AM
DEFORMATION BEHAVIOR OF Sn, SnSb and SnAg ELECTRONIC SOLDERS FROM CREEP AND AUTOMATED BALL INDENTATION TESTS: M. D. Mathew[1]; Hong Yong[1]; Sashidhar Movva[1]; *K. L. Murty*[1]; [1]North Carolina State University, Nuclear Engineering, Hillsborough St., Raleigh, NC 27695-7909 USA

The deformation behaviors of tin, Sn5Sb solid solution and Sn3.5Ag eutectic alloys were investigated using creep, and automated ball indentation (ABI) tests. The temperature range spanned from ambient to 423 K. Creep tests were performed under constant load while ABI tests were conducted at varied strain-rates. The strain-rate dependence of the true tensile strength was investigated using constant strain-rate tests from which the stress exponent and the activation energy for deformation were derived. Long-term creep data correlated with those derived from short-term ABI tests. For SnSb, power-law stress dependence (n=5) is noted at low stresses or strain rates and high temperatures, while exponential stress variation is observed at high stresses. The activation energy derived from the power-law region data was low (13 kCal/mol) but in agreement with both creep and tensile data reported earlier. Transitions in deformation mechanisms were observed with distinct values for the stress exponent, and in Sn3.5Ag, the low temperature dislocation climb due to dislocation core diffusion was identified at high stresses. In addition, single lap shear tests were performed on 33x33 solder bump array of Sn3.5Ag which exhibited relatively more scatter. Underlying deformation mechanisms will be discussed.

11:55 AM
TRANSFORMATION SUPERPLASTICITY OF TITANIUM AND Ti-6Al-4V MATRIX COMPOSITES: *Christopher A. Schuh*[1]; Peter Zwigl[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 North Campus Dr., Room 2036, Evanston, IL 60208 USA

Although titanium-based composites exhibit high strength, stiffness, and abrasion resistance, low ductility and hence expensive processing requirements hinder their use. Transformation superplasticity is a deformation mechanism where necking is inhibited and large failure strains

are produced by thermally cycling a material about a solid-state phase transformation under the action of an applied stress. In this paper we investigate the feasibility of forming Ti/TiC and Ti-6Al-4V/TiC composites by transformation superplasticity. The plastic strain induced on each thermal cycle about the transformation range of the matrix is measured as a function of applied stress. The results are considered in the framework of existing continuum mechanics-based models. The room temperature tensile properties of superplastically deformed specimens are related to microstructural evolution during thermal cycling.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Fundamentals

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee, Jt. Extraction & Processing Division and Materials Processing and Manufacturing Division, Synthesis, Control, and Analysis in Materials Processing Committee, Light Metals Division
Program Organizers: Nagy El-Kaddah, University of Alabama, Dept. of Met. & Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Stein Tore Johansen, SINTEF Materials Technology, Process Metallurgy & Ceramics, Trondheim, NTH N-7034 Norway; David G. Robertson, University of Missouri-Rolla, Dept. of Metall. Eng., Rolla, MO 65409-1460 USA; Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Minneapolis, MN 55414-2196 USA

Monday AM Room: 2
March 1, 1999 Location: Convention Center

Session Chairs: Nagy El-Kaddah, The University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Stein Tore Johansen, SINTEF, Materials Technology, Trondheim, Trondelag 7034 Norway

8:30 AM INTRODUCTION AND WELCOME

8:40 AM
MODELLING INTERACTING PHENOMENA DURING THE SOLIDIFICATION OF METALS IN THE CASTING PROCESS : *C. Bailey*[1]; S. Bounds[1]; M. Cross[1]; G. Moran[1]; K. Pericleous[1]; G. Taylor[1]; [1]University of Greenwich, Centre for Numerical Modelling and Process Analysis, Wellington St., Woolwich, London SE18 6PF UK

A variety of interacting complex phenomena takes place during the casting of metallic components. Here molten metal is poured into a mould cavity where it flows, cools, solidifies and then deforms in its solid state. As the metal cools, thermal gradients and solidification shrinkage will promote flow from feeders (risers) towards the mushy zone. Also, as the evolving solid regions of the cast component deform they will form an air-gap at the cast-mould interface. This gap may change the rate of solidification in certain parts the casting. Both fluid flow and solid deformation, occurring throughout the component, will govern the solidification process and the formation of porosity type defects. This paper will present a multiphysics modelling approach to this complex process. Emphasis will be placed on the modelling techniques used. The effects of fluid flow and solid deformation on the solidification process and porosity formation will be detailed. Some comparisons with plant data will also be given.

9:00 AM
MASS TRANSFER MECHANISMS AT FLUID-FLUID INTERFACES: *Stein Tore Johansen*[1]; Knut Bech[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2B, Trondheim, Trøndelag 7034 Norway

The mass transfer at fluid-fluid interfaces due to turbulent agitation is investigated theoretically. In particular a theory is derived for interface stresses due to interface energy and surface renewal. This theory indicates that surface renewal may take place if the geometry is sufficiently large and if surface tension is sufficiently low. By using a model for a typical turbulent structure we compute the interface mass transfer for a stressless interface and a fully rigid interface. Results for the general situation are not presently available. The computed results are used to derive analytical expressions for the mass transfer based on turbulent kinetic energy and freestream turbulent length scales. One important application of the theory is the mass transfer at the electrolyte side of the bath-metal interface in the Hall-Heroult process. For this process we show that mass transfer should be expected to be of the rigid surface type.

9:20 AM
A CFD ANALYSIS OF THE AIR ENTRAINMENT RATE DUE TO A PLUNGING STEEL JET COMBINING MATHEMATICAL MODELS FOR DISPERSED AND SEPARATED MULTIPHASE FLOWS: *Harald Laux*[1]; *Stein Tore Johansen*[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2B, Trondheim 7034 Norway

In most of the steel plants worldwide the major part of alloying material is added during tapping of steel furnaces. In a recent work it has been shown that the yield of the alloying process can be optimized if the alloy size, the alloy injection point, and the addition timing are chosen properly (Berg et al, 1998). As for plunging water jets it can be assumed that the plunging steel jet in the tapping operation entrains air into the steel bath. Possible effects of such air entrainment to the flow pattern in the ladle, as bubble-induced flow, have not been included into the analysis of Berg et al (1998). In air-water models or the tapping process, however, e.g. Tanaka et al (1993) have shown that air entrainment can have a profound impact on the circulation pattern. Air entrainment is also important with respect to undesired nitrogen pick-up by the steel (Choh et al, 1983). Of special interest are the air-entrainment rate, the penetration depth of emerging bubbles, and their average bubble size. For air-water systems measurements of these quantities are easily achieved and thus a large number of experimental works on this topic have been performed as reviewed by Bin (1993). Corresponding experiments are difficult to perform in liquid metals under operation conditions and therefore, to our knowledge, almost no experimental correlations are found in the literature that allow calculating the air entrainment rate of a plunging metal jet. The purpose of this work is therefore to show that the air entrainment rate and its effect to the circulation pattern in the ladle can be obtained by means of CFD. In particular, the presented CFD analysis combines mathematical models and numerical techniques for separated and for dispersed multiphase flows. In a first step the air entrainment rate is computed by using a volume-of-fluid model (VOF; Johansen, 1998). The VOF model is designed for separated flows and can easily provide the air entrainment rate. Because the typical length scales of the gas-bubbles and the overall ladle geometry differ by orders of magnitude, the bubbles, that emerge from the sheet of entrained air that surrounds the jet underneath the surface, can not be resolved on a feasible computational mesh. In a second step, therefore, the computed air entrainment rate is used as inlet condition for a two-fluid model (designed for dispersed multiphase flows; Laux, (1998). The two-fluid model comprises governing equations for the liquid metal including equations for turbulence in the liquid phase (two-equation turbulence model) and the governing equations for the air bubbles. New to the model is an equation that describes the transport of the average diameter of the bubble distribution in each cell of the computational mesh. Essential to this so -called dispersed diameter model is that coalescence and break-up are described. The steel velocities are coupled to the local bubble diameter through the drag term and therefore the effect of the bubbles to the metal flow pattern can be computed. The results of such computations show how the flow circulation pattern of the steel is affected by the entrained air and allows computing the surface area of the bubbles in the steel bath. The latter quantity gives an indication of the possible nitrogen pick-up. Transient and axisymetric computations for both air-water and air-steel systems for one ladle geometry will be presented and compared to each other. The case definitions are such that actual operation conditions in the steel plant are simulated. The results for the air-water system are compared to results from empirical corelations. The obtained computational results will reveal weaknesses of the chosen tapping configuration and will indicate if the mass flow rate of steel, the tapping height, or the alloy addition point have to be changed in order

to improve the yield of the tapping process. References Berg H., Laux H., Johansen S. T. and Klevan O. S. (1998), Flow patterns and alloy dissolution during tapping of steel furnaces, submitted to Ironmaking and Steelmaking. Bin A.K. (1993), Gas entrainment by plunging liquid jets, Chemical Engineering Science, Vol. 48, No. 21, pp. 3585-3630. Choh T., Iwata K. and Inouye M. (1983), Estimation of oxygen and nitrogen absorption of liquid steel during tapping from converter, Transactions ISIJ, Vol. 23, pp. 680-689. Johansen S.T. (1998), Large scale simulation of separated multiphase flows, Third International Conference on Multiphase Flows, Lyon June 8-12, 1998. Laux H. (1998), Modeling of dilute and dispersed fluid-particle two-phase flows, PhD thesis Norwegian University of Science and Technology, submitted April 1998. Tanaka M., Mazumdar D. and Guthrie R.I.L. (1993), Motions of alloying additions during furnace tapping in steelmaking processing operations, Metallurgical Transactions B, Vol. 24B, pp. 639-648.

9:40 AM

SOLID-STATE FLOW ASSOCIATED WITH THE FRICTION-STIR WELDING OF DISSIMILAR METALS: : *L. E. Murr*[1]; Ying Li[1]; R. D. Flores[1]; E. A. Trillo[1]; J. C. McClure[1]; [1]The University of Texas at El Paso, Dept. of Metall. and Mats. Eng., 500 West University Ave., Room M 201, El Paso, TX 79968 USA

Complex vortex and swirl-like solid-state flow phenomena have been observed in the residual microstructures characterizing the friction-stir welding (FSW) of copper to 6061 aluminum and 2024 aluminum as well as 2024 aluminum to 6061 aluminum. These flow phenomena are characterized by intercalation lamellae composed of essentially dynamically recrystallized grain structures of the dissimilar metals which provide a super-plastic-like mechanism for friction-stir weld flow in the solid state; there is no melting and temperatures at the weld center do not exceed about 0.8 TM (where TM is the absolute melting temperature (K)). In experiments to be described optical metallography and transmission electron microscopy techniques are utilized in examining residual FSW microstructures corresponding to tool rotation (stirring) speeds ranging from 400 to 1200 rpm, and traverse (or actual welding) speeds of 1 to 2 mm/s. Supported by NASA-Marshal Space Flight Center Cooperative Agreement NCC-8-137.

10:00 AM BREAK

10:20 AM

FREE SURFACE HORIZONTAL WAVES GENERATED BY LOW FREQUENCY ALTERNATING MAGNETIC FIELDS: S. Daugan[1]; *Yves Fautrelle*[1]; J. Etay[1]; [1]Institut National Polytechnique de Grenoble, CNRS-EPM ENSHMG, B.P. 95, 38402 Saint Martin d'Heres Cedex France

When a liquid metal pool is submitted to an A.C. magnetic field, electrical currents are induced in the liquid metal and interact with the applied magnetic field to create electromagnetic body forces. Those Lorentz forces comprise both a mean value (time average) and an oscillating part. In the particular case where the frequency of the applied magnetic field is low (a few Hertz), the oscillating becomes predominent as compared with the mean one and generate free surface motions. The present paper deals with an other particular case of free surface instabilities generated by low frequency alternating magnetic fields. The liquid pool consists in a mercury layer, which is set on a plane substrate. The liquid puddle is submitted to a vertical low frequency A.C. uniform magnetic field. The present experiment represents a idealized cold model of semi-levitated liquid metal pools. For a given magnetic field frequency, we increase the magnetic field strength from zero to approximately 0.2 T. The observations exhibit three main flow patterns: (i) axisymmetric regime (ii) non-symmetric waves (iii) unstructured regimes From our experiments, we conclude that a uniform low frequency A.C. vertical magnetic field may destabilize a horizontal liquid metal layer.

10:40 AM

MAGNETIC DAMPING OF JETS, VORTICES AND TURBULENCE: *P. A. Davidson*[1]; [1]University of Cambridge, Eng. Dept., Trumpington St., Cambridge CB2 1PZ UK

We discuss the influence of a static magnetic field on jets, vortices and turbulence. This is particularly relevant to D.C. braking in slab casting. Our main conclusion is that, typically, the magnetic field destroys mechanical energy, via joule dissipation, but is unable to destroy momentum. Rather, it continually rearranges the momentum in such a way that the energy falls.

11:00 AM

MHD TURBULENT SHEAR LAYERS: EXPERIMENT AND MODELISATION AT HIGH HARTMANN NUMBER : *Y. Delannoy*[1]; V. Uspenski[1]; K. Messadek[1]; R. Moreau[1]; [1]MADYLAM-ENSHMG, BP95, 38402 St. Martin d'Heres, Cedex France

The MATUR program (Magneto-hydro-dynamic TURbulence) is devoted to the study of turbulent shear flows in high magnetic fields, in conditions such that the large turbulent structures are two dimensional. The presence of a forcing mean shear stress makes this study closer from metallurgical applications than most of the studies of the last decades, that focused on homogeneous turbulence. A new mercury model experiment has been built and tested in uniform magnetic fields of up to 6T, (Ha~1000), ensuring a better two-dimensional behavior than earlier versions (limited to B~0.2T). The quality of the experimental results has been improved: wave number spectras in place of frequency spectras, higher signal levels due to the higher magnetic fields (making it possible to measure directly the vorticity of the turbulent flow). In parallel with these experiments, a numerical Navier-Stokes code was used to perform some two dimensional "direct" simulations, with a source term modelling the Hartmann layer. The condition of closure of the electric current allows to transfer the Hartmann effect into the 2D core flow without simulating the layer itself. No subgrid scale modelling is needed in such a two dimensional situation where most of the energy is carried by the large scales. Two shear layers parallel to the magnetic field are present in our configuration: one is a free shear layer, which becomes highly turbulent at high velocities as shown by the experiments, and the other is a wall shear layer, which seems to remain much more stable even at high velocities. The numerical simulations reproduce this behavior and provide some insight about a regular vorticity eruption from the wall shear layer towards the core flow. This phenomenon can hardly be analysed experimentally because of the low thickness of this wall layer. Its effect on the mean flow and on the momentum transfer will be detailed.

11:20 AM

DIRECT NUMERICAL SIMULATIONS OF HOMOGENEOUS MHD TURBULENCE: *O. Zikanov*[1]; A. Thess[1]; [1]Technical University of Dresden, Institute for Aerospace Engineering, Center for Physical Fluid Dynamics, Dresden D-01062 Germany

Direct numerical simulation methods are applied to study the influence of a constant magnetic field on turbulent flows of liquid metals. The flow is assumed to be homogeneous and the problem is reduced to the classical case of the turbulent flow in a 3D box with periodic boundary conditions. The main subject of the study is the anisotropy developing in a liquid metal flow affected by constant magnetic field. To investigate the long-time evolution of initially isotropic flow the large-scale forcing is applied to maintain the flow energy at a statistically steady level. It is found that the flow evolution depend strongly on the magnetic interaction parameter (Stuart number). In the case of small Stuart number, the flow remains three-dimensional, turbulent, an approximately isotropic. At large Stuart number (strong magnetic field) the turbulence is suppressed rapidly and the flow becomes two-dimensional and laminar. Very interesting is the intermittent flow evolution at moderate Stuart number. Long periods of almost two-dimensional, laminar behavior are interrupted by strong turbulent three-dimensional bursts. The influence of a constant magnetic field on scalar transport properties of liquid metal turbulence is investigated using the simplified formulation of homogeneous flow driven by an imposed mean temperature gradient. Such a flow consists primarily of two turbulent antiparallel jets providing an effective mechanism for heat transfer. It is shown that the magnetic field parallel to the mean temperature gradient stabilizes the jets and, thus, enhances heat transfer considerably.

11:40 AM

ANALYTICAL AND NUMERICAL ANALYSIS OF THE MHD FLOW AROUND A SPHERE IN CROSSED ELECTRIC AND MAGNETIC FIELDS: *Nagy El-Kaddah*[1]; Ashish D. Patel[2]; Thinium T. Natarajan[3]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., P.O. Box

870202, Tuscaloosa, AL 35487-0202 USA; [2]Carpenter TechnologyCorporation, Research Center, Reading, PA 19601 USA; [3]U.S. Steel, Technical Center, Monroeville, PA 15146 USA

The phenomenon of particle migration in a conducting fluid under an applied force field finds its application in selective sorting of minerals and more recently in inclusion removal from molten metal. This paper deals with the analysis of the expulsion force and the flow around a spherical particle in crossed uniform electric and magnetic fields in infinite and in confined medium. Analytical solutions will be presented for the electric and magnetic fields as well as the induced flow at the limits of zero Hartmann number. The flow around the particle was investigated numerically at low and high Hartmann numbers. It will be shown the flow damping effect of the magnetic field is only significant when Hartmann number is larger than 1.0, and the decrease of the velocity at higher Hartmann numbers occurs in the direction of the current as would be expected. It will be also shown that flow modification at high Hartmann number has little effect on the net force on the sphere particle. The significance of these findings on electromagnetic separation of particles in suspensions will be discussed.

GENERAL ABSTRACTS: Session 1 - Mechanical Properties I

Sponsored by: TMS

Program Organizers: Garry W. Warren, University of Alabama, Dept. of Metals and Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Ray D. Peterson, IMCO Recycling Inc., Irving, TX 75039 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Monday AM Room: 12
March 1, 1999 Location: Convention Center

Session Chairs: Mukul Kumar, LLNL; Michael Vinarcik, Ford

8:30 AM

BERYLLIUM-CAPSULE DENSIFICATION AND STRENGTH ENHANCEMENT BY BIASED DEPOSITION: *Alan F. Jankowski*[1]; [1]Lawrence Livermore National Laboratory, Chemistry & Materials Science, P.O. Box 808, L-352, Livermore, CA 94551-9900 USA

A mechanical testing technique is developed to load thin-walled spherical capsules of beryllium under uniaxial tension at constant strain. In addition to the measurement of elastic behavior, application of the tensile load to failure produces yielding and fracture. The capsules are prepared by magnetron sputter deposition of Be onto spherical polymer mandrels. The application of an applied bias to the substrate holder is densifies the columnar microstructure and dramatically increases the material strength. A detailed assessment of capsule mechanical properties is now routinely available using this testing technique which will facilitate the design of spherical capsules for high pressure vessels. This work was performed under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under Contract No. W-7405-ENG-48.

8:55 AM

ON THE MECHANISM OF DEFORMATION INDUCED MAGNETIC TRANSITION IN FeAl: *Ian Baker*[1]; Yong Yang[1]; Patrick Martin[2]; [1]Dartmouth College, Thayer School of Engineering, Tuck Drive, Hanover, NH 03755 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA

Single crystals of Fe-40Al were cold-rolled to a variety of strains up to 48%, when cracking occurred. The rolled crystals were heated at 10 K/min to 973K in a differential scanning calorimeter, and three exothermic peaks were observed. The cold rolling also induced a transition from paramagnetism to ferromagnetism. At room temperature, the ferromagnetism disappeared upon annealing above the lowest temperature exothermic peak, but at temperatures below 225K the annealed specimen still showed a larger magnetic susceptibility compared to the virgin single crystal. Analysis of the possible contributions to the ferromagnetic behavior suggests that antiphase boundaries (APB), principally in APB tubes, are the source. This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences through Contract DE-FG02-87ER45311.

9:20 AM

DEFORMATION IN TANTALUM SINGLE CRYSTALS: TEMPERATURE DEPENDENCE AND LATENT HARDENING.: *Rajeev Kapoor*[1]; [1]University of California, San Diego, Mats. Sci., 9500 Gilman Drive, La Jolla, CA 92093 USA

Uniaxial compression tests were carried out on single crystal tantalum to study the temperature sensitivity of flow stress at high strain rates (3000/s) on the {211}<111> and {101}<111> slip systems. The temperature dependence at high strain rates for the two systems, {211}<111> and {101}<111>, was found to be very similar. Compressive deformation carried out at 77K on single crystal tantalum exhibited twin formation down to strain rates of 0.01/s. On the other hand, high strain rate experiments carried out at room temperature did not show any features resembling twin formation. Further experiments were carried out to study latent hardening at high strain rates on the {211}<111> slip system. A latent hardening ratio of 1.10 was observed on intersecting {211} planes. The temperature dependence of the latent system was similar to that of the primary system. From this it was concluded that latent hardening occurs because of an increase in the athermal component of stress, with the thermal component of stress remaining unchanged. It is emphasized that this conclusion is valid only for the {211}<111> family of intersecting slip systems.

9:45 AM

INFLUENCES OF STRAIN RATE AND GRAIN SIZE ON YIELD AND SERRATED FLOW IN Al-Mg ALLOY 5086: *Matthew Wagenhofer*[1]; Marjorie E. Natishan[1]; *Ronald W. Armstrong*[1]; Frank J. Zerilli[2]; [1]University of Maryland, Dept. of Mech. Eng., College Park, MD 20742 USA; [2]Naval Surface Warfare Center, Indian Head Division, 101 Strauss Ave., Indian Head, MD 20640 USA

Laboratory tests have been performed at two strain rates (0.001 and 0.36 [1/s]) and for two different material conditions spanning the treatment of as-supplied commercial pipe stock material, as follows: (1) annealed material at larger and smaller grain sizes; and, (2) after the 20% cold work portion only of the H32 treatment. Dynamic strain aging (from dislocation-solute interactions) produced significant serrations in flow curves obtained at the lower strain rate and, consequently, a reversed strain rate effect manifested by higher true stress - true strain curves, including greater uniform strains — for all material conditions. Also, the cold worked material exhibited a substantially greater Hall-Petch (stress versus reciprocal square root of grain size) dependence that was employed on a comparative basis to demonstrate that the recovery part of the H32 treatment trades flow strength reduction for the advantage of enhanced ductility. The results are connected with Zerilli-Armstrong predictions (J. Appl . Phys. 1987) of expected strength increase with strain rate on a dislocation intersections basis and with a Hall-Petch comparison of different Mg alloy strengthening behaviors (Armstrong and Douthwaite 1995).

10:10 AM BREAK

10:20 AM

EFFECTS OF PLASTIC DEFORMATION ON MAGNETIC AND MECHANICAL PROPERTIES OF 3Y-ZrO2/BaFe12O19 COMPOSITES: *Yoshikazu Suzuki*[1]; Masanobu Awano[1]; Naoki Kondo[1]; Tatsuki Kondo[1]; [1]National Industrial Research Institute of Nagoya, 1-1 Hiratecho, Kita-ku, Nagoya, Aichi 462-8510 Japan

3Y-ZrO2/BaFe12O19 (barium M-type hexaferrite) composites were fabricated by powder metallurgical processes, and their mechanical and magnetic properties were evaluated. Plastic deformation on the composites improved their mechanical and magnetic properties due to the formation of anisotropic microstrucuture.

10:45 AM

THE EFFECT OF IMPURITIES ON SLIDING BEHAVIOR IN SUPERPLASTIC Zn-22%Al ALLOY (PART 1): *Kim Duong*[1]; [1]University of California, Irvine, Dept. of Chem. & Biochem. Eng. and Mats. Sci., 916 Engineering Tower, Irvine, CA 92697-2375 USA

Present work was undertaken to study the effect of impurity content on boundary sliding behavior in regions I, II, and III. In conducting the investigation, three grades of Zn-22% Al were used: grades 1 and 2 contain 180 and 100 ppm of impurities, respectively, whereas grade 3 is a high-purity grade containing 6 ppm of impurities. The experimental results show that at intermediate strain rates (region II), the sliding behavior of the three grades of Zn-22% Al is similar and that the contribution of boundary sliding to the total strain, x, is about 60%. By contrast, the experimental data reveal that at low stain rates, the three grades exhibit significant differences regarding the sliding contribution. These differences are manifested in the following observations: (i) ξ in grade 3 at low strain rates is essentially equal to that at intermediate strain rates (region II), (ii) ξ in grade 1 or 2 is considerably lower than that at intermediate strain rates, and (iii) for the same low strain rate, ξ in grade 2 is higher than that in grade 1. The above observations regarding the effect of impurity level on boundary sliding behavior in Zn-22% Al are consistent with the concept of the interpretation of superplastic flow at low strain rates (low stresses) in terms of boundary segregation.

11:10 AM

FORMATION OF CAVITY STRINGERS DURING SUPERPLASTIC DEFORMATION: *Ahmadali Yousefiani*[1]; Farghalli A. Mohamed[1]; [1]University of California at Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA

The Zn-22 pct Al eutectoid was utilized as a model material to investigate cavity stringer formation during superplastic deformation. Apart from an elevated testing temperature, the main requirement for the occurrence of micrograin superplasticity in metallic systems is a fine and stable grain size of less than 10 μm. Such a condition is easily achieved in Zn-22 pct Al through solution treatment above the eutectoid temperature, followed by rapid quenching. Microstructural observations on Zn-22 pct Al following solution treatment have indicated the presence of residual grain boundaries (referred to as former α boundaries). These former α boundaries (FαBs) represent domains that encompass groups of fine α (Al-rich) and β (Zn-rich) phases and consist of fine elongated α grains. The present results show that, during superplastic deformation, FαBs exhibit two primary characteristics: (a) they serve as favorable cavity nucleation sites, and (b) they change their orientation and become aligned with the tensile axis (i.e., they act as natural tracers for superplastic flow). A comparison between the behavior of FαBs and the characteristics of cavity stringers, which form in Zn-22 pct Al and align parallel to the tensile axis during deformation, has revealed a direct correspondence between the evolution of these two substructural features. The findings, which are primarily based on quantitative correlations among the morphological features of FαBs (ex. average size, alignment, and local deformation) and those of cavity stringers (ex. total length and distribution during deformation), have not only rationalized the origin of cavity stringers in Zn-22 pct Al, but also provided a general explanation for cavity stringer formation under superplastic conditions.

11:35 AM

THE INFLUENCE OF IMPURITY TYPE ON SUPERPLASTIC FLOW AND CAVITATION IN Zn-22 PCT Al: *Ahmadali Yousefiani*[1]; Farghalli A. Mohamed[1]; [1]University of California at Irvine, Department of Chemical and Biochemical Engineering and Materials Sciences, 916 Engineering Tower, Irvine, CA 92697-2575 USA

The present investigation was conducted to study the effect of Cu, as a selected impurity, on superplastic deformation and cavitation in Zn-22 pct Al. The results show that Zn-22 pct Al-0.13 pct Cu exhibits two primary characteristics: region I is absent and cavitation is not extensive. These characteristics, which are essentially similar to those reported previously for high-purity Zn-22 pct Al but are different from those documented for a grade of the alloy containing a comparable atomic concentration of Fe, suggest that Cu has little or no tendency to segregate at boundaries. Indirect evidence in support of this suggestion is inferred from studying the effect of impurities on former α boundaries that form in the microstructure of Zn-22 pct Al as a result of solution treatment above the eutectoid temperature. The findings have led to the conclusion that, under the condition of superplastic deformation at low stresses, the emergence of region I and the occurrence of cavitation are controlled not only by impurity level but also by its type.

GENERAL ABSTRACTS: Session 2 - Ceramic & Refractory Materials & Ceramic/Metal Interfaces

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Program Organizers: Garry W. Warren, University of Alabama, Dept. of Metals and Mas. Eng., Tuscaloosa, AL 35487-0202 USA; Ray D. Peterson, IMCO Recycling, Inc., Irving, TX 75039 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Monday AM — Room: 13
March 1, 1999 — Location: Convention Center

Session Chairs: Viola L. Acoff, The University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Samuel Davis, TIMET, K-52, Henderson, NV 89009 USA

8:30 AM

NEW PORCELAIN BODIES IN THE SYSTEM OF HOLLOW GLASS MICROSPHERES-QUARTZ-ALUMINOUS CEMENT: *Kunio Kimura*[1]; Hiroshi Tateyama[1]; Noriyuki Yamada[1]; Kazuhiko Jinnai[1]; Weon-Pil Tai[2]; [1]Kyushu National Industrial, 807-1 Shuku-machi, Tosu, Saga 841-0052 Japan; [2]Japan Small Business Corporation, Information and Technology Depart., Toranomon, Minato-ku, Tokyo 105-8453 Japan

New porcelain bodies with lightweight and high-strength properties were fabricated using non-plastic raw materials, such as hollow glass microspheres, quartz and constant content of 20wt% aluminous cement. Hollow glass microspheres were produced using natural volcanic glass particles by heat treatments. Green strength became constant with hydration time ranges of 24h. The phases formed by heat treatment at 1300ϒC for 1h were alpha-quartz, alpha-cristobalite, anorthite, glass and a small amount of alpha-Al2O3. The characteristics of the fired body of 40wt% hollow glass microspheres - 40wt% quartz - 20wt% alminous cement body were as follows; flexural strength was about 60MPa, bulk density was about 2.0g/cm3 and water absorption was almost 0%. This work was supported by Japan Small Business Corporation, as part of the Creative and Fundamental R&D Program for SMEs.

8:55 AM

GELCASTING FOR QUARTZ-ALUMINA SUSPENSION BY USING TWO-FLUID NOZZLE: *Noriyuki Yamada*[1]; Kunio Kimura[1]; Hiroshi Tateyama[1]; Kazuhiko Jinnai[1]; Weon-Pil Tai[2]; [1]Kyushu National Industrial Research Institute, 807-1 Shuku-machi, Tosu, Saga 841-0052 Japan; [2]Small Business Corporation, Information and Technology Depart., Toranomon, Minato-ku, Tokyo 105-8453 Japan

Gelcasting method was attempted to quartz-alumina suspension using gelatin as a gelation substance. Glutaraldehyde and formaldehyde were used as an agent of crosslinking of the gelatin. Two-fluid nozzle was used for casting of the suspension into the mold, because it was simultaneously able to feed both of the suspension and the crosslinking agent. The influence of composition of the suspension on the gelation time and the toughness of the green body were investigated in this study. The suspension was flowed downward into the mold in order to fill all the space of the mold. A retard agent for gelation was added with vibrating and tapping of the mold. Solidification time was shortened with increasing additives, such as the gelatin, the crosslinking agent and the retared

agent. Green body with the same physical properties as an ordinary plastic green body was obtained by using two-fluid nozzle. In this case, solidification time was less than 30 minute. This work was supported by Japan Small Business Corporation, as part of the Creative and Fundamental R&D Program for SMEs.

9:20 AM

INTERFACIAL REACTIONS BETWEEN Cu and Al2O3 DURING EUTECTIC BONDING: *Seonghoon Yi*[1]; Kevin P. Trumble[1]; David R. Gaskell[1]; [1]Purdue University, School of Mats. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907-1289 USA

The interfacial reactions between Cu and Al2O3 which occur during the eutectic bonding process have been examined metallographically to determine the conditions required for the formation of CuAlO2. The experimentally-determined invariant state in which solid and liquid copper, CuAlO2 and Al2O3 are in equilibrium was found to be in good agreement with that calculated from independent thermodynamic data. At lower temperatures the CuAlO2 phase forms as isolated accicular needles and at higher temperatures no CuAlO2 was observed to form at the interface between Al2O3 and hypo-eutectic melts in the system Cu-O. Partial isothermal sections of the phase diagram for the system Cu-Al-O have been constructed from observed microstructures.

9:45 AM

USE OF METALLIC-GLASSES IN CERAMIC-METAL JOINING: *Rajendra U. Vaidya*[1]; Partha Rangaswamy[1]; Mark A. M. Bourke[1]; Darryl P. Butt[1]; [1]Los Alamos National Laboratory, Materials Science Division, Mail Stop G 755, Los Alamos, NM 87545 USA

Residual stresses due to mismatch in elastic and thermal expansion properties in ceramic-metal joints can lead to failure at the interface or within the brittle ceramic. Low temperature brazing techniques coupled with ductile interlayers alleviate this problem. However, the use of precious metal based brazes and the incorporation of the interlayer add to the complexity and cost of the joining process. To overcome these problems, we propose the use of metallic-glass brazes. This is a new idea that will eliminate the need for separate interlayers in ceramic-metal joining. Metallic-glasses can be bent and twisted into complicated geometries. Since they melt more uniformly and at lower temperatures (compared to the base metal from which they are derived), diffusion and dissolution at the joint is enhanced and should provide greater joint strengths. Furthermore, metallic-glasses are cheaper than conventional brazes which can consist of large amounts of precious metals such as gold and silver. We present the results of our preliminary joining experiments using metallic glasses. Stainless steel 316L and molybdenum disilicide were successfully brazed using a cobalt based metallic glass. Issues pertaining to the interfacial chemistry, joint strength and residual stresses are presented here.

10:10 AM

ACTIVE BRAZING OF ALUMINA WITH VANADIUM ADDITIONS*: *F. Michael Hosking*[1]; Chuck H. Cadden[1]; S. Jill Glass[1]; John J. Stephens[1]; Paul T. Vianco[1]; Chuck A. Walker[1]; [1]Sandia National Laboratories, P.O. Box 5800, MS1411, Albuquerque, NM 87185 USA

The development of active brazing for engineered ceramics requires a fundamental understanding of the braze microstructure and interfacial reactions. This presentation discusses the materials and processing issues associated with brazing alumina and cermet in hydrogen with V-containing active filler metals. The alloys were based on the Au-18Ni system. Wetting behavior, braze microstructure, and tensile strength were studied. Wetting was generally good, although braze flow was limited. SEM/TEM analysis identified a spinel reaction at the braze interface. Most joints were hermetic. Tensile response was also good. The 94% alumina samples had a nominal strength of 90-100 MPa, with failures in the ceramic or at the braze interface. * Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

10:35 AM

BONDING REFRACTORY METALS BY PLASMA ACTIVATED SINTERING AND ITS EVALUATION: *Shin-ichi Sumi*[1]; Yoshiki Mizutani[1]; ZhengMing Sun[1]; Toshihiko Abe[1]; [1]Tohoku National Industrial Research Institute, Materials Engineering Division, 4-2-1, Nigatake, Miyagino-ku, Sendai, Miyagi 983-8551 Japan

Purpose of this research is to make the ion gun parts (arc chamber) of ion implantation devices by bonding method by plasma activated sintering. Usually, molybdenum is used for the parts. With increasingly high integration in IC memory, the problem of contamination of wafer due to molybdenum arose and therefore the parts made of tungsten are preferred by the industries. However, tungsten is too hard to be formed into the parts by conventional machining process. The bonded joints of tungsten in the different bonding conditions were evaluated by an optical microscope, an ultrasonic imaging and four point bending test. As a result, bending strength of the joints with interlayer of tantalum powder, tungsten powder and without any interlayer was measured to be about 400, 200 and 100MPa, respectively.

11:00 AM

THERMAL SPRAY AND MECHANICAL PROPERTIES OF NANOSTRUCTURES OXIDE COATINGS: Leon L. Shaw[1]; Ruiming Ren[1]; Daniel Goberman[1]; Maurice Gell[1]; Stephen Jiang[2]; You Wang[2]; T. Danny Xiao[2]; Peter R. Strutt[2]; [1]University of Connecticut, Institute of Mats. Sci., Stores, CT 06269; [2]Inframat Corporation, North Haven, CT USA

Nanostructure coatings can provide significant improvements in wear and erosion resistance deriving from enhanced hardness and toughness. In this paper, Al203 - 13 wt.% Ti02 coating formed via thermal spray approach using reconstituted nanosized Al203 and Ti02 powder feeds are described. The microstructure, microhardness, indentation toughness, grain size and wear resistance of the coatings from the reconstituted nano-powder feeds have been characterized and compared to those obtained from commercial coating counterparts. The properties of the coating obtained from reconstituted nano-powder feeds are discussed and related to thermal spray conditions.

11:25 AM

METALLOTHERMIC PREPARATION OF SILICON FROM LOCAL RAW MATERIALS: *S.Z. El-Tawil*[1]; K.A. El-Barawy[1]; I.M. Morsi[1]; M.M. Nasr[1]; M.R. El-Dessouki[1]; [1]Central Metallurgical R & D Institute, P.O. Box 87, Helwan, Cairo, Egypt

Silica is the main raw material for the production of silicon metal. In Egypt, there are huge amounts of silica raw materials such as white sands, quartz and quartzite at different localities varying in geological amounts of reserves and grades in Eastern Desert and Sinai Peninsula. Also, there are secondary resources of silica produced as dust from the ferrosilicon plants at Idfu (EFACO) and Aswan (KIMA), Egypt. The diversity of important applications of elemental silicon and its compounds has led to the development of different processes for its preparation depending on the required quality of the end product. Commercial application of feasible process for the production of silicon metal is of a vital importance to the national economy. Also, the exploitation of the natural resources such as silica for the production of such important product to substitute the imported one and supply the local market needs, thus saving hard currency, is profitable and essential. The recent study dealt with the processing of local primary and secondary silica resources for the preparation of silicon metal using the aluminothermic reduction technique (thermite process). This process proceeds without the use of electric furnaces and employs simple and cheap equipment. It consists of three stages; firstly, aluminothermic reduction of silica to prepare Al-Si alloys with a silicon content $\geq$ 50%; secondly, recovery of elemental silicon from the produced alloys by hydrochloric acid leaching; and thirdly, refining of produced silicon by an additional leaching with acid mixtures. The operating technical parameters affecting the preparation process were studied. Polycrystalline silicon of ~99.99% purity has been prepared as a final product and was examined by X-ray diffraction, scanning electron microscopy and inductively coupled plasma-emission spectrometry (ICP-ES).

HIGH-TEMPERATURE SUPERCONDUCTORS: SYNTHESIS, FABRICATION AND APPLICATION: YBCO Superconductors

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Superconducting Materials Committee
Program Organizers: U. Balu Balachandran, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 212, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, 450 Old Niskayuna Rd., Latham, NY 12110 USA; Chandra Pande, Naval Research Lab, Mats. Sci. & Tech. Div, Washington, D.C. 20375-5000 USA

Monday AM Room: 18
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Session Chairs: Paul J. McGinn, University of Notre Dame, Dept. of Chem. Eng., Notre Dame, IN 46556 USA; Donglu Shi, University of Cincinnati, Dept. of Mats. Sci. & Eng., Cincinnati, OH 45221-0012 USA

8:30 AM INVITED PAPER
CATION SUBSTITUTION, SECOND PHASE PRECIPITATION, SUPERCONDUCTIVITY, AND FLUX PINNING IN LR1+xBa2-xCu3O7: *Ralph William McCallum*[1]; Hengning Wu[1]; Mathew J. Kramer[1]; Kevin W. Dennis[1]; [1]Iowa State University, Ames Laboratory, 106 Wilhelm, Ames, IA 50011 USA

Unlike YBa2Cu3O7+d which forms only a stoichiometric compound, the light rare earth (LRE) elements form a solid solution of LRE1+xBa2-xCu3O7+d (LRE123ss) with increasing substitution of the rare earth for Ba2+ as the ionic radii of the rare earth increases. The effects of oxygen partial pressure (Po2) on the solubility limits of LRE123ss (LRE = Pr, Nd, Sm, Eu, and Gd) were studied by differential thermal analysis, X-ray diffraction and measurement of superconducting transition temperature (Tc). An understanding of the Po2 and temperature dependence of the solubility limits will not only provide guidelines for obtaining high Tc in LRE123ss but also clarify the flux pinning mechanism in these systems. For LRE= Nd, Sm, Eu and Gd, varying the Po2 between high and low temperature heat treatments allows the precipitation of a non superconducting second phase. If the distribution of this phase is properly controlled as is easily accomplished for Nd, the microstructure results in enhanced flux pinning.

8:50 AM INVITED PAPER
DEPOSITION OF C-AXIS TEXTURED YBa2Cu3Ox ON A FLEXIBLE METALLIC SUBSTRATE THROUGH DIRECT PERITECTIC SOLIDIFICATION: *Donglu Shi*[1]; Dehui Qu[1]; Xuejun Wen[1]; Brian A. Tent[1]; Mike Tomsic[2]; [1]University of Cincinnati, Dept. of Mats. Sci. and Eng., 493 Rhodes Hall, Cincinnati, OH 45221-0012 USA; [2]Plastronic Co., 11641 N. Dixie Dr., Tipp City, OH 45371 USA

Previous work in the development of YBa2Cu3Ox (YBCO) superconducting wires and tapes has been focused on the deposition of YBCO on buffered metallic substrates. Although such an approach has proved successful in terms of achieving grain texturing and high transport current density, critical issues involving continuous processing of long length conductors and stabilization of the superconductor have not yet been entirely settled. We have developed a novel process, the so-called direct peritectic growth (DPG), in which textured YBCO thick films have been successfully deposited directly onto a silver alloy substrate. No buffer layer is employed in the film deposition process. The textured YBCO grains have been obtained through peritectic solidification over a wide range of temperatures and times. The substrate materials have not demonstrated any observable reaction with the YBCO melt at the maximum processing temperature near 1010ŸC. The transport Jc has reached a respectable value of 104 A/cm2 at 77 K and zero magnetic field. Based on the experimental results in this work, we show that the DPG method offers an effective alternative for the fabrication of long-length YBCO conductors. Also reported is a physical explanation of the texturing mechanism on the metal substrate.

9:10 AM INVITED PAPER
THERMAL CYCLE METHOD FOR OBTAINING LARGE 123 SINGLE DOMAINS: *Rudi Cloots*[1]; Françoise Auguste[1]; Paulette Clippe[2]; Nicolas Vandewalle[2]; *Marcel Ausloos*[2]; [1]University of Liege, SUPRAS, Institute of Chemistry, B6, Liege B-4000 Belgium; [2]University of Liege, SUPRAS, Institute of Physics, B5, Liege B-4000 Belgium

Numerical and experimental investigations of a new method allowing for the growth of large 123 single domains have been implemented. The process is based on the hypothesis that large grain coarsening occur when the system is slightly remelted after the initial cooling below the peritectic temperature. Various isothermal and variable temperature conditions have been investigated. For comparison seeding processing conditions have been considered. Microstructural investigations have been performed.

9:30 AM INVITED PAPER
MAPPING LOCAL MAGNETIC, OPTICAL AND MAGNETO-OPTICAL CHARACTERISTICS OVER THE SURFACE OF A MELT TEXTURED HIGH-Tc SUPERCONDUCTOR: Valter Ström[1]; *K. V. Rao*[1]; B. Balachandran[2]; T. H. Johansen[3]; M. Baziljevich[3]; [1]Royal Institute of Technology, Dept. of Condensed Matter Physics, Stockholm SE 100 44 Sweden; [2]Argonne National Laboratories, Ceramics and HTSC, Argonne, IL 60439 USA; [3]University of Oslo, Physics, Oslo 0316 Norway

A new method to determine and map local susceptibility over a submicron range of surfaces has been developed using a pair of reade/write Head probe. By this approach we determine and map over a large area the local magnetic anisotropy and its evolution with temperature, both below and above the superconducting transition in a HTSC. In our studies of a bulk melt quenched YBCO we find that only the 'in-phase' uniaxial fundamental suceptibility is uneffected by the temperature and is precisely related to the local crystal orientation. By mapping the higher harmonics of the suceptibility we determine the distribution of Tc, as well as the critical current Jc over the surface. The susceptibility studies are then compared with MOKE-imaging investigations of the same surface. Thus a correspondence between the boundaries of different crystal orientation and the penetrating magnetic fields is now possible. Our approach provides a universal approach to map and interpret local magnetic, optical and magneto-optical properties of a surface.

9:50 AM INVITED PAPER
EFFECTS OF GROWTH CONDITIONS ON SUPRECONDUCTING PROPERTIES OF MELT GROWN (Sm_xGd_{1-x})-Ba-Cu-O SUPERCONDUCTORS: *Seok-Jong Seo*[1]; Naomichi Sakai[1]; Masato Murakami[1]; [1]SRL-ISTEC, Div. 3, 16-25, Shibaura 1-Chome, Minato-ku, Tokyo 105 Japan

REBa2Cu3Oy (RE123, RE; Nd, Sm, Eu, Gd) and their intermixture compounds are known to exhibit high Tc with sharp superconducting transition and large Jc in high fields at 77 K when they are melt-processed in a reduced oxygen atmosphere. In this study, we systematically investigated the growth rates, microstructures, the distribution of (SmxGd1-x)211 phase and superconducting properties (Tc and Jc-B) of the binary mixed (SmxGd1-x)123 bulk superconductors melt-processed under various conditions. The melt process was performed with/without Nd-Ba-Cu-O seed crystals in 1%O2-Ar atmosphere. The growth rates of melt-grown (SmxGd1-x)123 bulks showed an increasing trend with increasing undercooling. The Jc-B properties of melt-grown (SmxGd1-x)123 bulks were sensitive to x, while Tc was almost independent of x. It was also found that Nd-Ba-Cu-O seeds were effective in fabricating textured (SmxGd1-x)123 single grains. This work was partially supported by NEDO.

10:10 AM BREAK

10:20 AM INVITED PAPER
FABRICATION AND MICROWAVE PROPERTIES OF $YBa_2Cu_3O_y$ FILMS ON BUFFERED POLYCRYSTALLINE COPPER SUB-

STRATES: *Kyoko Kawagishi*[1]; Kazunori Komori[1]; Masao Fukutomi[1]; Kazumasa Togano[1]; Jian-Fei Liu[2]; Shigemi Inagaki[2]; Kiyomitsu Asano[2]; Eiji Ezura[2]; [1]National Research Institute for Metals, 1st Research Group, 1-2-1 Sengen, Tsukuba-shi, Ibaraki 305-0047 Japan; [2]High Energy Accelerator Research Organization, Accelerator Research Group, 1-1 Oho, Tuskuba-shi, Ibaraki 305-0801 Japan

For some microwave applications of high-temperature superconducting films, use of polycrystalline substrates might be desirable. We fabricated $YBa_2Cu_3O_y$ (YBCO) thin films on buffered Cu substrates using a vapor plating technique. The application of tri-buffer layers; chromium, amorphous yttria-stabilized zirconia (YSZ), and in-plane textured YSZ layers was attempted to solve the problem of the chemical and structural mismatches between YBCO and Cu. The amorphous YSZ worked successfully as a stress relaxation layer, resulting in excellent adhesion at the interface between the textured YSZ and Cr layers. The textured YSZ layers were deposited by a modified bias sputtering (MBS) technique we previously proposed. Microwave surface resistance, Rs of YBCO films obtained was measured using a dielectric resonator technique at 13 GHz. The lowest Rs attained so far was 4 mΩ at 77 K. A strong correlation was observed between the Rs and the in-plane texturing of the YBCO films. More effort is underway to reduce the surface resistance of YBCO films by improving the degree of texturing of MBS-YSZ buffer layers. These results are encouraging for potential cavity applications of these materials.

10:40 AM INVITED PAPER
THE EFFECTS OF Mg-Ce ADDITIONS ON THE MAGNETIC PROPERTIES OF TEXTURED YBa2Cu3O7: *Paul J. McGinn*[1]; Sharon C. Yeung[1]; [1]University of Notre Dame, Dept. of Chem. Eng., 178 Fitzpatrick, Notre Dame, IN 46556 USA

The effects of $BaCeO_3$ additions in combination with MgO additions on the magnetic properties of melt textured $YBa_2Cu_3O_{7-x}$ have been investigated. The additions lead to improvements in the magnetic properties of $YBa_2Cu_3O_{7-x}$ compared to samples with either addition alone or with no additions. The Ce-Mg addition combination produces a "peak effect" in the magnetic hysteresis loop. This is postulated to be due to the formation of pinning centers. Both Ce and Mg ions are thought to substitute in the $YBa_2Cu_3O_{7-x}$ lattice, creating defects that produce a "peak effect" in the magnetic hysteresis loop. Mg additions alone lead to a reduced Tc, while Ce additions restore the Tc and enhance the magnitude of the peak. Similar effects have also been observed in $ErBa_2Cu_3O_{7-x}$ doped with the Ce-Mg combination.

11:00 AM
MANIPULATION OF YBa2Cu3O7-x THICK FILM PROCESSING TO GIVE IMPROVED MICROSTRUCTURAL CHARACTERISTICS: *Jason B. Langhorn*[1]; *Paul J. McGinn*[1]; [1]University of Notre Dame, Dept. of Chem. Eng., South Bend, IN 46656 USA

It is apparent from the characterisation of superconducting YBa2Cu3O7-x (YBCO) thick films processed by melt texturing on yttria-stabilised zirconia substrates that the microstructural properties are highly dependent upon the stoichiometry of the precursor powder. Increased grain sizes and texture have been observed in thick films processed both by a modified powder melt process (PMP) and a solid-liquid melt-growth (SLMG) method. These processes involve the use of BaCuO2 / CuO / Y2BaCuO5, and BaCuO2 / CuO / Y2O3 precursors respectively, mixed in the cationic ratio of 1Y: 2Ba: 3Cu. Cross sectional analysis of such films has also shown a decreased size and increasingly homogeneous distribution of Y2BaCuO5 (211) particles throughout the matrix with respect to films processed from YBCO precursors. It has also been proposed that the spherulitic nucleation and growth characteristics of YBCO thick films, melt processed on yttria-stabilised zirconia (YSZ) substrates, occurs due to the reaction of the melt with the YSZ. By the manipulation of the substrate film interface it has been shown that the position at which nucleation of the spherulites occurs can be controlled.

11:20 AM INVITED PAPER
FLUX-PINNING-INDUCED MAGNETOSTRICTION AND INTERNAL STRESS DISTRIBUTIONS IN BULK SUPERCONDUCTORS: *Tom H. Johansen*[1]; Jens Lothe[1]; [1]University of Oslo, Dept. of Phys., P.O. Box 1048 Blindern, Oslo 0375 Norway

The magneto-elastic behavior of (RE)Ba2Cu3Ox, which today can be grown with melt-texturing to large-size monoliths, is an important issue for their use in high-field applications like trapped-field magnets etc. A severe problem with high-field conditions is that the pinning-induced stresses easily grow to values that will cause material fracture. We have investigated theoretically the irreversible behavior of stress and strain in two realistic geometries; a thick superconductor of circular (i) and square (ii) cross-section. In contrast to case (i), where the circular shape is conserved, the non-central bodyforces in case (ii) are shown to generate large shape distortion with a wide variety of deformation types. Exact analytical results for the quantitative deformation and internal stresses are presented. Special emphasis is put on the magnetized states with large tensile stress, where cracking easily destroys the usefulness of the monoliths because the supercurrent loops shrink in size and reduce the total magnetic moment.

11:40 AM INVITED PAPER
EVOLUTION OF CUBE-TEXTURE IN LAMINATED Ni/Ag/SS310S SHEET: Hee-Gyoun Lee[1]; *GyeWon Hong*[1]; [1]Korea Atomic Energy Research Institute, Functional Materials Laboratory, P.O. Box 105, Yusong, Taejon 305-600 Korea

Strong metallic substrate having (100)<001> cube texture was successfully fabricated by joining of three different metal sheets followed by cold rolling and texture anneal. Joining of Ni and stainless steel was performed by vacuum brazing method using Ag as filler metal. After heat treating the thin Ni/Ag/SS310S sheet at 900℃ for 2h, Ni (111) pole figure for the nickel surface demonstrated the development of (100)<001> cube texture. Quantitative chemical analysis of EPMA was made for the cross-section of the Ni/Ag/SS310S sheet. EPMA results showed that Ag diffusion into the Ni layer, which may suppress the cube texture development, was negligible. Small amount of Cr, Fe atoms was detected in the Ni layer. It showed that the role of Ag as a chemical barrier of alloying element atoms in Ni layer for the Ni/Ag/SS310S sheet was successful so that strong cube texture was developed for the Ni layer in the Ni/Ag/SS310S sheet.

HIGH TEMPERATURE COATINGS III: Thermal Barrier Coatings - I

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee; Jt. ASM International: Materials Science Critical Technology Sector/TMS Structural Materials Division, Corrosion and Environmental Effects Committee
Program Organizers: Janet Hampikian, Georgia Tech, School of Mats. Sci. & Eng., Atlanta, GA 30332-0245 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Ctr. for Laser Applications, Tullahoma, TN 37388 USA

Monday AM Room: 19
March 1, 1999 Location: Convention Center

Session Chairs: William P. Allen, Pratt Whitney, East Hartford, CT USA; Janet M. Hampikian, GA Institute of Technology, School of Mats. Sci. and Eng., Atlanta, GA 30332-0245 USA

8:30 AM OPENING REMARKS

8:35 AM KEYNOTE
MICROSTRUCTURAL STUDIES OF THERMAL BARRIER COATINGS: *Manfred Ruhle*[1]; E. Schumann[1]; E. Sommer[1]; [1]Max-Planck-Institut Fur Metallforschung, Seestrasse 92, Stuttgart D-70174 Germany

The performance of thermal barrier coatings is determined predominantly by the behavior of the 1μm thick oxide scale, formed beneath the zirconia layer. Several factors may affect the cracking behavior of that

interface. These factors include the composition of the alloy substrate and the coating as well as thermal stresses. Studies by advanced TEM techniques reveal the microstructure, composition, and bonding at the interfaces and regions close to interfaces in that area. The results will be reported and discussed using different models which are required for an explanation of the failure behavior. Migration of metal elements (of the bond coat) into the metallic substrate, segregation to the interface between the bond coat and the oxide scale and formation of stresses in the scale and formation of stresses in the scale are expected to play a major role.

9:05 AM

PLASMA PROCESSING OF EB PVD THERMAL BARRIER COATINGS TO REDUCE THERMAL CONDUCTIVITY: *J. R. Nichols*[2]; K. J. Lawson[2]; D. S. Rickerby[3]; P. Morrel[3]; M. B. Henderson[1]; [1]Defence Evaluation and Research Agency, Structural Materials Centre, Griffith Bldg., Ively Rd., Farnborough, Hampshire GU14 OLX UK; [2]Cranfield University, School of Indust. and Manuf. Sci., Cranfield, Bedford MK43 OAL UK; [3]Rolls Royce PLC, Surface Eng. Group, P.O. Box 31, Derby DE24 8BJ UK

EB-PVD thermal barrier coatings (TBC's) are extensively being researched as a coating system that can be applied to rotating components within the modern gas turbine. The electron beam, physical vapour deposited coatings have a columnar microstructure, which provides strain tolerance, and can reduce metal surface temperatures by up to 150ϒC. The measured thermal conductivity of this type of coating is typically 1.8 2.0 W/mK, falling short of the values reported for plasma sprayed ceramics (0.9 1.0 W/mK). This paper examines the role of the EBPVD ceramic microstructure on the thermal conductivity, reviews methods by which the thermal conductivity can be reduced, and then demonstrates that by introducing layers within the columnar microstructure the thermal conductivity of the EB-PVD TBC can be reduced 30 - 40%. The layers are introduced by plasma enhanced, electron beam physical vapour deposition. The benefits of such layered structures in scattering the thermal wave is discussed.

9:25 AM

ON THE EVOLUTION OF TEXTURE AND POROSITY IN EB-PVD TBCs: *Scott G. Terry*[1]; Jennifer R. Litty[1]; Carlos G. Levi[1]; [1]University of California, Mats. Dept., Bldg. 503, Rm. 1355, Santa Barbara, CA 93117 USA

Thermal barrier coatings (TBCs) grown by electron-beam physical vapor deposition (EB-PVD) exhibit rather unique microstructures consisting of crystallographically aligned columnar grains, separated by long ribbonlike voids at the columnar boundaries and containing intracolumnar porosity at a much finer scale. The pattern and distribution of porosity influence the coating compliance, and hence its resistance to spallation, as well as the thermal conductivity, and hence the requisite thickness for a given degree of insulation. Crystallographic texture, in turn, bears on the column shape and, in principle, on the characteristics of the porosity. The present study aims to advance our understanding of the evolution of these microstructural features during growth. Deposition of 7%YSZ TBCs has been performed on stationary and rotating substrates at temperatures of ~900-1100°C. Results will be presented illustrating the effects of substrate temperature and vapor incidence angle on the crystallography of growth, and the content and distribution of inter- and intra-columnar porosity. Particular emphasis will be placed on the role of substrate rotation in microstructure evolution.

9:45 AM

DURABILITY, BOND STRENGTH AND BOND STRESS FOR FIVE PRODUCTION THERMAL BARRIER COATINGS: *Maurice Gell*[1]; Eric Jordan[1]; [1]University of Connecticut, Metall. and Mats. Eng., 97 North Eagleville Rd., U-136, Storrs, CT 06269-3136 USA

Bond strength and bond stress were determined for five production thermal barrier coatings as a function of furnace thermal cycling to 1121ϒC (2050ϒF). Of the five coatings, two were deposited by electron beam physical vapor deposition and three by air plasma spray. Bond coats include vacuum plasma sprayed MCrAlYs and a platinum aluminide. Bond strengths were measured as a function of thermal cycling using a modified ASTM direct pull test. The spallation failure mode in the direct pull test duplicated the failure mode in the thermal cycle test and in field service for each of the coatings. Bond stresses were determined in the thermally grown oxide as a function of thermal cycling using laser photostimulated luminescence. Changes in bond strength and stress will be related to localized compositional and microstructural changes, and to initiation and progression of interface debonding. The fracture lives of the five coatings will be compared and related to the initial and cyclic values of bond strength and stress.

10:05 AM BREAK

10:25 AM

THERMAL STABILITY OF AN EB-PVD THERMAL BARRIER COATING SYSTEM ON A SINGLE CRYSTAL NICKEL-BASE SUPERALLOY: U. Kaden[2]; *C. Leyens*[1]; M. Peters[2]; W. A. Kaysser[2]; [1]Oak Ridge National Laboratory, Corrosion Science and Technology Group/Metals and Ceramics Division, P.O. Box 2008, Bldg. 4500, M.S. 6156, Oak Ridge, TN 37831-6156 USA; [2]DLR-German Aerospace Center, Institute of Materials Research, Cologne Germany

Commercial thermal barrier coating (TBC) systems consist of a metallic bond coat deposited on a nickel-base superalloy substrate and a ceramic top coating. For optimal performance, the bond coat has to serve two purposes: 1) provide oxidation protection to the substrate alloy and 2) safely bond the ceramic top coating to the metal component. Both tasks are essentially performed by the alumina layer between the TBC and the bond coating, which is initially formed during coating processing and grows during service. In the present study, the interaction between the four components of a TBC system, substrate, bond coating, alumina scale and ceramic top coating will be discussed with respect to interdiffusion phenomena and their effects on the microstructure and chemical composition of the respective layers. The TBC system investigated consists of commercial single-crystal superalloy CMSX-4 (trademark of Cannon Muskegon), EB-PVD Ni-22Co-20Cr-12Al-0.1Y (wt.%) bond coating and EB-PVD 7wt.%YSZ TBC. Exposure to air at 1100 and 1200ϒC for up to 1000h revealed that considerable interdiffusion occurred between the substrate and the bond coating leading to precipitation of refractory-element rich plates and needles in the interdiffusion zone. Furthermore, diffusion of elements from the substrate through the bond coat into the alumina scale was observed to influence TBC adhesion.

10:45 AM

PHASE FORMATION AND CRYSTAL STRUCTURE IN REACTIVELY SPUTTER DEPOSITED ZIRCONIA AND YTTRIA STABILIZED ZIRCONIA (YSZ) COATINGS: Z. Ji[1]; *J. M. Rigsbee*[1]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., Birmingham, AL 35294-4461 USA

A series of zirconia-yttria (0 - 4.5 mol% Y_2O_3) coatings were produced by reactively sputtered elemental zirconium and yttrium targets in an argon and oxygen plasma. Phase formation and crystal structure as a function of substrate bias and Y_2O_3 content were investigated by x-ray diffraction (XRD) and transmission electron microscopy (TEM) techniques. The results showed that the crystal structure of pure zirconia coating changed from random equilibrium monoclinic, to random metastable tetragonal and finally strong (111) oriented tetragonal crystalline when the substrate bias was varied from 0 to -850 V. Furthermore, a highly (111) preferred orientation of tetragonal and cubic zirconia was found in 2.0 mol% and 4.5 mol% Y_2O_3 zirconia coatings, respectively, and each of these coatings was grown by sputtering with an applied substrate bias of -400 V. XRD and TEM analyses revealed that biased-sputtering could effectively decrease crystalline size in the as-deposited coating, which resulted in room temperature stabilization of the metastable tetragonal phase. XRD analysis of annealed coatings showed that the cubic phase was retained at temperatures up to 1200 ϒC. Conversely, transformation of the tetragonal to monoclinic phase occurred during annealing, with the fraction transformation being dependent on bias potential, annealing temperature and Y_2O_3 content.

11:05 AM

MECHANICAL PROPERTIES OF NANOSTRUCTURED ZIRCONIA MOCVD THERMAL BARRIER COATINGS*: *Ronald J. DiMelfi*[1]; Guido Soyez[2]; Jeffrey A. Eastman[2]; Loren J. Thompson[2]; John

M Kramer[1]; [1]Argonne National Laboratory, Engineering Research, RE-208, 9700 S. Cass Ave., Argonne, IL 60439-4803 USA; [2]Argonne National Laboratory, Mats. Sci., MSD-212, 9700 S. Cass Ave., Argonne, IL 60439-4803 USA

Nanocrystalline yttria stabilized zirconia (YSZ) coatings have been deposited on appropriate metallic substrates by metal organic chemical vapor deposition (MOCVD) processing. Nanoscale grain-size processing of these coatings is a promising method of enhancing their thermal resistance, and thereby rendering them more efficient thermal barrier coatings. However, it is important that such coatings be strong, tough and adherent to the substrate. Nanocrystalline ceramics can be both stronger and tougher than their conventionally grain-sized counterparts, and enhanced mass transport in these materials can improve bonding. Miniaturized disk bend testing is used to probe, in a single test, both the strength and adhesion of these coatings. This test is performed in a way that allows one to determine the stresses at which the coating fractures and at which delamination occurs. The results will be reported in relation to findings on the mechanical behavior of conventional YSZ thermal barrier coatings. *This work was performed under the auspices of the United States Department of Energy Technology Support Programs and Basic Energy Sciences, Division of Materials Science, under Contract No. W-31-109-ENG-38 and by a grant from Argonne's Coordinating Council for Science and Technology.

11:25 AM
SURFACE APPEARANCE CHARACTERIZATION OF THERMAL BARRIER COATINGS OF COMBUSTION COMPONENTS: *Javaid Qureshi*[1]; Robert Greenlaw[2]; [1]Westinghouse Electric Corporation, 4400 Alafaya Trail Quadrangle, Orlando, FL 32826-2399 USA; [2]Sermatech International, Sugar Land, TX USA

Combustion turbine (CT) components are coated with Yttria stabilized zirconia (YSZ) by plasma spray processes to increase component life. Plasma spray coating process variables are known to affect the coating quality and surface appearance of coated components. The typical surface quality and surface appearance of YSZ in the as-sprayed condition is antique white; however, minor variations in the spray conditions change the appearance from and antique white to a gray color. The gray surface appearance on coated CT components raises concerns regarding coating quality, thickness and service performance. To resolve these concerns, a comparative study of metallography, oxidation, X-ray diffraction, and quantitative oxygen analysis between specimens of differing surface appearance was conducted. Coating parameters were modified to produce specimens with varying surface appearances. A qualitative discussion of the parameter effect on surface appearance is presented. This analysis concluded that a minor variation in oxygen content and a phase variation in the top ceramic coating contributed to the gray color. No correlation between the surface appearance and the coating quality and thickness was observed. The coated components have been exposed to units and service performance is being evaluated.

11:45 AM
INVESTIGATION OF DAMAGE MECHANISMS IN THERMAL BARRIER COATINGS BY ACOUSTIC EMISSION: H. Echsler[1]; *M. Shutze*[1]; [1]Karl-Winnacker-Institut der DECHEMA e.V., Theodor-Heuss-Allee 25, Frankfurt, am Main 60486 Germany

The life-time of thermal barrier coating systems is determined by fracture processes in the region between bond coat and ceramic top coat which are strongly influenced by the oxidation processes going on at service temperature. In order to assess the quantitative effect of oxidation on the mechanical behavior of TBC systems an investigation technique has been developed which combines a modified 4-point-bending test at temperatures up to 1100ϒC with in-situ acoustic emission measurements. Mechanical data are measured by a load cell and a strain gauge yielding stress-strain data which are converted into data describing the load strain situation in the layered system with the help of finite element calculations. The load/strain situation is correlated with the acoustic emission data which allows the determination of critical levels for layer fracture (cracking, detachment, spallation, etc.). Analyzing the characteristic AE-data like energy, rise-time, signal duration, etc. yields information on the type of failure mechanism. The tests are performed after different preoxidation times taking into account the influence of bond coat oxidation. In the paper the technique will be discussed in detail and results from measurements will be exemplary.

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Packaging Technology and Reliability

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging & Interconnection Materials Committee
Program Organizers: Gautam Ghosh, Northwestern University, Dept. of Mats. Sci., Evanston, IL 60208-3108 USA; Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; Rao Mahidhara, Cypress Semiconductor Corporation San Jose, CA 95134 USA; Ephraim Suhir, Bell Labs., Murray Hill, NJ 07974 USA

Monday AM Room: 17A
March 1, 1999 Location: Convention Center

Session Chairs: G. Ghosh, Northwestern University, Dept. of Mats. Sci., Evanston, IL 60208-3108 USA; P. Vianco, Sandia National Laboratories, Albuquerque, NM USA

8:30 AM WELCOME AND OPENING REMARKS

8:35 AM INVITED PAPER
CURRENT METALLURGICAL ISSUES IN FLIP CHIP TECHNOLOGY: *K.-N. Tu*[1]; [1]UCLA, Dept. of Mats. Sci. and Eng., 405 Hilgard Ave., Los Angelos, CA 90095-1595 USA

When flip chip technology is applied to attaching a chip directly to a card or board, low melting point solders should be used because of the polymeric nature of the substrate. Low melting point solders are typically high-Sn, which are known to react rapidly with Cu, especially a Cu film. Hence the conventional under-bump thin film metallization of Au/Cu/Cr is unsuitable to card or board substrtates, not even the phased-in Cu-Cr metallization. Since the chip-to-card or chip-to-board packaging can be widely used in low cost and large volume consumer products, the electronic industry is exploring ways to replace the Cu-based under-bump metallization. While Ni reacts slower than a high Sn solder, Ni films are known to possess high stresses. In this talk, the issue of rapid reaction vs. high stress which challenges the flip chip technology will be addressed.

9:05 AM
DEVELOPMENT OF UNDER BUMP METALLIZATIONS FOR FLIP CHIP BONDING TO ORGANIC BOARDS: T. M. Korhonen[1]; P. Su[1]; S. J. Hong[1]; *M. A. Korhonen*[1]; C.-Y. Li[1]; [1]Cornell University, Dept. of Mats. Sci. and Eng., Ithaca, NY 14853 USA

In order to use flip chip bonding directly to organic boards, solders with relatively low reflow temperature must be used to avoid damage to the board during reflow. The most commonly used solders on the circuit board level (such as eutectic Pb-Sn) contain large amounts of tin, which makes them incompatible with the Cr/CrCu/Cu/Au underbump metallization (UBM) scheme used in conventional flip chip bonding. The tin in the solder reacts with the copper layer of the UBM, depleting the UBM of copper and causing a weak interface. UBM schemes with Ni as the wettable layer show slower reaction with the solder and have been identified by the semiconductor industry as preferable replacements to Cu-based UBM's. However, Ni-containing metallizations tend to have high stresses that may lead to peeling off of the metallization. In this research, the goal was to develop relatively low stress metallization schemes which contain sufficient amount of Ni to supress the growth of intermetallics. Several different metallization schemes were deposited on Si wafers and patterned into UBM pads, after which a reflow was performed to obtain flip chip bonded test joints. The joints were mechanically tested to assess the quality and reliability of the interface.

Stress was measured by the wafer bending technique from each of the metallized wafers before patterning the UBM pads, and the measured stresses were compared to the performance of the corresponding UBM scheme in the mechanical tests.

9:30 AM

RELIABILITY OF FLIP-CHIP PACKAGES THERMALLY LOADED BETWEEN -55 AND 125ϒC: *Elizabeth S. Drexler*[1]; [1]N.I.S.T., Div. 853, 325 Broadway, Boulder, CO 80303 USA

The low-temperature reliability of flip-chip packages has been source of concern for manufacturers of the innovative package. Packages that perform well and have excellent lifetime projections when thermally cycled from 20 to 120ϒC fail at an unacceptable rate when the temperature excursion is extended down to -55ϒC. Electron-beam (e-beam) moiré was used to study local deformations in a flip-chip package and the interactions among the various materials found within the package. A cross section through the solderballs of the flip-chip package was instrumented with crossed-line gratings with a pitch of 450 nm at the edge of the Si die, 1/4 of the way across the die, and at the mid-point of the die. As the specimen was thermally loaded, images of the moiré fringe patterns were acquired from each location and compared. The specimen was subjected to a total of ten complete thermal cycles from -55 to 125ϒC over several nonconsecutive days. Non-recoverable deformations were first observed in the specimen at the grating located at the mid-point of the die, but only after completing one full thermal cycle (that is, -55 to 125, then back down to -55ϒC). It appears that debonding initiated between the solderball and the solder mask where that interface meets the printed circuit board. The debond continued to grow through the solder mask and into the underfill during the next three thermal cycles, then arrested after circumscribing approximately 1/4 of the way around the solderball. Deformation was also induced within the solderball, becoming more pronounced with more thermal cycles. Some slip also occurred in the pattern located 1/4 of the way across the die at the solderball/solder mask interface, appearing after the second complete thermal cycle. After the ten cycles were completed, inspection of the region where the solderball/solder mask meets the printed circuit board revealed holes approximately 200 nm across. Void formation and coalescence, leading to crack initiation and growth seems to be a likely fracture mechanism. However, at this location the voids never coalesced to form a crack. No slip was observed in the pattern located at the edge of the die. Results will be further discussed, displacements quantified, and conclusions offered.

9:55 AM INVITED PAPER

NOVEL Cu-INTERCONNECT APPROACH FOR MULTI-CHIP MODULES WITH FLEXIBLE SUBSTRATES: *M. McCormack*[1]; H. Jiang[1]; S. Beilin[1]; [1]Fujitsu Computer Packaging Technologies, 3811 Zanker Rd., San Jose, CA 95134 USA

Modern electronic products including computers, telecommunication equipment, automobile electronics, and consumer electronics require circuit interconnections. Increases in circuit density have traditionally been desirable from the perspectives of product miniaturization and cost reduction. From a high-speed performance perspective, increases in circuit density must be accompanied by decreases in conductor lengths in order to minimize the effects of packaging parasitics. This is especially true in the case of multi-chip modules where shorter, less resistive conductive lengths that minimize parasitics are desirable for faster and more efficiently routed chip-to-chip communications. A new and novel approach to provide interconnections in multi-chip modules is presented and discussed in terms of the superior performance, ease of manufacture, and high reliability provided by the development of a unique combination of materials and processing.

10:25 AM BREAK

10:35 AM

A NOVEL TEST CIRCUIT FOR DETECTING ELECTROCHEMICAL MIGRATION: *W. Jud Ready*[1]; L. J. Turbini[1]; R. Nickel[2]; J. Fischer[2]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA; [2]Naval Air Warfare Center, Weapons Division, Code 471C00D, China Lake, CA 93555-6001 USA

The rapid growth of the global electronics manufacture environment has brought about the onset of a variety of new, untested materials and processing chemicals. The interactions between substrates and processing chemicals that can occur during manufacture, storage and use must be assessed in order to determine long-term reliability. Surface insulation resistance (SIR) testing is a standard industry technique used to assess processing chemicals (e.g., soldering fluxes) and substrates. SIR test method conditions vary in terms of the temperature and humidity used to accelerate the normal failure modes. Typically, a 45 to 50 volt bias is applied to an interdigitated comb pattern, and periodic SIR measurements are made using a 100 volt test. Pass/fail criteria (e.g., 100 M) based solely on SIR electrical values, however are inadequate. Often the electrical measurement fails to reveal the presence of surface dendrites due to contaminants related to processing chemicals. This occurs because the dendrite burns out between electrical readings when the circuit continues to be biased at 50 volts. Thus, the electrical reading does not recognize that the dendrite was present. A new linear test circuit has been developed to overcome this deficiency. The circuit uses an operational amplifier to detect the formation of a surface dendrite between electrodes on the comb pattern. When the dendrite shorts the circuit, voltage to the comb pattern is removed. Thus, the presence of the dendrite is captured electrically, and the dendrite is preserved for further analysis. This paper will present the circuit used and data showing its effectiveness.

11:00 AM INVITED PAPER

MODELING SHIFT OF A SOLDER-ALIGNED OPTICAL FIBER: *Adam Clayton Powell*[1]; Christopher Bailey[2]; Daniel Wheeler[2]; Mark W. Beranek[3]; James A. Warren[1]; [1]NIST, Metallurgy Div., MATLS B164, Gaithersburg, MD 20899 USA; [2]University of Greenwich, Dept. Comp. and Math. Sci., Wellington St., Woolwich, London SE18 6PF UK; [3]The Boeing Company, P.O. Box 3999, Mailstop 3W-51, Seattle, WA 98124-2499 USA

Optical fibers are often attached to electronic packages using solder. These fibers are observed to shift during solder wetting and solidification. In the present study, two finite element models are used to calculate the extent of this shift, in order to understand this phenomenon and make design changes to control fiber alignment. While in the liquid state, it is assumed that the solder surface quickly reaches its equilibrium shape, and the extent of shift is determined by the balance between capillary forces and elasticity of the fiber; this shape and shift are calculated by the Surface Evolver software. The surface shape is then used to create a volume mesh which is used in a transient model of heat transfer, solidification and solid mechanics based on the PHYSICA software package, to calculate the displacement of the fiber and the residual stress field in the solder. The direction of solder droplet solidification is observed to have a strong effect on final droplet shape and fiber displacement. Approaches to modeling fluid flow to feed solidification shrinkage are discussed. This modeling effort is supported by the NIST Solder Interconnect Design Team.

11:30 AM INVITED PAPER

MATERIALS REQUIREMENTS FOR CSPS: AN OVERVIEW: D. Speece[1]; *G. J. Ewell*[1]; [1]The Aerospace Corporation, M.S. 4-987, Los Angeles, CA 90009-2957 USA

The recent explosive increase both in development efforts and in testing related to chip scale packaging (CSP) has resulted in the identification of needs for improved packaging materials. Several companies and consortia are now working to create and produce the materials that will increase the reliability and robustness of chip scale packages as well as their ability to manage the large amount of thermal energy generated. The authors will present both an overview of the needs identified for package integrity and robustness, as well as for thermal management requirements, and partial results of a test program to characterize some of the materials available with respect to those needs. Test results include findings of thermal stability, short-term elevated temperature exposure, thermal cycling, and moisture resistance. These findings should directly interest both those people interested in package characterization as well as those interested in developing such materials.

12:00 PM
RELIABILITY OF SOLDER INTERCONNECTION IN BGA PACKAGE: *R. Mahidhara*[1]; W. Zohni[1]; V. Solberg[1]; J. Fjelstad[1]; T. DiStefano[1]; [1]Tessera, Inc., 3099 Orchard Dr., San Jose, CA 95134 USA

Devices furnished in plastic lead-frame type packaging are proving to be too large for newer generations of hand-held portable electronic products. Smaller size coupled with higher performance seems to be the requirement of the day. Using unpackaged or bare die may be the ultimate goal for companies attempting to reduce product size. However, when actual assembly process complexity (underfill) and poor yield for multiple die applications are considered, many may choose an alternative packaging methodology, chip-scale or even chip-size (CSP) packaging. CSP devices using a ball grid contact array is proving to be a technology that can provide the same benefit of bare die but not the headaches. In addition, an optional contact type for the µBGA unit is to combine solder attachment capability as well as socket capability by adapting a solid copper ball coated that is attached to the CSP with a high temperature solder. This will allow for socketing while still compatible with conventional SMT solder pa ste reflow mounting to the PCB. In this study, various lead-containing and lead-free solder systems are evaluated for attaching solid copper balls to the chip scale package. The reliability of the solder joints are then assessed and compared.

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Annealing Twins

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

Program Organizers: S. Ankem, University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742-2115 USA; Chandra Pande, Naval Research Lab, Mats. Sci. & Tech. Div., Washington, D.C. 20375-5000 USA

Monday AM — Room: 17B
March 1, 1999 — Location: Convention Center

Session Chairs: George R. Yoder, Office of Naval Research, ONR 332, Arlington, VA 22217-5660 USA; Chandra S. Pande, Naval Research Laboratory, Mats. Sci. and Tech. Div., Washington, D.C. 20375-5000 USA

8:30 AM OPENING REMARKS: Prof. S. Ankem, University of Maryland, College Park, MD, USA

8:35 AM INVITED PAPER
ANNEALING TWINS IN FCC METALS AND ALLOYS: *Bhakta B. Rath*[1]; Chandra S. Pande[1]; M. A. Imam[1]; [1]Naval Research Laboratory, MS & CT Directorate, Code 6000, Washington, D.C. 20375-5320 USA

Presence of annealing twins has impact on the properties of many materials. The mechanism of formation of these twins have been subject of numerous studies over fifty years. Although much progress has been made in recent years, a universally accepted view of these twins has not yet emerged. This brief review will focus on the nature of these twins, the mechanism of their formation and the methods to reduce their density. It will describe recent studies including those at Naval Research Laboratory to understand these features of annealing twins. These studies have established a relation between twin density and grain size, temperature and material properties. A model of the mechanism of their formation based on the emergence of Shockley partial loops on consecutive {111} planes during grain migration has also been developed. It is argued that various experimental and theoretical results obtained over the years can be satisfactorily explained by this model.

9:10 AM INVITED PAPER
A CONSTITUTIVE DESCRIPTION FOR THE INITIATION OF TWINNING: *Marc Andre Meyers*[1]; Otmar Voehringer[2]; Y. J. Chen[1]; [1]University of California, Dept. of Ames, Mail Code 0411, La Jolla, CA 92093 USA; [2]University of Karlsruhe (TH), I. für Werkstoffkunde1, Postfach 6980, 76128 Karlsruhe, Baden Germany

A constitutive equation is developed that predicts the critical stress for twinning as a function of external (temperature, strain rate) and internal (grain size, stacking- fault energy) parameters. Plastic deformation by slip and twinning being competitive mechanisms (it is, of course, recognized that twinning requires dislocation activity), the twinning constitutive relationship is equated to a slip relationship based on the flow by thermally assisted movement of dislocations over obstacles (such as the Voehringer, the Zerilli-Armstrong, or the MTS equations); this leads to the successful prediction of the slip-twinning transition. The model is applied to metals representative of the different crystalline structures: Fe, Cu, and Ti. As a consequence of the model, the critical twinning stress in shock-wave deformation can be predicted, using the Swegle-Grady equation which relates the shock stress to the strain rate at the shock front. Research supported by the Humboldt Foudation and U. S. Army Research Office MURI Program.

9:45 AM INVITED PAPER
EVOLUTION OF ANNEALING TWINS IN FCC CRYSTALS: *S. Mahajan*[1]; C. S. Pande[2]; M. A. Imam[2]; B. B. Rath[2]; [1]Arizona State University, Dept. of Chem., Bio and Mats. Eng., P.O. Box 876006, Tempe, AZ 85287-6006 USA; [2]Department of the Navy, Naval Research Laboratory, 4555 Overlook Ave., S.W., Washington, D.C. 20375-5343 USA

We have developed a microscopic model for the evolution of annealing twins in FCC crystals. We argue that twins evolve as a result of growth accidents occurring on migrating {111} steps associated with a moving grain boundary. The higher the velocity of the boundary, the higher the twin density. The influence of annealing temperature on twin density, the absence of twins in high stacking fault energy materials, and the various observed twin morphologies can be rationalized in terms of the model.

10:20 AM BREAK

10:30 AM INVITED PAPER
ROLE OF TWINNING IN THE OPTIMIZATION OF THE GRAIN BOUNDARY CHARACTER DISTRIBUTION: *Adam J. Schwartz*[1]; Wayne E. King[1]; Mukul Kumar[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci., 7000 East Ave., L-355, Livermore, CA 94550 USA

The grain boundary character distribution (GBCD) is a relatively new microstructural property that describes the proportions of special and random boundaries as defined by the coincident site lattice model. Recently, there has been increased attention on determination of the GBCD and manipulation of the relative fractions in the recrystallized microstructure through thermomechanical processing in order to improve materials properties like corrosion and creep resistance. Most of the "optimization" treatments reported in the literature have been performed on fcc materials with relatively low stacking fault energies and result in microstructures with high fractions of $\Sigma 3$, $\Sigma 9$, and $\Sigma 27$ boundaries. It can be interpreted that annealing twins are solely required to improve the GBCD. However, in order to optimize the properties, it appears imperative that the formation of annealing twins disrupt the connectivity of the random boundary network, thus implying that $\Sigma 3_n$ reactions and resultant triple lines are critical. Experiments to control the GBCD of oxygen free electronic Cu and Inconel 600 through thermomechanical processing will be presented and discussed in light of orientation imaging microscopy and transmission electron microscopy observations of the deformed and recrystallized microstructures. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

11:05 AM INVITED PAPER
INTERFACIAL DISLOCATION STRUCTURE AND DYNAMICS AT INCOHERENT TWIN BOUNDARIES: *Douglas L. Medlin*[1]; [1]Sandia

National Laboratories, Mats. and Eng. Sci. Center, Org. 8715 M.S. 9402, 7011 East Ave., Livermore, CA 94551 USA

Although the structures for incoherent twin boundaries in FCC metals are now well understood on the basis of experimental observations and atomistic modeling, there is still much to learn about the interactions and properties of interfacial dislocations at such interfaces. Two types of interfacial dislocations are anticipated and observed: namely, dislocations with Burgers vector a/6<211> and a/3<111>. The a/3<111> dislocation plays a particularly interesting role: because its Burgers vector lies in the (112) boundary plane, it is possible for the dislocation to move by glide at this interface. This concept is confirmed by experimental observations as well as by atomistic calculations showing only a small energetic barrier to translation of the dislocation. In addition to gliding, these dislocations play a role in coherent twin formation by a non-conservative climb process. Here we will discuss HRTEM observations showing the incorporation of a/3<111> dislocations into a growing coherent twin lamellae. Analysis of the tip of the advancing twin suggests that nucleation of the coherent twin segment may be initiated by the absorption of a lattice dislocation at the grain boundary. This research is supported by the U.S. Department of Energy, Office of Basic Energy Sciences, under contract number DE-AC04-94-AL85000.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: Applications of Gamma Titanium Aluminide Alloys

Sponsored by: Structural Materials Division, Titanium Committee, Structural Materials Committee; ASM International: Materials Science Critical Technology Sector, Materials Synthesis & Processing Committee

Program Organizers: Young-Won Kim, UES, Inc., Mats. & Proc. Division, Dayton, OH 45432-1805 USA; Dennis M. Dimiduk, Wright-Patterson AFB, WL/MD, WPAFB, OH 45433 USA; Michael H. Loretto, University of Birmingham, IRC, Birmingham B15 2TT UK

Monday AM Room: 8
March 1, 1999 Location: Convention Center

Session Chairs: Young-Won Kim, UES, Mats. and Proc. Div., Dayton, OH 45432 USA; Howard Merrick, Allied Signal Engines, P.O. Box 52181, Phoenix AZ 85072-2181 USA

8:30 AM OPENING REMARKS

8:40 AM INVITED PAPER

ADVANCED WROUGHT GAMMA ROTOR PROGRAM - SUBSCALE TiAl ROTOR RESULTS: *Theodore Fecke*[2]; Dwight E. Davidson[1]; [1]Pratt & Whitney Aircraft Engines, Advanced Technology Engine Programs, P.O. Box 19600, West Palm Beach, FL 33410-9600 USA; [2]Air Force Research Laboratory, AFRL/PRTC, 1950 Fifth St., Wright-Patterson AFB, OH 45433-7251 USA

With increased demands from the commercial and military sectors for longer range, greater endurance and improved durability aircraft, thus requiring more efficient turbine engines, Pratt and Whitney has embarked on an initiative to increase overall turbine engine systems performance. One of the technologies selected involves the use of gamma titanium aluminide as a rotating disk structure. In order to achieve these improvements, increases in engine system temperatures and pressure, and decreases in weight must be simultaneously accomplished. Most turbine engine rotor structures today are nickel based. An Advanced Gamma Titanium Rotor Program, whose goal is to demonstrate the rules, tools and design intent for transitioning new materials (i.e. via characterization, component test and life system validation), will help to accomplish these increased airframe mission requirements. The "overall" gamma titanium rotor program goals are to demonstrate that an "all titanium" rotor component can survive harsh engine environments. These environments can be simulated via structural analysis and component spin test. Prior to engine testing, many criteria have to be met, similar to those criteria required for a nickel based rotor structure. They include TiAl material capability, mission capability, rotor dynamic behavior, impact evaluation, blade out behavior, surge/stall capability, alloy enhancements, processing, manufacturing, and spin testing. This presentation will highlight pertinent data, thus providing technical insight eluding to an "all titanium high spool core". Where applicable, spin test, crack growth or other component or specimen results will be provided. The final outcome of several cyclic spin tests will not be available until after May 1999.

9:10 AM INVITED PAPER

TITANIUM ALUMINIDE APPLICATIONS IN THE HIGH SPEED CIVIL TRANSPORT (HSCT): *Paul A. Bartolotta*[1]; David L. Krause[1]; [1]NASA Lewis Research Center, 21000 Brookpark Rd., MS 51-1, Cleveland, OH 44135 USA

The High Speed Civil Transport (HSCT) is a second-generation supersonic commercial aircraft for the next century. It is projected that within the next two decades, overseas air travel will increase to over 600,000 passengers per day. This equates to 500 -1500 HSCT type of aircraft will be required to meet this demand. In order to meet EPA environmental goals, the HSCT propulsion system will require advanced technologies to reduce exhaust and noise pollution. Part of the strategy for noise attenuation is the use of an extremely large exhaust nozzle. Critical exhaust nozzle components will be fabricated from titanium aluminide in two different forms. The divergent flap will use wrought gamma and the nozzle sidewall will be a hybrid fabricated out of both wrought gamma face sheet and cast gamma substructure. This paper will describe the HSCT program and the use of titanium aluminide for its components.

9:40 AM INVITED PAPER

IMPLEMENTATION OF GAMMA TITANIUM ALUMINIDES: *Curtiss M. Austin*[1]; Thomas J. Kelly[1]; [1]GE Aircraft Engines, MPED, M89, One Neumann Way, Cincinnati, OH 45215 USA

The gamma titanium aluminide community has made considerable progress in the lengthy and arduous process of bringing a technology from infancy to a state of near-readiness. An ingot process has been devised to meet the special requirements of the casting approach. Chemistry methods have been developed that can confirm that aluminum level is within range. Casting processes have been identified that can make sound parts of a difficult configuration - namely low pressure turbine blades. Many details of the associated manufacturing processes have been developed as well. Some of the first-tier design methods and issues have resolved. The most serious performance issues have either 1) been retired (tip rubs, for example), 2) become the subject of a few community-wide investigations (impact resistance, for example), or 3) been resolved as much as possible short of engine test (wear, for example). Further progress relies on overcoming two hurdles. First, hardware must be produced by a production-ready process and then engine-tested in a manner that directly addresses the remaining performance issues. Second, the economics of implementation must be assessed, comparing hardware price forecasts with the value of the technology as manifest in the engine sales market.

10:10 AM INVITED PAPER

GAMMA TiAl: CONSIDERABLE POTENTIAL - BUT NOT YET FLYING: Wayne Voice[1]; David Rugg[1]; [1]Rolls-Royce Plc, Dept. of Mats., Elton Rd., (Elt38), P.O. Box 31, Derby, Derbyshire DE24 8BJ UK

Gamma Titanium Aluminides have shown 'considerable potential' and 'attractive properties' for many years. Despite numerous tantalising aero-engine applications having been seriously considered, no gamma parts are in service. This paper reviews an aerospace materials 'life cycle' in order to explain the apparent discrepancy. Conventional titanium alloys/components will be used to provide a baseline for comparison.

10:40 AM

ENDURANCE OF TiAl ALLOY AS A TURBINE WHEEL OF TURBOCHARGER AND THE EFFECT OF COMPOSITION ON IT: *Toshimitsu Tetsui*[1]; [1]Mitsubishi Heavy Industries, Nagasaki R&D, 5-717-1 Fukahori-Machi, Nagasaki 851-0392 Japan

Turbochargers for passenger cars using two kind of as cast TiAl alloys (low Nb and high Nb) for their turbine wheels were manufactured and attached to a 2.5-liter Diesel engine for engine testing at 1123K up to 600h. Damage to the TiAl turbine wheels after the endurance test was investigated in order to evaluate TiAl endurance in an actual operating environment and to examine the effects of chemical composition and microstructure on it. Although oxidation was milder in engine exhaust gas than in the air, showing satisfactory endurance for both alloys, erosion of blade tips was found only in low Nb alloy. This erosion is estimated to have been caused by the collision of fine particles, and it was found that fully lamellar structure of very fine colony size which was formed in blade tips of a wheel of high Nb alloy showed superior resistance to this erosion.

11:00 AM

GAMMA-BASED TITANIUM ALUMINIDE STRUCTURES FOR SOME SPECIFIC HIGH TEMPERATURE AEROSPACE APPLICATIONS: *Robert LeHolm*[1]; Helmut Clemens[2]; [1]BF Goodrich Aerospace, Aerostructures Group, 850 Lagoon Dr., Chula Vista, CA 91910 USA; [2]University of Stuttgart, Institut fuer Metallkunde, Seestrasse 71 D-70174 Germany

Gamma titanium aluminides (g-TiAl's) are a group of very potentially promising, low density intermetallic materials that offer many attractive properties for various high temperature aerospace applications, in both aircraft and spacecraft structures. This paper presents some of the work performed to characterize, test and qualify thin g-TiAl sheet for commercial use as a replacement for superalloy aerospace structures, in the 650ϒC - 900ϒC range. In addition, some mention of g-TiAl foil products and their development will be included. Any potential g-TiAl component must be economically manufacturable and must survive severe turbine exhaust gas and/or space re-entry conditions. An extensive test and development program was pursued to: (a) determine the high temperature behavior (up to 900ϒC) of thin g-TiAl sheet and (b) establish key producibility parameters (for forming, bonding, machining, etc.) and a proof of concept for manufacturing a g-TiAl prototype component. Based on the results of this test and development program, g-TiAl sheet structures may prove to be viable, both economically and structurally, for some specific high temperature aerospace applications.

11:20 AM

PROPERTIES OF LOW COST TiAl AUTOMOTIVE VALVES PRODUCED BY COLD WALL INDUCTION MELTING AND PERMANENT MOLD CENTRIFUGAL CASTING: *Matthias Blum*[1]; Alok Choudhury[1]; Harald Scholz[1]; Georg Jarczyk[1]; Georg Frommeyer[2]; Peter Busse[3]; Sven Knippscheer[2]; [1]ALD Vacuum Technologies GmbH, R&D, Rueckinger Str. 12, Erlensee, Hessen 63526 Germany; [2]MPI fuer Eisenforschung, Max-Planck-Str. 1, Duesseldorf 40237 Germany; [3]ACCESS e.V., Intzestrasse 5, Aachen 52056 Germany

Initiated by ALD Vacuum Technologies GmbH a new manufacturing process for an economical mass production of TiAl valves has been developed by a joint research project which is financially supported of the Federal Ministry for Education and Research of Germany. The new process enables the production of TiAl valves in high annual volumes and at cost comparable to conventional steel exhaust valves. The expected price is feasible through the very high level of process integration. Melting, alloying, purification and casting are integrated in a single step. The two main features of the manufacturing process are the use of a modified induction cold crucible and a heatable metallic permanent mold in an evacuable centrifugal casting unit. Based on the results of numerical process simulation as well as casting experiments a pilot plant has been built. In order to minimize the effort for the optimization of melting and casting parameters, regarding the mechanical properties of the valve, an experimental programme based on modern DoE- technique was performed. The development of the process and the results of this programme will be presented.

11:40 AM

POSTWELD HEAT TREATMENT EFFECTS ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ELECTRON BEAM WELDED CAST GAMMA TITANIUM ALUMINIDES: Chris M. Jensen[4]; *Han Zhang*[1]; William A. Baeslack[2]; Tom J. Kelly[3]; [1]The Ohio State University, 1248 Arthur E. Adams Dr., Columbus, OH 43221 USA; [2]The Ohio State Edison Joining Technology Center University, Bricker Hall, 190 North Oval Mall, Columbus, OH 43210 USA; [3]GE Aircraft Engines, One Neumann Way MD M89, Cincinnati, OH 45215 USA; [4]Homwet, 1500 South Warner St., Whitehall, MI 49461 USA

The cast Ti-48Al-2Nb-2Cr Electron beam (EB) welds were produced with aluminum contents from 46.3 to 48.3 at.%. Postweld heat-treated (PWHT'ed) were performed at temperatures of 1050, 1150 and 1250ϒC. It was found that the EB welds experienced dendritic solidification for all of the Al contents investigated. A transition from primary b solidification in the plates containing 46.3 and 47.1at% Al to a b/a solidification in the plates containing 47.1 at% Al and higher were observed at the fusion zone (FZ). The welds exhibited a predominantly lamellar g/a2 FZ microstructure in the as-welded condition, with increasing proportions of equiaxed g grains, and a decreasing volume proportion of intergranular a2 phase, observed with increasing aluminum content. PWHT promoted recrystallization and growth of the g grains. FZ fracture toughness decreased with increasing aluminum content, and increasing PWHT temperature. FZ ductility was improved by PWHT.

12:00 PM

THE EFFECT OF IRON CONTENT ON THE WELDABILITY OF CAST GAMMA TITANIUM ALUMINIDES: *Ponnusamy Anand*[1]; Viola L. Acoff[1]; [1]The University of Alabama, Metall. & Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

In the majority of engineering applications for which gamma TiAl is being considered, fusion welding, in particular gas tungsten arc welding, is the primary form of joining. Current-generation gamma alloys, notably Ti-48Al-2Cr-2Nb meet the requirements of many components. Presently, the composition of iron is kept to the least value possible (0.07 atomic %) because it is suspected that Fe has a detrimental effect on the weldability of cast gamma TiAl. However, this practice increases the cost of processing the alloy. To date, the effect of iron content on the weldability of gamma TiAl has not been determined or investigated experimentally. The purpose of this study was to investigate the effect of iron on the weldability of cast gamma TiAl and its effect on the kinetics of gamma transformation during heating. The structure-property relationship of the fusion zone were examined as a function of welding current and characterized using light microscopy, SEM, TEM, Knoop microhardness testing and nanoindentation testing.

LEACHING THEORY PROCESS DEVELOPMENT & INDUSTRIAL PRACTICE: Leaching General

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee, Copper, Nickel, Cobalt Committee

Program Organizers: Akram Alfantazi, Falconbridge Ltd., Falconbridge Technology Centre, Falconbridge, Ontario P0M 1S0 Canada; Arash Kasaaian, Elkem Metals Company, Marietta, OH 45750 USA; Alexandre J. Monteiro, Indosuez Capital Emerging Markets, Sao Paulo, SP 01311-902 Brazil

Monday AM Room: 1B
March 1, 1999 Location: Convention Center

Session Chairs: D. Ashman, Cominco, Ltd.; D. E. Krause, INCO, Ltd.

8:30 AM

MAGNESIA HYDRATION IN MAGNESIUM ACETATE SOLUTIONS: Dimitrios Filippou[1]; Nikolaos Katiforis[1]; *Nymphodora Papassiopi*[1]; Katerina Adam[1]; [1]National Technical University of Athens, Dept. of Mining Eng. and Metall., Laboratory of Metall., Zographos, Attiki GR-157 80 Greece

Magnesium hydroxide is used in a number of industrial applications, from the neutralisation of acid effluents to the production of pharmaceuticals. High-quality magnesium hydroxide powders can be produced by hydrating slow-reacting magnesia in dilute magnesium acetate solutions. The magnesia hydration process shows many similarities with a typical hydrometallurgical leaching operation; however, its kinetics are crucial not only for process design and control, but also for the production of powders with a desirable particle morphology. This presentation will show the results of an experimental work whereby industrial heavily-burned magnesia powders were hydrated in 0.01 to 0.1 mol/L magnesium acetate solutions at temperatures ranging between 333 and 363 K. Examination of the magnesium hydroxide produced and the analysis of the kinetic data suggest that the hydration of heavily-burned magnesia in magnesium acetate solutions is a dissolution—precipitation process controlled by the dissolution of magnesia particles. The activation energy was estimated to be 59.7 kJ/mol, while the reaction order with respect to magnesium acetate concentration was found to be about one.

8:50 AM

OPERATION OF AND IMPROVEMENTS TO THE LONRHO PLATINUM BASE METAL REFINERY: [1]*Nico Steenekamp,* Lonrho Platinum Base Metal Refinery, South Africa

The Lonrho Platinum Base Metal Refinery was commissioned in 1985. The original process design was done by Sherrit Gordon. Ni- Cu converter matte is treated in a refinery which utilises both atmospheric and pressure leaches for the dissolution of base metals to produce $NiSO_4$crystals copper cathode and a Pgm concentrate. The paper will cover the original and current flow sheets, the 14 years of operation, increases in capacity and the improvements implemented during this period. The improvements include modifications to the autoclave operations to implement a flash recycle cooling arrangement and a continuous upgrading leach in the same autoclave. The practice and changes in the first stage atmospheric leach and the batch upgrading leaches of PGM concentrate will be discussed.

9:10 AM

NITRIC ACID IN HYDROMETALLURGY: *Fathi Habashi*[1]; [1]Laval University, Dept. of Mining and Metall., Cite Universitaire, Quebec City G1K7P4 Canada

Nitric Acid has been used for over 50 years in the uranium industry and in phosphate fertilizer manufacture. Its application in the treatment of sulfide concentrates is relatively recent and is limited to one plant. It is necessary to capture nitric oxide formed during leaching to economise the acid Consumption. A comparison with oxygen as an oxidizing agent is given.

9:30 AM

MODELLING OF HEAP-LEACHING FOR LOW GRADE LATERITE ORES: *P. A. Lobarinhas*[1]; J.C. F. Teixaira[1]; F. Castro[1]; [1]Universidade do Minho, Escola de Engenharia, 4800 Guimardes Portugal

Surphuric acid heap leaching of low-grade nickaliferous latrites became an increasingly interesting technology for the recovery of these metals from such ore reserves. In the present work the transient leaching process is modeled as a porous media flow with chemical reaction which will depend upon the contact area between the ore and the liquid solution, the solution concentration and the flow pattern inside the pile. The flow in porous media (such as that in soils) is described by the Darcy's law, which on a Cartesian two-dimensional takes the form for the hydraulic gradients, where the soil parameters are estimated as a function of the grain size assuming a compact packing and a random distribution through the ore. This equation is discretized on a regular orthogonal grid using a fully implicit ceil vertex control volume technique. As boundary conditions are concerned, it is assumed there is no liquid solution accumulation at the side and bottom boundaries (ah/ax-o). At the top boundary the liquid solution is fed into the pile and, therefore, ah/a=constant, a function of the volumetric flow rate of acid solution.. Adding fresh acid to the process (or changing its concentration) is taken into accounts in the model. Once the solution to equation (1) is obtained, the flow through the physical domain can be calculated. From this, the amount of metal extracted is determined using a simple chemical model for the dissolution kinetics for each metal present in each one of the phases of the ore. Experimental data, obtained by column leaching experiments at the laboratory, have been employed to validate the model. Good agreement was observed.

9:50 AM

LEACHING FLY ASH TO RECOVER METAL VALUES: *George Kazonich*[1]; [1]U.S. Department of Energy, Federal Energy Technology Center, P.O. Box 10940, Environmental Science and Technology Division, Pittsburgh, PA 15236 USA

More than 100 million tons of coal combustion by-products are generated by U.S. power plants each year. Fly ash makes up 60% of that total and typically contains 90% inert particles. The remaining 6 million tons/yr of metals and metal oxides could be recovered by leaching. Valuable metals include calcium, copper, lead, manganese, nickel, zinc, and others. The toxic metals antimony, arsenic, barium, cadmium, mercury, selenium, etc. are also found in trace amounts. Most of the metals are easily leached from the fly ash. The DOE is investigating the leaching of fly ash in 5-cm by 1-meter columns. Seven common lixiviants from pH 11 to pH 11 were used. Each column is leached with 150m1/day of one lixiviant for 30 to 120 days. The leachate is analyzed for metals, sulfate, pH, alkalinity, and conductivity. Tests have been completed on 28 fly ash samples. Metal recoveries varied from 1O to 95% in the tests examined. Extraction could be improved in most cases.

10:10 AM

WET OR DRY, INTEMPERANCE IN THE MINERAL FIELDS: INTOXICATION AND PROHIBITION REGARDING HYDROMETALLURGICAL PROCESSES AND THE EXERCISE OF LOCAL OPTIONS: *Larry M. Southwick*[1]; [1]L. M. Southwick & Associates, Process Design, Extractive Metall. and Chem. Eng., 992 Marion Ave., Suite 306, Cincinnati, OH 45229 USA

Smelters are doomed! The smelter as it exists today will be an institution of only historic interest in a few years! These cries have been heard off and on the 30 last years regarding the "inescapable benefits" of hydrometallurgical processes when compared to pyrometallurgical options. What is perhaps not well known is that these cries were also heard 75 to 100 years ago. Costs were a major driven force then, although environmental issues were also raised. What was said then is still true now, "Patient offices are cluttered with schemes that are theoretically sound. But when duplicating the work in 100-ton units, difficulties arise. Delicate reactions that can be controlled to a nicety in the laboratory are not so docile in the large plant. Caustic or add solutions, often times heated, that can be handled without any thought in glass receptacles, go on a rampage when the containers are wood and iron. This paper will review developments and advances in the hydrometallurgical field in the time frame 1890-1930, looking at dump and heap leaching, vat leaching, recovery methods and choice of lixiviant (including gas-house liquors). It was finally realized that local conditions prevailed, no fixed process could be applied to all cases. Lessons learned then reward review and study in the present day. The success or failure of several examples in the copper, lead and zinc fields will be discussed. It will be seen that then, as now, while considerable progress was made with wet methods, the portents of doom for dry methods were bit premature. Temperance and intemperance found it necessary to coexist.

10:30 AM

RECYCLING WASHER WATER IN THE KROLL TITANIUM LEACHING PROCESS: *Samuel A. Davis*[1]; Delton R Lyon[1]; Jim A. Owens[2]; Daphne L. Sprayberry[1]; Hal S. Osborne[1]; [1]TIMET, K-52, Henderson, NV 89009 USA; [2]IFC Kaiser, Dept. of Eng., Gateway View Plaza, 1600 W. Carson St., Pittsburgh, PA 15219 USA

In July of 1996, the Kroll process began production again at TIMET's Henderson, NV, manufacturing facility. This, after being shut down for several years due to low market demand for titanium. Faced with limited pond capacity and increasing costs for water and wastewater disposal and storage; process changes were made to recycle water used to wash Kroll titanium sponge back into the sponge leaching process. This idea was

revisited from 1992-93, when three consecutive attempts to recycle water for this application were unsuccessful due to early pump failure. Since it was uncertain why previous attempts failed, a complete understanding of the existing system was first sought. This included reviewing piping and flow diagrams; taking field measurements for all the piping, valves, and fittings; analyzing the wastewater composition; and modeling the fluid dynamics and control of the system. The fluid dynamics of the system were modeled by breaking the piping network up into nodes and applying the Bernoulli equation to each segment. The fluid flowrate control valves were modeled for this purpose also and the models were used to help specify the required processing equipment and control ranges and tolerances. Once the project was completed, an operating procedure was prepared for operation, startup, and shutdown of the washer water recycle system.

LIGHT WEIGHT ALLOYS FOR AEROSPACE APPLICATIONS V: Aluminum-Lithium Alloys

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Eui W. Lee, Naval Air Warfare Center, Code 4342, MS5, Patuxent River, MD 20670 USA; William Frazier, Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670-1908 USA; K. Jata, Wright-Patterson Air Force Base, WL-MLS, Dayton, OH 45433-7718 USA; Nack J. Kim, Center for Adv. Aerospace Mats., Pohang 790-330 Korea

Monday AM Room: 9
March 1, 1999 Location: Convention Center

Session Chair: Omar Es-Said, Loyola Marymount College, Dept. of Mats., Los Angeles, CA 90045-8145 USA

8:30 AM

FRICTION STIR WELDED JOINTS OF AN ALUMINUM-LITHIUM ALLOY: *Kumar V. Jata*[1]; [1]Air Force Research Laboratory, 2230 Tenth St., WPAFB, OH 45433 USA

An Al-Li-Cu alloy was friction stir welded in T3 and T8 tempers to understand the effect of joining on grain structure, precipitation and mechanical properties. Metallographic analysis shows a recrystallized microstructure in the entire weld region and TEM revealed the weld nugget to be in a solution heat treated condition with some faint ed (delta) reflections. On either side of the weld nugget T1 precipitates are observed. Aging treatments without solution heat treatment suggest that although the hardness and strength of the weld can be improved (or recovered) the ductility is not regained. This paper will discuss the observed microstructure-property relationships with particular emphasis on the ability to develop property combinations required for structural applications.

9:00 AM

DETERMINING THE EFFECT OF MICROSTRUCTURE AND HEAT TREATMENT ON THE MECHANICAL STRENGTHENING BEHAVIOR OF AN ALUMINUM ALLOY CONTAINING LITHIUM PRECIPITATION HARDENED WITH THE Al_3Li INTERMETALLIC PHASE: *James M. Fragomeni*[1]; [1]Ohio University, Mech. Eng., 251 Stocker Center, Athens, OH 45701 USA

The effect of the thermal treatment on microstructure and subsequent mechanical behavior of an Al-2.6wt.%Li-0.09wt.%Zr alloy that was solution heat treated and artificially aged for a series of aging times and temperatures was studied. The underaged, peak-aged, and overaged thermal heat treatments were studied to determine the effect of the microstructure on the properties. A model was subsequently developed for predicting the precipitation hardening response of a particle strengthened alloy determined from the microstructure, composition, and heat treatment. The precipitates in the microstructure which impede dislocation motion and control the precipitation strengthening response as a function of aging practice were used as the basis for determining the strength depending on the actual size distribution of the particles. The average particle size and size distribution were determined from the microstructure via. the heat treatment and composition. Consequently, a micromechanical model was developed for predicting the variation in yield strength with aging time, aging temperature, and composition. The overall micromechanical model which was determined from the particle coarsening kinetics, dislocation mechanics, thermodynamics, resolved shear stress, as well as the dislocation particle shearing and bypassing mechanisms was used to predict the mechanical strength of the alloy. For this alloy, the primary strengthening is a consequence of ordered coherent d? (Al3Li) intermetallic face centered cubic particles which are uniformly distributed throughout the microstructure. The d? (Al3Li) particle size, distribution, spacing, and volume fraction are closely related to the mechanical behavior and a direct result of the heat treatment and composition.

9:30 AM

EFFECT OF SLIP CHARACTERISTICS ON NEAR THRESHOLD FATIGUE CRACK PROPAGATION BEHAVIOR OF Al-Li ALLOYS: *C. H. Yang*[1]; K S Shin[1]; [1]Seoul National University, School. of Mats. Sci. and Eng., Center for Advanced Aerospace Materials , San 56-1, Shinrim-dong, Seoul 151-742 Korea

The effects of R ratio on near threshold fatigue crack propagation behavior of T8 and T6-like heat treated 8090 Al-Li alloys were examined in air and in vacuum. As the R ratio increased delta K th, maintained the constant values in each test environment. For the quantitative analysis, all test data including Kel, th were compared with the calculated results from a newly proposed superposition model and the results showed good agreements. From the analysis it was found that the high crack closure level of 8090 alloy in air was caused by the enhanced contribution of roughness-induced crack closure, and the low crack closure level in vacuum despite rougher crack surface was caused by the lack of mode II displacement.

10:00 AM

PREDICTION OF ANISOTROPY IN PRECIPITATION HARDENING 2090 DURING OFF AXIS STRETCHING USING STATISTICAL MECHANICS ANALYSIS: *H. Garmestani*[1]; [1]FAMU-FSU, College of Eng., Dept. of Mech. Eng., Tallahassee, FL 32317 USA

A statistical mechanics crystal plasticity model is used to predict the effect of off-axis stretching on the mechanical properties of 2095. The inhomogeneous distribution of T1 precipitates in Al-Li alloys results in an additional anisotropy once the material is deformed in specific orientations. This paper discusses the use of a continuum statistical theory to predict the evolution of the precipitation hardened material with an inhomogeneous distribution of T1 precipitates. The distribution and morphology of the microstructure is represented by two and three point probability functions. A two Green's function solution to the equations of stress equilibrium originally proposed by Molinari etal is utilized to obtain the constitutive law for the hetrogeneous medium. The concepts of statistical continuum theory are introduced into the localization relation to obtain a closed form solution. The effective properties are then calculated using the result of the initial texture. Simulation results are presented for several states of stress stretching.

10:30 AM

TIME-TEMPERATURE-PRECIPITATION BEHAVIOR IN AN Al-Cu-Li ALLOY 2195 : *P. S. Chen*[1]; B N Bhat[2]; [1]IIT Research Institute, Metallurgy Research Facilities, Bldg. 4628, NASA, Marshall Space Flight Center, AL 35812 USA; [2]NASA, Marshall Space Flight Center, Mats. and Process Laboratory, MSFC, AL 35812 USA

Al-Cu-Li alloy 2195, with its combination of good cryogenic properties, low density, and high modulus, has been selected by NASA to be the main structural alloy of the Super Light Weight Tank (SLWT) for the Space Shuttle. Alloy 2195 is strengthened by an aging treatment that precipitates a particular precipitate, labeled as T1 (Al2CuLi). Other phases, such as GP zone, q*, q*, q, d*, S* are also present in this alloy. Motivation for the TTP study at lower temperature (lower than 350

*F) came from a recent finding that the cryogenic fracture toughness of alloy 2195 is greatly influenced by the phases present in the matrix and subgrain boundaries. The study of TTP behavior at higher temperature (400 to 1000°F) was prompted by the fact that the SLWT requires a welded construction. As a result, considerable changes in HAZ microstructure and mechanical properties are expected during the construction of the SLWT. Therefore, TTP diagrams can help develop a guideline to select appropriate heat treatment conditions for the desirable applications and serve to understand the thermal history of the alloy by analyzing the welded microstructure. This paper will unveil the TTP diagrams and detail the nucleation and growth behavior of various precipitates in Al-Cu-Li alloy 2195.

MICROMECHANICS AND MICROMECHANISMS OF DEFORMATION AND FRACTURE: A SYMPOSIUM IN HONOR OF PROFESSOR ALI S. ARGON: Session I

Sponsored by: Structural Materials Division, Mechanical Metallurgy Committee, High Temperature Alloys Committee

Program Organizers: K. Jimmy Hsia, University of Illinois, Dept. of Theoretical & Appl. Mech, Urbana, IL 61801 USA; Mary Boyce, Massachusetts Institute of Technology, Dept. of Mech. Eng., Cambridge, MA 02139 USA; Tresa M. Pollock, Carnegie Mellon University, Dept. of Metall. Eng. & Mat. Sci., Pittsburgh, PA 15213 USA

Monday AM Room: 14B
March 1, 1999 Location: Convention Center

Session Chairs: David M Parks, MIT, Dept. of Mech. Eng. Cambridge, MA 02139 USA; William D. Nix, Stanford University, Dept. of Mats. Sci. & Eng., Stanford, CA 94305-2205 USA

8:30 AM OPENING REMARKS

8:35 AM INVITED PAPER

MODELING PLASTIC DEFORMATION AND STRAIN HARDENING IN THIN METAL FILMS ON SUBSTRATES: *William D. Nix*[1]; Omar S. Leung[1]; [1]Stanford University, Dept. Mat. Sci. & Eng., Peterson Bldg., Stanford, CA 94305-2205 USA

The problem of dislocation motion in a thin, single crystal slab bounded on one or both sides by materials that are either unstrained or non-shearable, is discussed, with particular reference to plastic deformation and strain hardening of thin metal films on substrates. A simple model in which a pure edge dislocation is imagined to propagate in a film leaving a pure screw dislocation in its wake, at the film/substrate interface, is used to describe thin film plasticity. The method of images is used to determine the energy of the "misfit" dislocation left in the wake of the moving dislocation and this leads to an estimate of the strength of the film, through a work argument. This simple geometry permits a study of the effects of elastic rigidity of the substrate, and any passivating film or underlayer that might be present, on the strength of the metal film, again using the method of images. The method of images allows exact expressions for film strength to be developed in some limiting cases. Strain hardening is described by considering the elastic interactions of moving dislocations with orthogonal screw dislocation obstacles already present at the film/substrate interface, using the method of images to estimate the energy of the misfit dislocation segment left near the obstacle in the passing process. Very high rates of strain hardening, consistent with experiment, are predicted. The strong Bauschinger effect observed during thermal cycling of thin metal films on substrates is also described and discussed in terms of the screw dislocation misfit model. Recent experiments on plasticity of gold films on silicon substrates, with and without passivation, using both substrate curvature experiments and bulge testing techniques, will also be described. Predictions of the modeling will be compared with experiment.

9:05 AM

SHEAR LOCALIZATION IN MATERIALS: MICROSTRUCTURAL EVOLUTION AND SELF-ORGANIZATION: *Marc Andre Meyers*[1]; Vitaly F. Nesterenko[1]; Qing Xue[1]; T. W. Wright[2]; C. J. Shih[3]; [1]University of California, San Diego, Dept. of AMES, Mail Code 0411, La Jolla, CA 92093 USA; [2]Army Research Laboratory, Aberdeen Proving Ground, Aberdeen, MD USA; [3]Ceradyne, Inc., 3169 Redhill Ave., Costa Mesa, CA 92626 USA

The seminal contributions of Prof. Argon in the plastic deformation of crystalline and non-crystalline solids will be reviewed, with emphasis on strain-rate effects. Shear localization has been found to be an important and sometimes dominant deformation and fracture mode in metals, fractured and granular ceramics, polymers, and metallic glasses at high strains and strain rates. Experiments involving the collapse of a thick - walled cylinder enable controlled and reproducible application of plastic deformation at very high strain rates to specimens. The initiation and propagation of shear bands has been studied in metals (Ti, Ta,Ti-6Al-4 V, and stainless steel), granular and prefractured SiC, a polymer (teflon) and a metallic glasses. For all materials, shear bands exhibit a clear self-organization, with a characteristic spacing that is function of a number of parameters. This self-organization is analyzed in terms of fundamental material parameters in the frame of Wright-Ockendon, Grady-Kipp and Molinari models. Another aspect that was investigated is the microstructural evolution inside the shear bands. A fine recrystallized structure is observed in Ti and Ta, and it is becoming clear that a recrystallization mechanism is operating. The short deformation and cooling times inhibit grain- boundary migration, and suggest a rotational mechanism, that is presented in terms of dislocation energetics. For the granular SiC, a novel mechanism of shear-induced bonding was experimentally identified inside the shear bands. Research funded by the US Army Research Office, Office of Naval Research, and the National Science Foundation

9:25 AM INVITED PAPER

WHAT DETERMINES THE SPACING BETWEEN DISLOCATION NUCLEATION SITES AT A CRACK TIP: EXPERIMENTAL OBSERVATIONS AND ANALYSIS: *K. Jimmy Hsia*[1]; Yun-Biao Xin[2]; Huajian Gao[3]; [1]University of Illinois at Urbana-Champaign, Dept. of Theoretical and Applied Mech., 216 Talbot Lab, 104 S. Wright St., Urbana, IL 61801 USA; [2]MEMC Electronic Materials, 1505 Lockwood Lane, St. Peters, MO 63376 USA; [3]Stanford University, Dept. of Mech. Eng., 267 Durand Bldg., Stanford, CA 94305 USA

It is now well documented through experimental studies that, in brittle single crystals such as silicon, nucleation of dislocation loops from a crack tip occurs in a heterogeneous fashion at discrete dislocation sources. It has also been argued that such nucleation can only occur at imperfections along the crack front such as ledges or atomic scale steps. It is not clear, however, what controls the spacing between these preferred dislocation loop nucleation sites, whether it is the spacing between the imperfections along the crack front, or the interaction stresses between the dislocation loops. An experimental technique was developed to study the dislocation nucleation condition at an atomically sharp crack tip in silicon single crystals. Four-point-bend specimens with a through thickness cleavage crack are loaded at elevated temperature under a stress intensity level slightly below the critical stress intensity for fracture. The whole loading period lasts sufficiently long so that the nucleated dislocation loops have enough time to move away from the crack tip region. The specimens are then cooled down to freeze the crack tip dislocations, and subsequently loaded to fracture. Fracture surface examination shows a periodic waviness of the fracture surface during subsequent fracture, indicating evenly spaced dislocation nucleation sites. The wavelength of this waviness is always on the order of one micron, a length scale significantly larger than the atomic scale such as the magnitude of the Burgers vector, regardless of the temperature and load level during high temperature holding test. An analytical model was developed to explain this observation. The model takes into account of the interaction between dislocation lines and the crack tip. The

result shows that, due to the interaction of crack tip dislocations, there should exist a preferred spacing between parallel dislocation lines. The estimate of this spacing based on the model is consistent with the experimental measurements.

9:55 AM BREAK

10:10 AM INVITED PAPER
GEOMETRICALLY-NECESSARY DISLOCATION DENSITY AND SCALE-DEPENDENT PLASTICITY: *David M. Parks*[1]; Tom Arsenlis[1]; [1]MIT, Mech. Eng, Rm. 1-308, Cambridge, MA 02139 USA

Recently, several models of scale-dependent crystal plasticity have been proposed in order to account for experimentally-observed scale effects such as indentor-size dependence of hardness and grain-size effects on polycrystalline strain hardening. A conceptual basis underlying many of the models is Ashby's interpretation of "geometrically-necessary" dislocation density as related to plastic slip gradient, with attendant hardening associated with the on-going interactions of glissile dislocations with both the "statistically-stored" and the "geometrically-necessary" dislocation densities. Although the fundamental concept of geometrically-necessary dislocation density is intimately related to the spatial organization of the lattice, several isotropic (lattice-less) versions of the models have been developed by introducing invariant measure(s) of the plastic strain gradient tensor and conjugate material length scales of order lattice spacing divided by yield strain. In contrast, we retain the lattice and its spatial curl (equivalently, the material curl of plastic deformation gradient) quantifying Nye's tensor as fundamental. For the FCC lattice, an algorithm uniquely associates geometrically-necessary densities of pure screw (six systems) and edge (twelve systems) dislocations with Nye's tensor. These densities, in conjunction with statistically-stored dislocation density, define the deformation resistances of each crystallographic slip system. The model is realized via finite elements within which both slip and Nye's tensor are evaluated. A typical application demonstrates the grain-size dependence of polycrystalline strain hardening. Results from both idealized planar double-slip models and full three-dimensional simulations are presented and discussed.

10:40 AM
A DISCRETE DISLOCATION ANALYSIS OF INDENTATION: H. H.M. Cleveringa[2]; E. van der Giessen[2]; *Alan Needleman*[1]; [1]Brown University, Eng. Dept., P.O. Box D, Providence, RI 02912 USA; [2]Delft University of Technology, Laboratory for Engineering Mechanics, P.O. Box 5033, 2600 GA, Mekelweg 2, Delft 2628 CD The Netherlands

A framework for analyzing boundary value problems, where plastic flow arises from the collective motion of large numbers of discrete dislocations, is described. Within this framework, the plastic stress-strain response and the evolution of the dislocation structure are outcomes of the boundary value problem solution. The dislocations are modeled as line defects in an isotropic linear elastic solid. The stresses and strains are written as superpositions of fields due to the discrete dislocations and complimentary (or image) fields that enforce the boundary conditions. This leads to a linear elastic boundary value problem for the image fields which is solved by the finite element method. Hence, the long range interactions between dislocations are accounted for through the continuum elasticity fields. Drag during dislocation motion, dislocation nucleation and annihilation are incorporated into the formulation through a set of constitutive rules. Although valid for full three-dimensional problems, attention is confined to a small strain, two-dimensional formulation. Here, this framework is used to analyze indentation within a plane strain context. The discrete dislocation predictions are compared with corresponding predictions of continuum plasticity theory.

11:00 AM
DIFFUSION ASSISTED DISLOCATION CLIMB IN INTERMETALLIC GAMMA-TiAl: *Fritz Appel*[1]; [1]GKSS Research Center, Institute for Materials Research, Max-Planck-Str., Geesthacht D-21502 Germany

Titanium aluminide alloys based on the intermetallic gamma-TiAl phase have received increasing attention over the past decade due to their potential as high temperature structural materials. However, for technical applications the materials suffer from insufficient creep resistance at the intended service temperature of about 700ϒC. The paper reports an experimental study of diffusion controlled deformation mechanisms in two-phase titanium aluminides which apparently cause the degradation of the strength properties at elevated temperatures. Electron microscope in situ heating studies were performed in order to characterize diffusion controlled dislocation climb. Climb velocities were analyzed in terms of diffusion coefficients and the critical vacancy supersaturation necessary for the operation of diffusion assisted dislocation sources. The experimental results will be discussed concerning the structural stability of two-phase titanium aluminides and potential factors for improving the high temperature strength.

11:20 AM
LATTICE REORIENTATIONS DURING COMPRESSION DEFORMATION OF A [110] TA SINGLE CRYSTAL: *Adam J. Schwartz*[1]; Wayne E. King[1]; Geoffrey H. Campbell[1]; James S. Stolken[1]; John Y. Shu[1]; David H. Lassila[2]; David D. Sam[3]; Brent L. Adams[4]; Shilei Sun[4]; [1]Lawrence Livermore National Laboratory, Chem. & Mats. Sci. Directorate, L-355, P.O. Box 808, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, B-Division, L-170, P.O. Box 808, Livermore, CA 94550 USA; [3]Lawrence Livermore National Laboratory, Mech. Eng., L-125, P.O. Box 808, Livermore, CA 94550 USA; [4]Carnegie Mellon University, Mats. Sci. and Eng., Wien Hall, Pittsburgh, PA 15213 USA

High-purity tantalum single crystal cylinders oriented with [110] parallel to the cylinder axis were deformed 10, 20, 30, and 40 percent in compression. The samples were subsequently sectioned for characterization using Orientation Imaging Microscopy (OIM) along two orthogonal sectioning planes: one in the plane containing [110] and [001] (longitudinal) and the other in the plane containing [110] and [1-10] (transverse). To examine local lattice rotations, the Euler angles relative to a reference angle at the section center were decomposed to their in-plane and out-of-plane components. The in-plane and out-of-plane misorientation maps for all compression tests reveal inhomogeneous deformation everywhere and particularly large lattice rotations in the corners of the longitudinal section. Of particular interest are the observed alternating orientation changes. This suggests the existence of networks of dislocations with net alternating sign that are required to accommodate the observed rotations. Rotation maps from the transverse section are distinctly different in appearance from those in the longitudinal plane. However, the rotation maps confirm that the rotations observed above were about the [1-10] axis. Alternating orientation changes are also observed on this section. Results will be directly compared with crystal rotations predicted using finite element methods. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

MILTON BLANDER INTERNATIONAL SYMPOSIUM ON "THERMODYNAMIC PREDICTIONS AND APPLICATIONS": Ionic Liquids

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee, ASM International: Materials Science Critical Technology Sector, Thermodynamics & Phase Equilibria Committee
Program Organizers: Ramana Reddy, University of Alabama, Dept. of Met. & Mats. Engr., Tuscaloosa, AL 35487 USA; Dr. A. D. Pelton, Montreal, Quebec H3C3A7 Canada

Monday AM Room: 4
March 1, 1999 Location: Convention Center

Session Chairs: Arthur D. Pelton, Ecole Polytechnique, CRCT, Montreal, Quebec H3C 3A7 Canada; L. A. Curtiss, Argonne National Laboratory, Chem. Div., Argonne, IL 60439 USA

8:30 AM INVITED PAPER

THE POWER OF THERMODYNAMIC MODELING. EXAMPLES FROM MOLTEN HALIDE MIXTURES: *Harald A. Oye*[1]; [1]Institute of Inorganic Chemistry, Norwegian University of Science and Technology, Trondheim N-7034 Norway

Although thermodynamics does not describe structure, models with complex species have often been very useful to describe and predict thermodynamic properties. An early example is Flood and Urnes prediction of the KCl-MgCl2 phase diagram on the KCl side by assuming the presence of MgCl4[2-]. Examples will be given from aluminium chloride-alkali chloride systems where total vapour pressure of binary systems was determined. Through modeling it was possible to describe the liquid phase, composition of the gas phase and phase diagram for binary and ternary systems. Determination of thermodynamic properties which was nearly inaccessible for experimental delamination was also accomplished through modeling.

9:10 AM

ACIDITY IN AMBIENT TEMPERATURE CHLOROALUMINATE IONIC LIQUIDS: *Robert A. Osteryoung*[1]; [1]North Carolina State University, Dept. of Chem., Raleigh, NC 27695-8204 USA

The ambient temperature chloroaluminate ionic liquids to be discussed are mixtures of AlCl3 and 1-ethyl-3-methylimidazolium chloride (ImCl). The systems are defined as acidic, basic, or neutral if the mole ratio of AlCl3 to ImCl is greater than, less than, or equal to unity. The dominant Lewis acid species is Al2Cl7[-], which can react with a weak base, B:, to form and adduct, B:AlCl3. A Bronsted acid, such as HCI, becomes a superacid in acidic melts. Neutral melts are of much interest in the they have a very wide electrochemical window of ≅4.5 volts. These system can be buffered to neutrality by addition of an alkali halide, MCI, to an acidic melt, where the reaction Al2Cl7[-]+MCl(arrow)2AlCl4[-]+M[+] takes place. The neutrality condition is that M[+]+Im[+]=AlCl4[-]. MCl is insoluble in the neutral melt. These buffered systems have unexpected acid/base properties, which will be discussed, as will the acidity of the proton in these buffered systems.

9:30 AM

SOLUBILITIES AND RAMAN SPECTRA OF NdOCl IN SOME CHLORIDE MELTS OF INTEREST FOR THE ELECTROWINNING OF MAGNESIUM FROM ITS OXIDE: H Mediaas[1]; O. Tkatcheva[2]; V. Drakopoulos[3]; G. N. Papatheodorou[3]; G. J. Kipouros[4]; *T. Ostvold*[1]; [1]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway; [2]Institute of High Temperature Electrochemistry, Ekaterinburg Russia; [3]University of Patras and Institute of Chem. Eng. and High Temp. Chem. Proc., Dept. of Chem. Eng., Forth, Patras Greece; [4]Dalhousie University, Dept. of Mining and Metall. Eng. (DalTech), P.O. Box 1000, Halifax, Nova Scotia B3J 2X4 Canada

Some fundamental data related to the solvent proposed for a new technical electrolytic process for magnesium production based on MgO as the raw material are presented. The new process seems mainly to be based on some phase diagram data determined by Russians from 1969 to 1975. The MgCl2-NdOCl phase diagram has been reinvestigated. The solubility of MgO and NdOCl in pure liquid NdCl3, in NdCl3-MgCl2 and in MgCl2-NdCl3-NaCl liquid mixtures has also been studied. These melts are of interest for the technical process. The solubility of MgO is decreased when MgCl2 is added to the pure NdCl3, and further by additions of NaCl as expected. A so far unknown compound having the composition $Mg_xNd_yOCl_{2x+3y-2}$ where x and y are larger than 1 is formed in these melts. This compound precipitates at temperatures higher than 910°C in the NdCl3-MgO quasi binary system containing about 8 mole%MgO, and seems to remain suspended in the melt down to about 750 C where the first liquid- solid phase transition was observed. XRD data of filtered samples of this solid show new X-ray lines not detected in MgCl2, NdCl3, NaCl, MgO and NdOCl. The published phase diagram of the quasi binary system MgCl2-NdOCl is, according to the present work, not correct, and the solubility of MgO is much less than reported previously. Raman spectroscopic data of NdCl3-MgCl2-NdOCl melts show the known features of the NdCl3-MgCl2 and NdCl3-NdOCl melts. Raman bands due to dissolved species of the new compound were not detected. In view of the obvious small concentration of this species in the liquid phase, this was reasonable.

9:50 AM

LANTHANIDE HALIDE MELTS: FROM FUNDAMENTALS TO TECHNOLOGICAL APPLICATIONS: *M. Gaune-Escard*[1]; Francisco Da Silva[1]; Leszek Rycerz[2]; Ryuzo Takagi[3]; Ashok Adya[4]; [1]IUSTI, CNRS-UMR 6595, Technopole de Chateau-Gombert, 5 rue Enrico Fermi, Marseille, Cedex 13 13453 France; [2]Technical University of Wroclaw, Institute of Inorganic Chemistry and Metallurgy of Rare Elements, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw Poland; [3]Tokyo Institute of Technology, Research Laboratory for Nuclear Reactors, Ookayama, Meguro-ku, Tokyo 152 Japan; [4]University of Abertay Dundee, Division of Applied Chemistry, School of Molecular & Life Sciences, Bell St., Dundee, Scotland DD1 1HG UK

From a fundamental point of view, the lanthanides constitute the longest series of chemically similar elements contained within an interesting region of the periodic table where the variety of structural and bonding possibilities is high. On the other hand, lanthanide halide melts and their mixtures with alkali halides play a major role in technological applications such as lanthanide and lanthanide-based alloys production, processing of nuclear wastes, recycling of spent nuclear fuel and the lighting industry. We have recently started a multidisciplinary research programme on the macroscopic (thermodynamic, physicochemical) and microscopic (structural) behavior of molten lanthanide halides and their mixtures with alkali metal halides. Computer simulations are then performed on these systems. Thermodynamic and physicochemical studies involve experimental determinations of several properties (such as temperature and enthalpy of phase transition, enthalpy of mixing, phase diagram, electrical conductivity, etc.) as modeling and numerical procedures (such as phase diagram optimization). Structural investigations are carried out by Neutron Diffraction Isotopic Substitution (NDIS) techniques. The Partial Distribution Functions (PDF) provided new information regarding the detailed structure of molten lanthanide melts on a short and intermediate range length scale. These experimental structure factors are then compared to those obtained by molecular dynamics (MD) computer simulation, based on the Rigid Ion Model (RIM) with Born-Mayer-Huggins pair potential or, very recently, on the Polarisable Ion Model (PIM) by including the effects of anion polarisation.

10:20 BREAK

10:30 AM

SOLUTIONS OF IRON OXIDES IN MOLTEN CRYOLITE: *Ernest W. Dewing*[1]; Jomar Thonstad[2]; [1]Retired, 648 Pimlico Pl., Kingston, Ontario K7M 5T8 Canada; [2]Norwegian University of Science and Technology, Institute for Industrial Electrochemistry, Trondheim 7034 Norway

Iron oxides (FeO, Fe3O4, Fe2O3 and FeAl2O4) appear to dissolve in cryolite-alumina melts as iron fluorides and alumina. Both Fe(II) and Fe(III) are present in the solutions, and experimental results are interpreted on that basis. Predictions are made of the variation of solubility with oxygen pressure, and the standard potential of the Fe[2+]/Fe[3+]redox couple is calculated. The anode and anode gas of an industrial Hall-Heroult cell appear to be insufficiently oxidizing to cause significant conversion of Fe(II) to Fe(III). An anomaly in the liquidus diagrams for FeF2-Na3AlF6 and FeO-Na3AlF6 is accounted for in terms of solid solution of FeF2 in cryolite.

10:50 AM

COUPLED EXPERIMENTAL AND THERMODYNAMIC MODELLING STUDIES FOR METALLURGICAL SMELTING AND COAL COMBUSTION SLAG SYSTEMS: *E. Jak*[1]; S. Degterov[2]; B. Zhao[1]; A. D. Pelton[2]; P. C. Hayes[1]; [1]The University of Queensland, Dept. of Mining, Minerals and Materials Engineering, St. Lucia, Queensland 4072 Australia; [2]Centre for Research in Computational Thermochemistry, Ecole Polytechnique de Montréal, P.O. Box 6079, Station Downtown, Montréal, Québec H3C 3A7 Canada

An extensive research program focused on the characterisation of various metallurgical complex smelting and coal combustion slags is being undertaken. The research combines both experimental and thermodynamic modelling studies. The approach is illustrated by work on the system PbO-ZnO-FeO-Fe2O3-CaO-SiO2-Al2O3. Experimental measurements of the liquidus and solidus have been undertaken under oxidizing and reducing conditions using equilibration, quenching and

electron probe X-ray microanalysis. The experimental program has been planned so as to obtain data for thermodynamic model development as well as for pseudo-ternary liquidus diagrams which can be used directly by process operators. The thermodynamic modelling has been carried out using the computer system FACT which contains thermodynamic databases with over 5000 compounds as well as evaluated solution models. The FACT package is used for the calculation of slag/solid/gas/matte/metal/salt equilibria in multi-component systems of industrial interest. A modified quasi-chemical solution model is used for the liquid slag phase. New optimisations have been carried out which significantly improve the accuracy of the thermodynamic models for lead/zinc smelting and coal combustion processes. Examples of experimentally determined and calculated liquidus diagrams are presented. These examples provide information of direct relevance to various metallurgical smelting and coal combustion processes.

11:10 AM

PHYSICAL CHEMISTRY OF THE MOLTEN NA2O-SO3-WO3 SYSTEM: *Ray Y. Lin*[1]; [1]University of Cincinnati, Mats. Sci. and Eng., M.L. #12, Cincinnati, OH 45221-0012 USA

Thermochemical properties of the Na2O-SO3-WO3 ternary oxide system have been investigated at 1200K by the thermogragravimetric method and the following emf cell, Pt(s), O2(g), Na2O-WO3 melt | Na, b-alumina | Ternary melt, O2(g), SO2(g), Pt(s). The thermochemical properties of the melt at the reference electrode (Na2O-WO3 melt), have been investigated previously. The SO3 isobars in this system at 1200K were determined by the thermogravimetric technique. The activities of Na2O and SO3 in the ternary Na2O-SO3-WO3 melt were calculated using the Gibbs-Duhem integration technique for the ternary system.

11:30 AM

CoCl+: UNIQUE IN ALL OF MOLTEN SALTDOM: Soghomon Boghosian[1]; Peter J. Tumidajski[2]; M. Blander[3]; *David S. Newman*[4]; [1]Foundationfor Research and Technology-Hellas, Institute of Chem. Eng. and High Temp. Chemical Processes, PO Box 1414, GR-265 00, Patras, Greece; Tumidajski Technologies, 259 Springfield Blvd., Toronto, Ontario Canada M4C 1Z8; [3]Quest Research, 1004 East 167 Place, South Holland, IL 60473-3114; [4]Dep't. Chemistry, Bowling Green State University, Bowling Green, OH 43403 USA

The UV-vis spectra of $CoCl_2$ dissolved in molten NaCl-$AlCl_3$ solutions were obtained as a function of composition from slightly basic compositions to fairly acidic compositions and the solubility of $CoCl_2$ was determined using an EMF technique. The cell used was Co/$CoCl_2$ - $NaAlCl_4$//(AgCl)x/Ag, where the mole fraction, x, is fixed. Saturation at 175°C was detected when [dE/dx($CoCl_2$)]T=0. A very sharp minimum in the solubility was found int he vicinity of the 50.00%NaCl-50.00%$AlCl_3$ composition and a distinct spectrum with three broad peaks belonging to neither tetrahedrally nor octahedrally coordinated Co++ was obtained in this composition region. The solution can be described in terms of six associated coalt species of the form CoCln(2-n), where n = 1 to 6, indicating that the solubility of $CoCl_2$ in the NaCl-$AlCl_3$ melts is related to the formation of chloro-metallic species that are present in much higher concentration than Co++ ions with no bound Cl-ions solvated with $AlCl_4$– ions. In the composiiton range where the unique three peaked specrum was observed, the species whose concentration is far greater than any other is CoCl+ and we think that this species is responsible for the observed spectrum. As far as we can discern, this is the only completely asymmetric species that has been identified as existing in a molten salt solution.

11:50 AM

THE NATURE OF ASH DEPOSITS ON THE MOLTEN SALT PROCESS FOR HAZARDOUS WASTE DISPOSAL: *S.J. Yosim*[1]; R.L. Gay[2]; [1]Retired from Rocketdyne, a division of Rockwell International, 23812 Killion St., Woodland Hills, CA 91367; [2]Molten salt consultant, 10012 Hanna Ave., Chatsworth, CA 91311USA

In the molten salt process for the destruction of hazardous waste, the waste is destroyed while submerged in a pool of Na_2CO_3 at 900-1,000°C. Many types of hazardous wastes have been destroyed in molten salts. Molten salt bench-scale tests showed that ash deposit formation on the vessel walls sometimes took place when high ash-content waste was processed. A series of bench-scale tests was performed in an attempt to learn about the nature and cause of these ash depsoits. These deposits formed only in the oxidizing mode (excess air) but not in the reducing mode (deficient air). Deposit formation is irreversible; once they have formed during the oxidizing mode, they cannot be removed by subsequently performing the reducing mode. The deposits which contain about 50% ash increase with increasing (1) ash content of the melt, (2) melt temperature, and (3) gas superficial velocity. A mixture of Al_20_3-SiO_2, presumably in the form of aluminosilicates, appears to be responsible for these ash deposits. The compounds SiO_2, Fe_2O_3, and SiO_2-Fe_2O_3 do not result in ash formation. More needs to be known about these ash deposits and the cause of their formation. For example, it is not known why ask deposits occur only in the oxidizing mode. An experimental program is recommended which will lead to a better understanding of the mechanism of ash deposit formation, to find methods of avoiding deposit formation in the oxidizing mode, and to study methods of removing ash deposits once they have formed.

NONDESTRUCTIVE EVALUATION (NDE) AND MATERIAL PROPERTIES IV: Nondestructive Evaluation (NDE) and Material Properties Session I

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Nuclear Materials Committee

Program Organizers: Peter K. Liaw, University of Tennessee, Dept. of Mats. Sci. & Eng., Knoxville, TN 37996-2200 USA; Richard J. Arsenault, University of Maryland, Nuclear Eng. Bldg., College Park, MD 20742-2115 USA; Robert E. Green, The John Hopkins University, Baltimore, MD 21218-2689 USA; K. Linga Murty, North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA; R. Thompson, Iowa State University, Ames Laboratory, Ames, IA 50011 USA

Monday AM — Room: 16A
March 1, 1999 — Location: Convention Center

Session Chairs: Peter K. Liaw, University of Tennessee, Dept. of Mats. Sci. and Eng., Knoxville, Tennessee 37996-2200 USA; Bruce R. Thompson, Iowa State University, Center for NDE, Ames, Iowa 50011 USA

8:30 AM INVITED PAPER

ULTRASONIC MONITORING OF RECRYSTALLIZATION IN ALUMINUM: *G. Liu*[1]; D. K. Rehbein[1]; R. B. Thompson[1]; [1]Iowa State University, Ames Laboratory, Ames, IA 50011 USA

Understanding recrystallization processes is an important step towards optimization of the processing of metals. Here, the use of ultrasound to monitor recrystallization in aluminum is considered, with the objective of monitoring the temporal evolution of texture during the hot rolling process. Included in the presentation will be a discussion of the information that can be gained from measurements of the velocity and attenuation of longitudinal and transverse waves propagating through the thickness and a report on progress towards measuring those quantities with electromagnetic-acoustic transducers, requiring no coupling to the part and hence able to make measurements at elevated temperatures on moving parts.—This work was sponsored by the Division of Materials Sciences of the U.S. Department of Energy and was performed at the Ames Laboratory in cooperation with the Center for NDE. Ames Laboratory is operated by Iowa State University for the USDOE under contract W-7405-ENG-82.

9:00 AM INVITED PAPER

EFFECT OF GRAIN ELONGATION ON ULTRASONIC ATTENUATION AND BACKSCATTERING: *P. D. Panetta*[1]; Y. Guo[1]; F. J. Margetan[1]; R. B. Thompson[1]; [1]Iowa State University, Center for NDE, 1915 Scholl Rd., Ames, IA 50011 USA

The determination of grain size from measurements of ultrasonic attenuation and/or backscattering is well established. However, these techniques usually assume that the grains are equiaxed. In this paper, we consider the case of elongated grains. As might be expected, experimental results in aluminum and titanium show that both the attenuation and backscattering can be highly anisotropic. However, less obvious is the fact that the directions of high attenuation are often those of low backscattering and vice versa. An interpretation of this phenomenon, in terms of both experimental observations of phase fluctuations of the beam in certain propagation directions and theoretical predictions of the effect of grain shape on the two quantities, will be presented. Implications for determining the size and shapes of grains will be discussed. This work was supported by the NSF Industry/University Cooperative Research Program and the Engine Titanium Consortium under the Federal Aviation Administration Grant No. 94-G-048.

9:30 AM

NEUTRON DIFFRACTION STUDY OF THE MECHANICAL BEHAVIOR OF NiAl-ZrO_2 COMPOSITES: *Hahn Choo*[1]; Mark Bourke[1]; Philip Nash[2]; [1]Los Alamos National Laboratory, Los Alamos Neutron Science Center, MS H805, Los Alamos, NM 87545 USA; [2]Illinois Institute of Technology, Mech., Mats. and Aerospace Eng., 10 W. 32nd St., Chicago, IL 60616 USA

Neutron powder diffraction was used to study the mechanical behavior of NiAl-based composites reinforced by zirconia (ZrO_2) particles. The composites with 0, 10, 20, 30 and 40 volume percent of ZrO_2 were processed by mixing NiAl and zirconia powders using a high energy attrition mill and hot pressing of the powder mixture. The ability to load the samples in the neutron beam permitted us to perform in-situ loading experiments. The evolution of strain in each phase in the composites was characterized as a function of the particle volume fraction and the applied load at room temperature. The thermally-induced residual stress in each phase was also studied and the results were correlated to the mechanical behavior of the composites.

10:00 AM

NEUTRON DIFFRACTION INVESTIGATION OF THE MECHANICAL AND CRYSTALLOGRAPHIC PROPERTIES OF SUPERELASTIC NiTi AND NiTi-TiC COMPOSITES: *Raj Vaidyanathan*[1]; Mark A. M. Bourke[2]; David C. Dunand[3]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., Room 8-139, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]Los Alamos National Laboratory, LANSCE/MST, MS H831, Los Alamos, NM 87545 USA; [3]Northwestern University, Dept. of Mats. Sci. and Eng., MLSB 1123, 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Austenitic NiTi alloys can deform by undergoing a stress-induced transformation from austenite to martensite resulting in uniaxial strains of up to 8%; upon removal of the load, martensite becomes unstable and reverts back to austenite with a concomitant recovery of the strain, a phenomenon called superelasticity. Superelastic NiTi samples with and without TiC particulate reinforcement were subjected to simultaneous uniaxial compressive loading and neutron diffraction. A Rietveld refinement procedure using information from the entire neutron spectra and incorporating a spherical harmonics texture formulation is used to analyze the data. Changes in texture are quantified as the martensite volume fraction varies during the forward transformation on loading and the subsequent back transformation on unloading. The discrete phase strain description includes a factor that tracks changes in the anisotropic component of the strain. For NiTi-TiC composites, load transfer is examined for the case where the matrix deforms by a stress-induced phase transformation in the presence of elastic reinforcement.

10:30 AM

ACOUSTIC EMISSION IN DETECTING FATIGUE AND FRACTURE BEHAVIORS: *M. Huang*[1]; L. Jiang[1]; P. K. Liaw[1]; C. R. Brooks[1]; R. R. Seeley[2]; D. L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 West Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

Acoustic emission (AE) is a technique to monitor the defect formation and failures in structural materials used in services or in laboratories. Moreover, the AE method has been developed and applied in numerous structural components, such as steam pipes, pressure vessels, as well as in the research areas of rocks, composite materials, and metals. In this paper, the basic concept, terminology, theoretical modeling, and common setup associated with AE are described. The applications of AE in fatigue and fracture investigations of metals and composites are also discussed. Most of the literature available uses the traditional AE technique that only captures AE parameters, including AE counts, peak levels, and energies. These parameters can be correlated with the defect formation and failures. Some of the researchers analyze the waveforms of AE as functions of sources and wave propagation mechanisms. Above all, AE was found to be an effective way of detecting fatigue and fracture behaviors of materials. The present work is supported by Haynes International, Inc. and the Center for Materials Processing, the University of Tennessee, Knoxville.

PROFESSIONAL DEVELOPMENT: Collaboration, Research, and Professional Skills

Sponsored by: Young Leaders Committee

Program Organizers: Elliot Schwartz, The Gillette Company, S. Boston, MA 02127-1096 USA; James Dunne, Westinghouse Electric Corporation, Commercial Nuclear Fuel Division, Ogden, VT 84404 USA; Livia Racz, Tufts University, Dept. of Mech. Eng., Medford, MA USA 02155; Mary Windfeld, REBL-Refractories Lab, Ltd., Surrey, BC V4N 1N1 Canada

Monday AM — Room: 14A
March 1, 1999 — Location: Convention Center

Session Chairs: Elliot Schwartz, The Gillette Co., S. Boston, MA 02127-1096 USA; Livia Racz, Tufts University, Dept. of Mech. Eng., Medford, MA 02155 USA

8:30 AM

HOW TO BUILD EFFECTIVE PARTNERSHIPS BETWEEN NATIONAL LABORATORIES AND INDUSTRY: *Siegfried S. Hecker*[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., MS G754, Los Alamos, NM 87544 USA

During my 12 years as director of the Los Alamos National Laboratory I learned a number of key lessons critical to building successful, sustainable partnerships with industry. First and foremost, the laboratory must have a compelling national mission to justify the government's investment and to replenish its intellectual resources. At Los Alamos, that compelling mission today is our responsibility to help reduce the global nuclear danger - through stewardship of the nation's nuclear weapons stockpile, guarding against the proliferation of nuclear weapons, and dealing with the legacy of 50 years of nuclear weapons production. Second, a laboratory should partner in its enlightened self interest. Industry performs more than 70 percent of all the R&D in the United States. The laboratories must work closely with industry to best conduct their own missions. They should chose to partner in those areas of technology that are key to their own missions. At Los Alamos, we have chosen to concentrate on areas such as modeling and simulation, high-performance computing and information technologies, and materials and smart sensors. Such partnerships can enhance the research environment at the laboratories and help stretch the time and risk horizons of industry. Simply stated, my philosophy is that to do world-class science, you must partner with universities and to do state-of-the-art technology, you must partner with industry.

8:55 AM

NATIONAL SCIENCE FOUNDATION OPPORTUNITIES FOR COLLABORATIVE RESEARCH: *Bruce A. MacDonald*[1]; [1]National Science Foundation, Division of Materials Research, 4201 Wilson Blvd., Suite 1065, Arlington, VA 22230 USA

The National Science Foundation encourages research collaborations through a number of program activities. This results from the recognition that significant science and engineering advances usually occur through the collective interactions of individuals. Furthermore, expanded opportunities for education of students are created through such collaborations. This presentation provides examples of collaborations within the NSF Individual Investigator Award (IIA) research programs, particularly the Grant Opportunities for Academic Liaison with Industry (GOALI) Program and the Faculty Early Career Development (CAREER) Program, aimed at junior people in tenure-track positions. Opportunities for collaboration within the Division of Materials Research include activities created by the presence of the Materials Research Science and Engineering Centers (MRSEC's) and the National Facilities Program. A main goal of the NSF Division of International Programs is promotion of collaborative research between U.S. researchers and researchers in foreign countries, and pertinent examples are discussed. Also covered is the Small Business Technology Transfer (STTR) Program, which encourages collaborative opportunities with universities as part of the NSF Small Business activity.

9:20 AM

MANAGING RESEARCH & DEVELOPMENT UNDER MULTI-COMPANY COLLABORATION: *Jerome Paul Reimann*[1]; [1]NCMS, 3025 Boardwalk, Ann Arbor, MI 48108 USA

During the 20 century, society has moved from an environment of propriety technology to an environment of abundance of technology. Instead of competing for the same customer base with the propriety technologies, companies that grow and prosper in this environment have found it necessary to look for new opportunities to expand their markets. Collaboration has been found to be an extremely effective methodology for the generation of ideas for companies to find new opportunities to grow and prosper. Since the National Cooperative Research Act was passed in 1984, many organizations have been formed to take advantage of pre-competitive collaborative research and development among competing laboratories and companies. In the more than fifteen years since the law passed, initial investment in this new experiment in manufacturing R&D has grown tremendously. At the same time, companies accustomed to "drawing down" public funds for manufacturing research are realizing that they are in a better position to drive the research, deploy the results and protect their intellectual property rights by funding research initiatives themselves in collaboration with others who are also willing to pool talent and share risks. This presentation will address a number of models for conducting pre-competitive R&D from large consortiums to small collaborative projects. Also addressed will be issues related to acamedia and national laboratory concerns versus private industry, handling of mixed funds from government and commercial entities, and concerns of intellectual property ownership. Fiercely competitive companies have been brought together to perform research and development in areas of new processes, materials, manufacturing tools as well as new methodologies for management and logistics.

9:45 AM

INTELLECTUAL PROPERTY MODELS FOR COLLABORATIVE R&D: *Patrick Ziarnik*[1]; [1]NCMS, Vice President and General Counsel, 3025 Boardwalk, Ann Arbor, MI 48108 USA

Since the National Cooperative Research Act was passed in 1984, many organizations have been formed to take advantage of pre-competitive collaborative research and development among competing laboratories and companies. In the more than fifteen years since the law passed, initial investment in this new experiment in manufacturing R&D has grown tremendously. But when companies and independent laboratories realize their standard practices for protecting their intellectual property rights are inadequate, issues arise on the best practices for handling this new experiment. Some organizations have refused to get involved with research initiatives that require collaboration with others who are also willing to pool talent and share risks. This presentation will address a number of models for handling intellectual property under pre-competitive R&D. Also addressed will be issues related to acamedia concerns, government rights, national laboratory initiatives, and private industry concerns as related to intellectual property ownership. Rather than avoid opportunities in R&D that is growing at an increasing rate, organizations need to arm themselves with the tools needed to participate in this new arena.

10:10 AM GENERAL DISCUSSION

10:30 AM BREAK

10:45 AM

EVERYTHING YOU NEED TO KNOW ABOUT BEING A MANAGER BUT NEVER LEARNED IN COLLEGE: *Ned Bahtishi*[1]; [1]Western Zirconium, 10,000 West, 900 South, Ogden, UT 84404-9799 USA

For many engineers, higher education consisted of a rigorous technical curriculum, an engineering ethics class, and a swift boot out the door. This is unfortunate for the engineer because nearly 20% of all engineers will find themselves in a management position after only their second career move. It is often assumed that management training will come with the job or through continuing education seminars; however, this takes place after the engineer is already in a management position and has fought their first battle coming away licking their wounds. This paper accounts one young engineer's management experience in an effort to enlighten others. Two topics of discussion will be: what is/is not a manager; and, what are the most valuable skills a manager can master. It will be assured that this paper will include plenty of anecdotes and practical references.

11:10 AM

EFFECTIVE PROJECT PLANNING AND MANAGEMENT: *James Bryan Dunne*[1]; [1]Westinghouse Electric Company, Commercial Nuclear Fuel Div., 10,000 West, 900 South, Ogden, Utah 84404 USA

Many projects are unsuccessful due to incomplete project definition and planning, scarce resource availability, and commitment from stakeholders. The majority of projects experience "killer" issues or radical changes that require the priority or desired outcome to change. Most projects will require modification if the goals of time, cost and performance are to be achieved. The topics discussed will be project definition and planning issues that must be complete before the project actually begins. Project management discussions and clear communication among stakeholders are vital to project success.

11:35 AM

SURVIVING A LAYOFF: *Elliot Schwartz*[1]; [1]The Gillette Company, Boston Research and Development, One Gillette Park, 6D-1, South Boston, MA 02127-1096 USA

Layoffs can occur under any economic conditions, including the favorable conditions that we are currently enjoying in the United States. Despite the lowest unemployment rates in decades, many engineering positions are being eliminated as part of downsizing at various companies. My recent experience with having my position eliminated and the resulting personal, financial, and professional issues will be discussed. The various aspects of my subsequent employment search to be detailed include working with an out placement service, career assessment, resume preparation, networking, interviewing, and negotiation. Many useful employment search tips will be presented.

REVIEW OF EXTRACTION PROCESSING, PROPERTIES & APPLICATIONS OF REACTIVE METALS: Session I

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizers: Brajendra Mishra, Colorado School of Mines, Dept. of Metall. & Mats. Eng., Golden, CO 80401-1887 USA; Georges J. Kipouros, Dalhousie University, Dal Tech., NS B3J2X4 Canada

Monday AM Room: 5B
March 1, 1999 Location: Convention Center

Session Chairs: Dr. B. Mishra, Colorado School of Mines, Golden, CO 80401 USA; G. J. Kipouros, Dal Tech, Dalhousie University, NS B3J2X4 Canada

8:30 AM OPENING REMARKS:
This symposium highlights the state-of-the-art in extraction, processing, properties and applications of several prominent reactive metals. Complete review papers on selected metals are being presented. The presentations are intended to provide a brief over-view of all aspects of metal sources, beneficiation, extraction (all processes) and recovery, metal refining, intermediate processing, casting and recycling as well as the thermochemical and physico-chemical properties. Relevant aspects of mechanical processing and physical metallurgy for the enhancement of properties in these metals will also be addressed. In addition, applications of these metals, both current and potential applications, shall be a major focus of these presentations.

8:30 AM
LITHIUM: *D. R. Sadoway*[1]; [1]Massachuetts Inst. of Technoloy, Dept. Mats. Sci. and Eng., 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

9:00 AM
BERRYLIUM: *B. Mishra*[1]; D. L. Olson[1]; [1]Colorado School of Mines, Dept. of Metall. & Mats. Eng., Golden, CO 80401 USA

9:30 AM
MOLYBDENUM: *M. C. Jha*[1]; [1]Qualchem, Inc., 18290 W. Highway 72, Arvada, CO 87007 USA

10:00 AM BREAK

10:15 AM
TITANIUM: *S. J. Gerdemann*[1]; [1]Albany Research Center, Dept. of Energy, 1450 Queen Ave. S.W., Albany , OR 97321-2198 USA

10:45 AM
SODIUM: *H. M. Blank*[1]; [1]E. I. DuPont-Specialty Chemicals R&D, Jackson Lab, 1094/228, Deepwater, NJ 08023 USA

11:15 AM
TANTALUM: *S. Yuan*[1]; [1]Cabot Corporation, Performance Metals, P.O. Box 1608, County Line Rd., Boyertown, PA 19512 USA

SHEET METAL FORMING TECHNOLOGY: Session I

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee
Program Organizer: Mahmoud Y. Demeri, Ford Research Labs, Manuf. Systems Dept., Dearborn, MI 48121 USA

Monday AM Room: 11B
March 1, 1999 Location: Convention Center

Session Chair: Mahmoud Y. Demeri, Ford Research Labs, Manuf. Systems Dept., Dearborn, MI 48121 USA

8:30 AM INVITED PAPER
ROBUST FORMING PROCESS BY PULSATING BLANKHOLDER FORCES, MULTIPOINT CUSHION SYSTEMS & CLOSED LOOP CONTROL: *Klaus Siegert*[1]; Stephen Wagner[1]; Michael Zierler[1]; [1]University of Stuttgart, Institute for Metal Forming Technology, Holzgartenstrasse 17, Stuttgart D-70174 Germany

The trend in stamping technology is to use single acting and transfer pressses with hydraulic multipoint cushion systems in the press table. Such systems control material flow between the binders of the draw die during the forming stroke. Special die designs are needed to implement such techniques. Pulsating blankholder forces can reduce friction between the blank and the binders. Closed loop controls for the blankholder force can react automatically to changes in input parameter such as lubrication.

9:00 AM
RECONFIGURABLE TOOLING FOR SHEET METAL FORMING: *John M. Papazian*[1]; David Hoitsma[1]; Lembit Kutt[1]; John Melnichuk[1]; Jerrel Nardiello[1]; Allan Pifko[1]; Robert C. Schwartz[1]; [1]Northrop Grumman, Technical Development, A01-26, Bethpage, NY 11714 USA

A computer-controlled, reconfigurable tooling system is being developed for production of sheet metal parts by stretch forming. The working surface of the tool consists of the ends of numerous discrete elements, currently 1.125 in. square pins with hemispherical tips. The position of each pin is controlled by a computer and can be changed in order to change the shape of the tool. Reconfiguration of the entire 4 ft. by 6 ft. tool will be accomplished in less than 10 minutes. Other essential elements of the system are a deformable polymer interpolating layer, a Deformation Transfer Function-based shape control loop, and process modeling and simulation. The polymer layer interpolates the discrete ends of the tool and provides a smooth working surface. The Deformation Transfer Function involves comparing a Fourier transform of the measured part shape to the desired shape and provides an error signal that is used to reconfigure the tool to ensure correct final shape of the part. This approach eliminates part shape errors induced by springback or other phenomena. A complete process model and simulation capability is part of the system. The simulation is used to guide process development, and accelerates convergence to the correct shape. Significant reduction in manufacturing costs for aerospace sheet metal components is anticipated through elimination of the fabrication, handling, and storage costs of fixed tooling, and the elimination of the assembly costs associated with incorrectly shaped parts.

9:20 AM
NON-SYMMETRIC PANEL FORMING OF AA6111-T4 USING ACTIVE DRAWBEADS: Rui Li[1]; *Klaus J. Weinmann*[1]; [1]Michigan Technological University, Mechanical Engineering Department, 1400 Townsend Drive, Houghton, MI 49931-1295 USA

Drawbeads have been widely used in sheet metal stamping for many years as control elements of sheet metal flow into the die. This paper describes an experimental investigation in which active drawbead technology is used to study the formability of AA6111-T4 non-symmetric

panels. Drawing limit curves in terms of draw depth versus blankholder force (BHF) are plotted and different drawbead trajectories are tested so as to determine an optimal drawbead trajectory scheme. A corresponding finite element model was also formulated. The excellent agreement between predicted results and experimental data validates the model.

9:40 AM

IMPROVEMENT OF CONSISTENCY IN STAMPED PART QUALITY USING PROCESS CONTROL: *Cheng-Wei Hsu*[1]; Mahmoud Y. Demeri[2]; A. Galip Ulsoy[1]; [1]The University of Michigan, Dept. of Mech. Eng. and Applied Mechanics, 2277 G. G. Brown Bldg., 2350 Hayward St., Ann Arbor, MI 48109-2125 USA; [2]Ford Motor Company, Scientific Research Lab., 20000 Rotunda Dr., Mail Drop 3135, Dearborn, MI 48121 USA

In sheet metal forming, the blank holder is used to control the material flow into the die cavity. It has been shown that adjustment of the blank holder force can improve formability and part accuracy. Process control has been applied to adjust the blank holder force in real time. Consistency in stamping quality is also important but has not been addressed thoroughly. In this paper, reasons for improvement of consistency in stamped part quality through process control will be investigated. The process control strategy based on the measurement of the punch force will be used. Improvement of formability and accuracy will be shown through selection of the reference punch force trajectory. Improvement of consistency will be shown through the intrinsic properties of feedback control.

10:00 AM BREAK

10:20 AM

SPRINGBACK CONTROL VIA VARIABLE RESTRAINING FORCE IN SHEET METAL FORMING: *Michael J. Saran*[1]; Nana Nkansah-Andoh[1]; Mahmoud Y. Demeri[2]; [1]Case Western Reserve University, Dept. of Mats. Sci. and Eng., 516 White Bldg., Cleveland, OH 44106-7204 USA; [2]Ford Motor Company, Manufacturing Systems Dept, Ford Research Labs, MD 3135, Dearborn, MI 48121 USA

Robust operating window and subsequent part quality are major concerns during sheet metal stamping. For a given part geometry, material, and lubrication conditions, the restraining force (RF) on the sheet metal is the key parameter controlling metal flow into the die cavity, thus influencing formability and quality (including springback) of the formed part. Recent advances in press and die building provide capability of (RF) variation during the stamping stroke. A methodology for evaluating springback sensitivities is proposed, based on virtual experiments (numerical simulation) and verified by physical experiments for key selected cases. In this study, a u-shaped channel geometry was selected. Laboratory tests and numerical simulations were performed, and the sensitivities of springback to variable restraining force (VRF) were calculated in an effort to better understand the effect of various (RF) trajectories on the resulting springback.

10:40 AM

FEM OPTIMIZATION OF SHEET METAL FORMING: *A. Scott-Murphy*[1]; M. Cardew-Hall[1]; P. Hodgson[2]; S. Kalyanasundaram[1]; [1]The Australian National University, Dept. of Eng., FEIT, Canberra, ACT 0200 Australia; [2]Deakin University, School of Eng. & Tech., Geelong, VIC 3217 Australia

This work presents a methodology of the integration of Finite Element Modelling (FEM), and optimisation methods for the sheet metal forming process. By taking into account the natural variation in process variables to be expected during a production run, it is possible to optimise all the input parameters available at that time to ensure that the part is being formed with the lowest possible defect rate. The variables involved in the optimisation include die geometry, material properties, and process variables. The paper uses a rectangular cup deep drawing process to illustrate the method. Experimental work in the deep drawing of the parts was performed on a 75ton press at Ford Geelong Stamping Plant. The FE model was created in I-DEAS, and simulated using ABAQUS Explicit. The punch, die and blank holder were modelled as rigid bodies, with the blank as S4R shell elements. The total number of elements for the complete model varied between 4597 and 5797. In order to capture as much of the forming data possible, without over complicating the optimisation process, an output variable extracted from the FLD process signature was used. This method captures the stress-strain state of the part, and uses an algorithm, for each defect mode of interest, to summarise into a single variable. These modes include splitting and wrinkling, and are expected to be expanded to include problems arising from low strain areas and springback.

11:00 AM

RELIABILITY ANALYSIS OF SHEET METAL FORMING: Naji Arwashan[1]; [1]LTV Steel Co., Automotive Development Group, 2000 Town Center, Suite 540, Southfield, MI 48075 USA

This paper addresses the issue of the 10% safety factor usually used in circle grid analysis of formed parts. Successful forming requires the strains to be lower than the forming limit. However, both the strains and the forming limit are undeterministic and can vary randomly, leaving a certain possibility for the pant to fail. The reliability analysis deals with this uncertainty in the input parameters by treating them as random variables. The structural reliability analysis which is well developed and used in structural engineering applications is presented here and adapted for use in forming applications. The procedure of calculation of the probability of failure for forming is outlined and illustrated by a numerical example. Sensitivity analysis of the variability of the thickness, the work hardening exponent (n) and the strain on the probability of failure is performed. Based on this approach, one can calculate, for every application, the safety factor that should be used in order to meet the safety requirement, and the material and forming conditions for that application. The other benefits of this approach and the need for further studies are also discussed.

11:20 AM

ELECTROMAGNETIC FORMING OF SHEET METAL - MODELING AND OPPORTUNITIES: *Glenn S. Daehn*[1]; Vincent Vohnout[1]; Hemant Panshikar[1]; Doug Everhart[2]; James C. Moore[2]; [1]Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Applied Research Association, Columbus Division, Columbus, OH 43210 USA

High velocity sheet metal forming, as may be developed through electromagnetic forming, offers many advantages not present in traditional sheet metal forming. Formability is improved. Wrinkling is inhibited. Good dimensional tolerance and surface finish is available. These techniques are also easily implemented. This presentation will review these process fundamentals. If these techniques are to be widely implemented, robust numerical modeling is required. Initial explorations using a smooth-particle hydrocode, GEM, developed by Applied Research Associates will be discussed. Numerical simulations are carefully tested against experimental observation. Implementing these concepts together to design electromagnetic forming systems will be discussed.

11:40 AM

NUMERICAL AND EXPERIMENTAL RESULTS ON PULSED TUBES CALIBRATION: *Sergey Fedorovich Golovashchenko*[1]; [1]Bauman Moscow State Technical University, Engineering Technologies, Bldg. #5, 2nd Baumanskaya St., Moscow 127349 Russia

Electropulsed forming technologies are based on high-voltage short duration discharge of capacitors through the conductive coil or between two electrodes in the water. These processes generate high pressure on the sheet or tubular blank being formed, calibrated or assembled with the other parts. The important benefit of pulsed technologies is based on the blank springback supression. During the blank impact on the die surface, a compressive stress state is generated. The numerical model of the contact interaction process is based on solid mechanics equations of motion and elasto-plastic flow rule for both the blank and the die. Experimental data were obtained on electromagnetic and electrohydraulic pulsed machines for tubes with the diameter and thickness of 50mm and 1mm and also 80mm and 1 mm. Both numerical and experimental results showed that the residual springback of the blank decreases with increase in the blank size and the die impact velocity.

STRUCTURAL SILICIDES: Basic Issues

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion & Environmental Effects Committee; ASM International: Materials Science Critical Technology Sector, Mechanical Behavior of Materials Committee

Program Organizers: J.H. Schneibel, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831 USA; Michael J. Kaufman, University of Florida, Dept. of Mats. Sci. & Eng., Gainesville, FL 32611-2066 USA; Matthew J. Kramer, Iowa State University, Ames Laboratory, Ames, IA 50011USA

Monday AM Room: 16B
March 1, 1999 Location: Convention Center

Session Chairs: J. H. Schneibel, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA; R. R. Judkins, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6084 USA

8:30 AM INVITED PAPER
BONDING AND THERMOELASTIC PROPERTIES OF Mo-Si ALLOYS: *C. L. Fu*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA

The structural and elastic properties of Mo-Si alloys have been studied from first-principles calculation. An important issue for the successful application of silicides as high-temperature structural materials is to understand the physical origin for high anisotropy in the thermal expansion coefficient for 5-3 silicides (but near isotropy for disilicides). We discuss the calculation of thermal expansion coefficients from first-principles and the application of the theory to Mo_5Si_3, $MoSi_2$, and Mo_5SiB_2. In addition to providing understanding of the intrinsic strength in terms of the existence of pronounced covalent bonding, these calculations show the origin of thermal expansion anisotropy in Mo_5Si_3 lies in the anisotropy of lattice anharmonicity, which arises from the difference in the bonding characteristics in the basal plane and along the c-axis. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S.DOE under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

9:00 AM INVITED PAPER
PHYSICAL PROPERTIES AND MECHANICAL BEHAVIOR OF Mo_5Si_3-BASED SILICIDES: *Fuming Chu*[1]; Dan J. Thoma[1]; Ken J. McClellan[1]; Pedro Peralta[1]; Eric Fodran[1]; F. Li[1]; [1]Los Alamos National Laboratory, Dept. of Mats. Sci. and Eng., MST-8, Mail Stop G755, Los Alamos, NM 87545 USA

The ultra-high temperature structural pentamolybdenum silicides (e.g., Mo_5Si_3, $Mo_5(Si, Al)_3$, and Mo_5SiB_2) have been systematically studied, including materials processing, physical properties and mechanical behavior. The polycrystalline samples of these silicides have been obtained and the high purity single crystals of Mo_5Si_3 have been synthesized. The structural, thermal, elastic, and transport properties of these silicides were experimentally measured. Room temperature Vickers indentation tests on the single crystal Mo_5Si_3 and on the polycrystalline $Mo_5(Si, Al)_3$, and Mo_5SiB_2 have been performed. From the indentation studies, the hardness and fracture toughness of Mo_5Si_3-based silicides have been obtained. In addition, the corresponding deformation and fracture modes have been revealed by microscopy studies. The materials processing and mechanical behavior of these silicides will be interpreted with respect to the physical properties of the materials. Finally, a comparison of pentamolybdenum silicides with other high temperature structural silicides, e.g., C11b $MoSi_2$ and C40 $NbSi_2$, will be discussed. (This work has been supported by the U.S. DOE-OBES).

9:30 AM INVITED PAPER
MICROSTRUCTURAL DESIGNS IN HIGH-TEMPERATURE (Mo,Nb)-Si-B ALLOYS: *J. H. Perepezko*[1]; R. Sakidja[1]; Jim Myers[1]; S. Kim[1]; H. Seiber[1]; G. Wilde[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

The potential of two-phase microstructures based on (Mo,Nb) + $(Mo,Nb)_5SiB_2$ (T_2 phase) for high-temperature structural applications is being evaluated. Isothermal investigations at 1200ϒC and 1600ϒC confirm the existence of the two-phase field in both Mo-B-Si and Nb-B-Si systems. The solidification pathways in the two-phase field yield an extensive compositional segregation (especially for Mo-Si-B alloys) that necessitates prolonged annealing at 1600ϒC to attain equilibrium. Rapid Solidification Processing (RSP) has been successfully applied to suppress the solidification segregation and microstructural designs have been identified involving novel duplex structures with high temperature stability. In addition, solid state reactions can yield the formation of Mo precipitates in the T_2-phase with specific crystallographic relations and contribute to an apparent enhancement in toughness. The support of the AFOSR (F 49620-96-1-0286) is gratefully acknowledged.

10:00 AM BREAK

10:20 AM
BORON SOLUBILITY IN $Mo_{5+y}Si_{3-y}$: *Matthew J. Kramer*[1]; Mufit Akinc[1]; Jesse J. Huebsch[2]; [1]Iowa State University, Ames Laboratory, 37 Wilhelm Hall, Ames, IA 50011 USA; [2]Seagate Technology, 7801 Computer Ave., Minneapolis, MN USA

Pure phase $Mo_{5+y}Si_{3-y}$ (T1) has the tetragonal W_5Si_3 crystal structure which corresponds to the I4/mcm space group. Nowotny's early work in the Mo-Si-B system suggested that 4 - 4.5 atomic percent (at%) boron was soluble in the T1 phase at 1600ϒC. Through x-ray analysis, optical microscopy, chemical analysis, and electron probe microanalysis (EPMA), it has been determined that the level of boron solubility in $Mo_{5+y}Si_{3-y}$ at 1800ϒC is limited to about 2 at%. Several compositions within the single phase $Mo_{5+y}Si_{3-y}B_x$ (T1) region were prepared to determine how lattice parameters varied with molybdenum to silicon ratio and with boron concentration. For a constant Mo:Si ratio of 1.61, a slight contraction in the lattice volume was observed with increasing boron content. For samples with a fixed boron content of 1 at%, the unit cell volume reached a maximum value near a Mo:Si ratio of 5:3. The Ames Laboratory is operated by the U.S. Department of Energy (DOE) by Iowa State University under Contract No. W-7405-ENG-82. This work was supported by the Office of Energy Research, Office of Computational and Technology Research, Advanced Energy Projects Division.

10:40 AM
IN SITU GENERATION OF STACKING FAULTS ON (001) PLANES IN $MoSi_2$ SINGLE CRYSTALS: *S. Guder*[1]; M. Bartsch[1]; U. Messerschmidt[1]; [1]Max-Planck-Institute for Microstructure Physics, Weinberg 2, 06120 Halle/S. Germany

Stacking faults on (001) planes in $MoSi_2$ single crystals are believed to be a result of Si loss during crystal growth. The low formation energy for thermal vacancies at Si sites and the fact that they can easily be quenched-in confirm the theory of collapsed vacancy clusters forming these Frank-type faults on (001) planes. However, during in situ heating experiments of predeformed $MoSi_2$ single crystals planar faults on (001) planes were generated very quickly. The faults were covered by two partial dislocations of different character. This is not in agreement with a Frank-type fault. We assume a spontaneous formation due to a dissociation of 1/2<111> dislocations probably caused by a strong temperature dependened fault energy. For a proof in situ heating experiments of dislocation-free $MoSi_2$ single crystals and of those containing 1/2<111> dislocations were performed in a high-voltage electron microscope. Results will be presented and the possible influence of these faults on the deformation behaviour of $MoSi_2$ single crystals when controlled by glide of 1/2<111> dislocations will be discussed.

11:00 AM
PHASE FORMATION AND INTERDIFFUSION IN SILICIDES OF Mo CONTAINING Re AND Al: *Edward J. Ciecko*[1]; Mysore A.

Dayananda[1]; [1]Purdue University, School of Mats. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907 USA

Diffusion structures developed by interaction of liquid Al in contact with disks of Mo and $MoSi_2$ with and without additions of Re are investigated for the formation of phases at selected temperatures between 700-900ϒC. The phases include binary and ternary aluminides and silicides of the system Mo-Si-Re-Al. These phases are also examined for their stability at temperatures up to 1200ϒC with the aid of selected solid-solid diffusion couples. The effect of Al additions on the interdiffusion of the components in the silicides of Mo will also be discussed.

11:20 AM

EFFECT OF ALUMINUM CONTENT ON THE MECHANICAL PROPERTIES OF MONOCRYSTALLINE C11b $Mo(Si,Al)_2$: *Pedro D. Peralta*[1]; James Garrett[2]; Fuming Chu[1]; Terence E. Mitchell[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., Center for Materials Science, Mail Stop K765, Los Alamos, NM 87545-0001 USA; [2]McMaster University, Mats. Sci., Brockhouse Institute for Materials Research, ABB 439, Hamilton, Ontario L8S 4M1 Canada

Single crystals of $Mo(Si,Al)_2$ with the C11b structure (less than 3at% Al) and different aluminum contents were grown by the Czochralski technique. The mechanical behavior of these crystals was studied as a function of the Al content using Vickers hardness testing for different orientations of the indentation plane, i.e., (001) and (110). It was found that increasing Al content decreases the hardness continuously at room temperature and produces dramatic changes on the slip behavior around the indents. The effect of Al content on the Vickers hardness and the slip behavior as a function of temperature for the single crystals was also studied up to 1300ϒC. The results are interpreted in terms of recent theoretical developments regarding the effects of Al additions on the bonding of $MoSi_2$.

11:40 AM

STRUCTURE AND SOLID SOLUTION HARDENING IN $(Mo,Re)Si_2$ AND $(Mo,Re)(Si,Al)_2$ ALLOYS: *A. Misra*[1]; T. E. Mitchell[1]; [1]Los Alamos National Laboratory, MS K765, Los Alamos, NM 87545 USA

The mechanical properties of solidification processed ternary $(Mo,Re)Si_2$ and quaternary $(Mo,Re)(Si,Al)_2$ alloys were investigated by hot hardness experiments. The substitution of Mo by Re in $MoSi_2$ is expected to increase, while the substitution of Si by Al in $(Mo,Re)Si_2$ is expected to decrease the electron-to-atom ratio, and hence, significant changes in the atomic bonding and mechanical properties may be expected. Re is found to be a potent solid solution hardening addition to $MoSi_2$ in the temperature range of 25-1300ϒC; much higher than that expected from the insignificant atomic size mismatch between Mo and Re, and the low elastic moduli mismatch between $MoSi_2$ and $ReSi_{2-x}$. Since rhenium disilicide is semiconducting and has a stoichiometry of Re_4Si_7 with vacancies on Si sites, the addition of Re to $MoSi_2$ may lead to defect complexes comprising of Re substitutionals and Si vacancies resulting in the high hardnesses observed. Preliminary experiments on the substitution of Si by Al reveal decreased hardness and enhanced plasticity of $(Mo,Re)Si_2$ alloys at room temperature. The temperature dependence of strength and the solid-solution strengthening of $MoSi_2$ by Re are related to the observed dislocation substructures.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Issues in Surface Engineering

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee

Program Organizers: Yip-Wah Chung, Northwestern University, Dept. of Mats. Sci. & Eng., Evanston, IL 60208 USA; Ashok Kumar, University of South Alabama, Dept. of Elect. & Comp. Eng., Mobile, AL 36688-0022 USA; John E. Smugeresky, Sandia National Labs, , Livermore, CA 94551-0969 USA; John J. Moore, Colorado School of Mines, Golden CO 80401 USA; John L. Lombardi, Advanced Ceramic Research, Tuscon, AZ 85706-50113 USA

Monday AM Room: 7B
March 1, 1999 Location: Convention Center

Session Chairs: Yip-Wah Chung, Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208 USA; J. Narayan, N.C. State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695 USA

8:30 AM WELCOME AND OPENING REMARKS: Yip-Wah Chung, Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208 USA

8:35 AM INVITED PAPER

CURRENT NSF ACTIVITIES IN SURFACE ENGINEERING AND EMERGING RESEARCH OPPORTUNITIES: *Jorn Larsen-Basse*[1]; [1]National Science Foundation, 4201 Wilson Blvd., Room 545, Arlington, VA 22230 USA

The historical development of NSF funding support for research in Surface Engineering is outlined and examples of ongoing projects are discussed. Support has increased rapidly over the past ten or so years from almost nothing to around $ 5 million per year or more, counting the various special initiatives that include some surface engineering activities as well as the regular programs in tribology and surface engineering, materials, and processing. Also presented is a personal view of some promising potential intermediate and long-term advances in surface engineering technology and of the corresponding needs for progress in underpinning basic and applied research areas. Future requirements will undoubtedly include the need to engineer surfaces and surface material to exacting performance specifications and will also include development of surfaces with smart or intelligent elements. In order to move in that direction there will also be a substantial need for developments in more mundane areas, such as design tools, process control, and technology transfer.

9:00 AM INVITED PAPER

DIRECTIONS IN PVD COATINGS: *Michael E. Graham*[1]; [1]Northwestern University, Advanced Coating Technology Group, 1801 Maple Ave., Evanston, IL 60201 USA

PVD, CVD, and TS have been categorized as “advanced coating technologies” over the past 25 years. As emerging-processes, they were defined by their respective benefits and limitations in order to fit each technology to a class of applications that it could address. While process fundamentals still govern the possible applications, the practical limitations are being overcome through significant technological advances. In the old paradigm, PVD was considered expensive, low rate, and best suited to electronic applications. CVD was only suitable for materials that could stand high temperature (1000ϒC). TS were good for thick coatings where modest adhesion and low density were adequate for the application. Improved understanding of the processes and the relationship of process conditions to coating structures and properties, has

driven the technology advances. PVD is commercially viable for coating 3-D engineering components and retail consumer products with high rate processes that are easily automated and that give reliable quality. CVD temperatures are lowered and the applications broadened with the use of metal-organic precursors and plasma assisted processing. TS (Plasma spray and HVOF) coating qualities have improved with denser, well adhered coatings. Coatings on the order of microns to tens-of-microns are possible. This presentation will highlight some major developments in these "advancing technologies", but focus primarily on developments in sputter coating technology and consider the directions of future development in PVD coatings.

9:25 AM

LOW-COST CHEMICAL VAPOR DEPOSITION OF THIN FILMS IN THE OPEN ATMOSPHERE: *A. T. Hunt*[1]; [1]Micro Coating Technologies, 3901 Green Industrial Way, Chamblee, GA 30341 USA

Combustion Chemical Vapor Deposition (CCVD, US Patent No. 5,652,021) is a radically new technology for the deposition of thin films on a wide variety of substrates even using low vapor pressure precursors. Recent experimental studies clearly demonstrate the technologyÆs potential in a variety of thin film applications, including the manufacture of corrosion and oxidation resistant, electronic, catalytic, and optical coatings. The CCVD process can (a) be conducted in the open atmosphere, is (b) environmentally friendly, and (c) does not require expensive reaction/vacuum chambers. This results in significantly lower coating costs than compared to more traditional processes such as CVD and PVD. Furthermore, CCVD coatings are typically of equal or higher quality than those obtained by vacuum-based methods. The CCVD process can readily be implemented in a production line environment, thus enabling continuous processing. MicroCoating Technologies has deposited over 60 different inorganic materials, mostly oxides, onto a variety of substrates ranging from metals and plastics to ceramics. Films and multilayer coatings of complex composition and well-defined stochiometry have been demonstrated.

9:40 AM INVITED PAPER

SUPERHARD NANOCRYSTALLINE COMPOSITES: PRESENT STATUS OF THE RESEARCH AND POSSIBLE INDUSTRIAL APPLICATIONS: *Stan Veprek*, Institute for Chemistry of Inorganic Materials, Technical University Munich, Lichtenbergstr. 4, D-85747 Garching b. Munich, Germany; M. Jilek, SHM LTD, Masarykovo Nam. 3, CZ-787 01 Sumperk, Czech Republic

After the first papers on the concept for the design of superhard nanocrystalline composites nc-M_nN/a-Si_3N_4 (M = Ti, V, W) few years ago [1], several research groups reproduced the results and extended them to other material combinations including, for example, superhard nc-TiN/BN, TiN-TiB_2, nc-TiN/CN_x and over stoichiometric carbides. The predicted [1] universality of the design principle which - by forming an interface with a high cohesive energy - avoids the usual softening due to grain boundary sliding in the nanocrystalline range of ? 2-3 nm appears to be strongly supported by these results. Also the preparation techniques were extended from the originally used plasma chemical vapor deposition to industrially more compatible techniques, such as vacuum arc evaporation and reactive sputtering. In the present talk we shall review the present status of the research and of the understanding of the properties of these materials achieved worldwide. The second part of the talk will concentrate on the progress towards the industrialization of these coatings. We shall discuss the applications where these materials could replace the conventional hard coatings, such as TiN and $Ti_{1-x}Al_xN$, and further extend their applications towards new areas, such as dry-, hard- and high-speed machining. The results of cutting tests will be included as well. [1] S. Veprek, S. Reiprich, Li Shizhi, Appl. Phys. Lett. 66 (1995) 2640; Thin Solid Films 268 (1995) 64; J. Vac. Sci. Technol. A 14 (1996) 46; B 16 (1998)19

10:05 AM INVITED PAPER

HIGH TEMPERATURE CERAMIC OXIDE GAS SENSORS: CHEMISTRY AT THE GAS-SOLID INTERFACE: *Sheikh A. Akbar*[1]; Prabir K. Dutta[1]; [1]Ohio State University, Center for Industrial Sensors and Measurements (CISM), 2041 College Rd., Columbus, OH 43210 USA

There is considerable need for reliable sensors capable of continuous operation in harsh environments. Examples of such environments would include automobiles and power plants as well as in various chemical processes. We will discuss the research at CISM in the area of high temperature sensors, with particular focus on CO and NO sensors. Two key properties of sensors are sensitivity and selectivity. The choice of the sensing materials, their microstructure and interfacial chemical reactions are integral to the sensor properties. We have been examining titanium dioxide as the base material for resistive sensing of CO. By suitable rare-earth and transition metal dopants, it has been possible to design a sensor that detects CO, but discriminates against CH4 and NO in the temperature range of 400 - 800ºC. Electron microscopy as well as various spectroscopic studies are helping in elucidating the chemistry of CO oxidation at the titania surface. For NO detection, we are examining a zeolite based catalyst integrated with a yttria-stabilized zirconia sensor. The oxidation of NO over the zeolite Y catalyst alters the local concentration of oxygen, which leads to alterations in the EMF across the sensor surface. The mechanisms of the NO reaction on the zeolite as well as the O2 sensing properties of this class of sensors will be discussed.

10:30 AM BREAK

10:45 AM INVITED PAPER

LOW-ENERGY ION AND HYPERTHERMAL NEUTRAL BEAMS FOR SEMICONDUCTOR AND METAL FILM GROWTH: EFFECTS ON NUCLEATION, MICROSTRUCTURE EVOLUTION, EPITAXIAL THICKNESS, ROUGHENING, AND STRAIN RELAXATION: *Joe Greene*[1]; [1]University of Illinois, Mats. Sci. Dept., Urbana, IL 61801 USA

Low-energy (10-100 eV) ion and hyperthermal neutral irradiation during film growth from the vapor phase are used to provide new chemical reaction pathways, modify film growth kinetics, and, hence, controllably alter film properties. During low-temperature epitaxial growth from hyperthermal Si beams, critical epitaxial thicknesses were increased by up to an order of magnitude over those obtained with MBE due to enhanced interlayer mass transport and more effective filling of interisland trenches. For heteroepitaxial Si1-xGex growth on Si(001), AFM and XTEM studies show that strain-induced roughening, which occurs at elevated growth temperatures, is strongly suppressed at Ts between 300 and 400ºC, with no indication of low-temperature kinetic roughening. The use of low-energy primary-ion beam sources — in which ion energy and ion/neutral flux ratios can be varied independently — during the growth of Al, Cu, ScN, and TiN polycrystalline layers on SiO2 will be shown to provide dramatic differences in nucleation rates, mosaicity, preferred orientation, strain, and microstructure evolution.

11:10 AM

ION IMPLANTATION SURFACE TREATMENTS FOR Al and Ni RESULTING IN IMPROVED TRIBOLOGICAL PROPERTIES: *M. T. Dugger*[1]; D. M. Follstaedt[1]; J. A. Knapp[1]; S. M. Myers[1]; [1]Sandia National Laboratories, Albuquerque, NM 87185-0340 USA

Ion implantation has been used to produce surface microstructures in aluminum and nickel that impart substantial increases in strength and wear resistance to these materials. Finite element modeling of ultra-low load indentation tests were used to extract the mechanical properties of the implanted layers, and low load unlubricated sliding experiments have been used to examine the impact of increased surface strength on tribological behavior. Implantation of oxygen into aluminum results in a high concentration of nanometer-sized precipitates that impede dislocation motion and increase the flow stress to 2.9 GPa. This surface strengthening impairs adhesive junction growth and prolongs the onset of adhesive wear in this material. Implantation of titanium and carbon into polycrystalline nickel results in an amorphous surface layer with flow stress up to 5 GPa. In addition to blocking dislocation motion, strong binding reactions between the Ti and C atoms contribute to the increased strength. Significant increases in the number of contact cycles to the onset of adhesive interactions were observed during unlubricated sliding. For both systems, the change in wear mechanism resulted in reductions in the sliding friction coefficient. The mechanisms of extreme strengthening identified in Al and Ni are being explored in other metal systems.

11:25 AM INVITED PAPER

EFFECT OF HUMIDITY AND TEMPERATURE ON THE TENSILE STRENGTH OF OXIDE/POLYIMIDE INTERFACES IN MULTI-LAYER DEVICES AND PACKAGES: *Vijay Gupta*[1]; Michael O. Õbrien[1]; [1]UCLA, Dept. of Mech. and Aerospace Eng., Los Angeles, CA 90095 USA

The interface between the passivation (polyimide) and the oxide layers limits device performance as it is susceptible to both moisture and temperature. A fundamental approach to device reliability is to understand the mechanism of strength degradation at such interfaces and quantitatively relate the degraded strength to the duration and magnitude of the humidity and temperature treatments. The tensile strength of interfaces was measured using a previously-developed laser spallation technique, in which a laser-generated compressive stress wave is used to pull apart a single or an assembly of multilayer interfaces. The polyimide films of 3.3 µm thickness were deposited on Si wafers covered with a 750 nm-thick layer of silicon nitride. The samples were exposed to 60% RH at 30ϒC for 48 hrs. The tensile strength before and after the preconditioning was measured, which showed a maximum strength degradation of almost 25%. In situ tensile strengths at elevated temperatures were also measured. Preliminary data suggests strength degradation starting only at 50ϒC. This may lead to a mechanical design limit to the highest device density achievable.

11:50 AM INVITED PAPER

STRESS AND MATERIALS ISSUES IN HIGH-REFLECTANCE Mo/Si and Mo/Be MULTILAYER COATINGS FOR EXTREME ULTRAVIOLET LITHOGRAPHY: *P. B. Mirkarimi*[1]; [1]Lawrence Livermore National Laboratory, Information Science and Technology Program, Livermore, CA 94550 USA

In order to continue to increase transistor density on integrated circuits it will be necessary to reduce channel widths even further. Extreme ultraviolet lithography (EUVL) is one of the leading technologies being developed to produce channel widths of < 100 nm. The imaging system to be used in an EUVL tool consists of several glass ceramic optics coated with Mo/Si or Mo/Be multilayers films designed to reflect at wavelength of 11-14 nm, and the throughput of such a tool depends heavily on the coating reflectivity. There are a number of materials related issues being addressed to improve coating performance. For example, the multilayer films have a several hundred Mpa stress, and the extremely stringent surface figure requirements for these optics make it desirable to reduce the deformation due to film stress. However, any techniques developed to mitigate stress effects should do so without degrading the multilayer coating reflectivity; for example, a mere 0.2 nm increases in the multilayer interfacial roughness reduces the reflectivity to undesirable levels. Work shall be presented which describes this and other materials related issues as well as recent results. One observation is that ultrasmooth buffer-layers can be used to compensate for almost all of the multilayer film stress while incurring less than a 1% decrease in the reflectivity.

12:15 PM

FINITE ELEMENT MODELING OF THERMAL RESIDUAL AND CONTACT STRESSES OF THIN FILMS ON ALUMINUM SUBSTRATES: *R. M. Souza*[1]; G. G. W. Mustoe[2]; J. J. Moore[1]; [1]Colorado School of Mines, Advanced Coating and Surface Engineering Laboratory (ACSEL), Golden, CO 80401 USA; [2]Colorado School of Mines, Div. of Eng., Golden, CO 80401 USA

In this work, the wear behavior of hard and elastic thin films on soft and elastic-plastic substrates was studied on different stresses that develop in the system. The finite element method (FEM) was used and the analysis was conducted in two steps. Initially, a processing temperature was assumed and the FEM was used to calculate the thermal residual stresses that develop upon cooling, due to the difference in the coefficient of thermal expansion of film and substrate. In the second step, the FEM was used to model a situation were a spherical indenter applies normal forces on the system with thermal residual stresses. The resulting stresses were then calculated and results for different film thickness, film elastic modulus, and processing temperature were compared. The results were also compared with ones previously obtained without the consideration of thermal stresses.

SYNTHESIS OF LIGHTWEIGHT METALS III: Overview, Titanium - I, and Competing Materials

Sponsored by: Light Metals Division, Aluminum Committee; Structural Materials Division, Titanium Committee; ASM International: Materials Science Critical Technology Sector, Materials Synthesis & Processing Committee

Program Organizers: F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; C.M. Ward Close, DERA Farnborough, Structural Mats. Ctr., Farnborough, Hampshire GUI14OLX UK; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Neqev Israel; P. g. McCormick, University of Western Australia, Res. Ctr for Adv. Min. & Mats. Proc., Nedlands, W.A. 6907 Australia

Monday AM Room: 10
March 1, 1999 Location: Convention Center

Session Chairs: F. H. (Sam) Froes, University of Idaho, IMAP, Moscow, ID 83844-3026 USA; C. Malcolm Ward-Close, DERA, Structural Materials Centre, Farnborough, Hampshire GU14 0LX UK

8:30 AM INVITED PAPER

SYNTHESIS/PROCESSING OF LIGHT-WEIGHT METALLIC MATERIALS - PART I: *F.H. (Sam) Froes*[1]; C. Malcolm Ward-Close[2]; P. G. McCormick[3]; D. Eliezer[4]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]DERA Farnborough, Metallics Division, Griffith Bldg. (A7), Structural Materials Centre, Farnborough, Hampshire GU14 0LX UK; [3]University of Western Australia, Nedlands, W.A. 6907 Australia; [4]Ben-Gurion University of the Negev, Dept. of Mats. Engr. P.O. Box 653, Beer-Sheva 84105 Israel

Light-weight metallic materials are important in a variety of uses ranging from aerospace to sports equipment. This paper will over-view the synthesis, microstructure, mechanical properties and applications of light-weight materials with emphasis on those based on aluminum, magnesium and titanium—in monolithic and composite forms.

8:50 AM INVITED PAPER

SYNTHESIS/PROCESSING OF LIGHT-WEIGHT METALLIC MATERIALS - PART II: *F.H. (Sam) Froes*[1]; C. Malcolm Ward-Close[2]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]DERA Farnborough , Metallics Division, Griffith Bldg. (A7), Structural Materials Centre, Farnborough, Hampshire GU14 0LX UK

Light-weight metallic materials are important in a variety of uses ranging from aerospace to sports equipment. This paper will over-view the synthesis, microstructure, mechanical properties and applications of light-weight materials with emphasis on those based on aluminum, magnesium and titanium—in monolithic and composite forms.

9:10 AM

FABRICATION AND APPLICATIONS OF ULTRA-LIGHTWEIGHT Ti64 and Be-Al POROUS CORE SANDWICH MATERIALS: *Donald S. Shih*[1]; Daniel S. Schwartz[1]; Mark Svilar[2]; Brian Norris[3]; Richard J. Lederich[1]; Donald A. Deuser[1]; [1]The Boeing Company, Mailcode S111 0141, P.O. Box 516, St. Louis, MO 63166-0516 USA; [2]Brush Wellman ,Inc., 17876 St. Clair Ave., Cleveland, OH 44110 USA; [3]BFG Aerospace, P.O. Box 878, Chula Vista, CA 91912 USA

Because of their high stiffness-to-weight ratio, Ti64- and Be-Al-based porous-core sandwich materials offer great potential in weight and/or cost savings for aircraft structures. The low-density-core (LDC) is an integral powder metallurgy process for making sandwich structures with porous core. Inert gas such as argon is first trapped and compressed in metal powders by HIP consolidation. The HIPed materials can then

be conventionally hot worked into various product forms. Subsequent annealing causes the entrapped gas to expand, forming discreet rounded pores throughout the core covered with solid facesheets. Using the LDC process, Ti64 sandwich panels with ~35% porous core have been produced up to a size of ~2,100 x 1,300 x 4 mm^3. AlBeMet® 162 sandwich materials have also been made using the LDC process, but currently on a smaller scale. Component manufacture processes for the LDC Ti64 sandwich panels were identified and demonstrated. They included drape forming, superplastic forming, drilling and countersinking. Mechanical property and structural efficiency analysis were evaluated. Many parts have been identified in Boeing products as application targets. For porous Ti sandwich panels they include outboard and inboard fuselage skins for the F/A-18E/F. The novel LDC process, mechanical properties, manufacturing technology and applications will be presented. This study is sponsored by DARPA and ONR, contract no. N00014-95-2-0007 and N00014-96-C-0398, and Boeing.

9:30 AM

CHARACTERIZATION OF A CARBON STEEL SHEET WITH A SUBMICROCRYSTALLINE STRUCTURE: *M. D. S. Pirzada*[1]; A. Zakirova[2]; R. Zaripova[2]; G. A. Salishchev[2]; O.N. Senkov[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Russian Academy of Science, Institute for Metals Superplasticity Problems, 39 Khalturina Str., Ufa 450001 Russia

Samples of a low carbon steel (0.2% C) were subjected to severe plastic deformation by multiple forging followed by warm rolling in random directions, which resulted in a submicrocrystalline structure. An increased solubility of carbon in α- Fe was detected. The microstructure evolution on heating was studied using TEM, EDS and X-ray diffraction methods. Rockwell hardness of the alloy was also studied. It increased after the deformation, probably due to the grain refinement and increased carbon solubility. One hour aging at 300-550ºC of the severe plastically deformed samples led to a maximum hardness at about 400ºC.

9:50 AM BREAK

10:05 AM

SYNTHESIS OF NANOCRYSTALLINE TiC AND ITS DISPERSION IN A METAL MATRIX: *E. G. Baburaj*[1]; S. K. Menon[2]; Dwight Linch[1]; Swati Ghosh[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Navel Postgraduate School, Dept. of Mech. Eng., Mech. Eng. Bldg. (245), Monterey, CA 93943 USA

Microcrystalline metal carbides, in bulk quantities, are currently manufactured by comminution of commercially available carbides and hence the cost of production increases in proportion to the reduction in particle size. Fine metal carbides have also been produced by plasma processing, mechanical alloying, carbothermal reduction of ultrafine oxides and chemical vapor deposition techniques. The challenge with all these techniques is to successfully scale up production to commercial volumes of nanocrystalline materials with properties and economies that allow their wide spread use. Preliminary work at the University of Idaho has demonstrated the feasibility of synthesizing titanium carbide by the reaction, $2TiCl_4 + CaC_2 + 3Mg$ * $2TiC + CaCl_2 + 3MgCl_2$, induced by mechanical alloying at ambient temperatures, in a short processing time of a few minutes. The reaction product after leaching and drying is TiC powder with a size distribution in the range of 10 to 300 nm. Detailed chemical analysis of the individual crystals by PEELS analysis does not show impurities. The nanocrystalline carbide powder has been dispersed in a copper matrix by mechanical alloying. Consolidation and characterization of the TiC/copper composite are in progress.

10:25 AM

EFFECT OF PROCESSING CONDITIONS ON THE PROPERTIES OF MECHANICALLY MILLED ALUMINUM/ALUMINA COMPOSITES: *Gordon Fisher*[1]; Paul G. McCormick[1]; [1]University of Western Australia, Special Research Centre for Advanced Mineral and Materials Processing, Nedlands 9907 Australia

Al/Al_2O_3 composites containing up to 40% Al/Al_2O_3 have been synthesized by mechanical milling. Mechanical milling was found to result in a homogeneous dispersion of 200 nm Al/Al_2O_3 particles in an aluminum matrix. Consolidation via drop forging resulted in sample densities exceeding 95% of theoretical. Measurements of the effect of the alumina content and heat treatment on the resulting mechanical properties were carried out. A significant improvement in properties was obtained through a two-stage milling process, which enabled enhanced cold welding of the milled particles through variation of the process control agent during milling.

10:45 AM

SYNTHESIS AND PROPERTIES OF NANOCRYSTALLINE PRODUCED BY MECHANICAL MILLING: *Steve Hwang*[1]; Paul G. McCormick[1]; [1]University of Western Australia, Dept. of Mech. and Mats. Eng., Nedlands 9907 Australia

Nanocrystalline magnesium powder with a grain size of approximately 40 nm was prepared by ball milling using a modified SPEX 8000 mixer mill. The modified milling operation markedly reduced the adhesion of the powder to the vial and balls in comparison to the normal milling process. Up to 90% of initial charge mass was recovered without addition of lubricant using the modified milling operation. Different milling ball sizes and mass charges were used to study the evolution of the grain size and strain during milling. The as-milled powder was vacuum cold pressed to form fully dense cylindrical specimens. The room temperature mechanical properties were determined using compression tests. The samples exhibited remarkably high values of ductility in all cases and a significant improvement in yield strength after sintering, as compared to commercially available pure magnesium bar.

11:05 AM

MODELLING CONSOLIDATION OF MATRIX-COATED FIBRE COMPOSITES: *J. Carmai*[1]; F.P.E. Dunne[1]; B. Derby[1]; [1]University of Oxford, Dept. of Eng. Sci., Parks Rd., Oxford OX1 3PJ England

Metal matrix composites are increasingly attractive for high temperature aerospace applications due to their high stiffness and strength. They can be manufactured from matrix-coated fibres by aligning the coated fibres into a die or a canister which is subjected to a high temperature process such as Hot Isostatic Press (HIP) or Vacuum Hot Pressing (VHP). During the consolidation process, densification occurs by the in elastic flow of the matrix. A simple predictive model for overall densification behavior of continuous matrix-coated fibre metal matrix composites has been developed and will be discussed in this paper. The model has been implemented into the finite element software, ABAQUS, by means of a user-defined subroutine. The results obtained from the simulation are compared with independent data available in the literature.

THE MARTIN E. GLICKSMAN SYMPOSIUM ON SOLIDIFICATION AND CRYSTAL GROWTH: Fundamental: Solidification and Crystal Growth

Sponsored by: Materials Processing and Manufacturing Division, Solidification Committee

Program Organizers: Dr. N. B. Singh, Northrop Grumman Corporation, Pittsburgh, PA 15235 USA; Dr. Steven P. Marsh, Naval Research Laboratory, Code 6325, Washington, D.C. 20375 USA; Krishna Rajan, Rensselaer Polytechnic Inst., Dept. of Mats. Sci. & Eng., Troy, NY 12180-3590 USA; Prof. Peter W. Voorhees, Northwestern University, Dept. of Mat. Sci. & Eng., Evanston, IL 60208 USA

Monday AM Room: 11A
March 1, 1999 Location: Convention Center

Session Chairs: Peter W. Voorhees, Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208 USA; Robert Schaefer, NIST, Dept. of Metall., Gaithersburg, MD 20899 USA; John I. Mickalonis, Westinghouse Savannah River Company, Dept. of Metall., Aiken, SC 29808 USA

8:30 AM INTRODUCTION

8:45 AM
COMPLEMENTARY COARSENING IN TWO PHASE ALLOYS: *Paula J. Crawford*[1]; Michael J. Shaw[1]; Martin E. Glicksman[1]; [1]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., 110 Eighth St., Troy, NY 12180 USA

Phase coarsening theory applied to the growth of a dispersed phase within a matrix indicates that the dispersed phase follows self-similar coarsening behavior. However, little attention has been focused on the coarsening behavior of the matrix. Marsh and Glicksman have developed a statistical theory describing the coarsening behavior of both the matrix and the dispersed phase. [1]Coarsening of the matrix and the dispersed phase are correlated through a linear transformation derived from self-similar dynamics. Recently, Bender and Ratke [2]presented experimental results for Cu-Co liquid phase sintering indicating that the matrix follows self-similar coarsening behavior. The current study will characterize both the particle and the matrix coarsening behavior in the solid-solid region of AgCu, AlCu or other suitable two phase alloys. Particle size distributions are analyzed using digital image analysis techniques. Experimental rate constants will be discussed for alloys with various volume fractions of the dispersed phase and compared to the theoretical values. [1]S. P. Marsh and M.E. Glicksman, Acta Mater. 44 (1996) 3761. [2]W. Bender and L. Ratke, private communication, 1997.

9:15 AM INVITED PAPER
SOLIDIFICATION OF FACETING ALLOYS: *R. Abbaschian*[1]; M. Beatty[1]; F Chen[1]; [1]University of Florida, Dept. of Mats. Sci. and Eng., Gainesville, FL 32607 USA

The dendritic growth and morphology for nonfacet forming materials are fairly well understood in large part due to the outstanding contributions of Glicksman and his co-workers. For facet forming materials, on the other hand, the situation is quite different. For a faceted material growth takes place by the lateral motion of steps, and interfacial kinetics anisotropy becomes dominant in the onset of morphological instability as well as dendrite formation. In this presentation, the influences of growth kinetics, anisotropy and alloy additions on the kinetics and morphological stability of faceted materials will be presented.

9:35 AM INVITED PAPER
OSTWALD RIPENING OF TWO-PHASE MIXTURES: A TEST OF THEORY: *J. Alkemper*[1]; V. Snyder[1]; *P. W. Voorhees*[1]; N. Akaiwa[2]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208 USA; [2]NRI for Metals, 1-2-1 Senger, Tsukuba 305-0047 Japan

The kinetics of Ostwald ripening are examined in a solid-liquid mixture that satisfies all assumptions of theory. To avoid the density driven sedimentation of the solid particles found on earth, the experiments were performed on the Space Shuttle during the MSL-1 and MSL-1R missions. We find that even at the longest coarsening time the samples are close to but never reach the steady-state coarsening regime. The spatial distribution of the particles, as measured by the radial distribution function on a plane of section, is also evolving in time. The coarsening rate agrees with predictions of the theories for transient Ostwald ripening to within the errors of the thermophysical parameters. These theories also describe the evolution of the particle size distribution very well. We thus conclude that the nonzero volume fraction theories of coarsening are sound. This work is supported by NASA

9:55 AM INVITED PAPER
EVOLUTION OF LOCAL MICROSTRUCTURES: SPATIAL INSTABILITIES OF COARSENING CLUSTERS: *Donald O. Frazier*[1]; [1]NASA Marshall Space Center, Space Science Laboratory, Huntsville, AL 35812 USA

This work examines the diffusional growth of discrete phase particles dispersed within a matrix. Engineering materials are microstructurally heterogeneous, and the details of the microstructure determine how well that material performs in a given application. Critical to the development of designing multiphase microstructures with long-term stability is the process of Ostwald ripening. Ripening, or phase coarsening, is a diffusion-limited process which arises in polydisperse multiphase materials. Growth and dissolution occur because fluxes of solute, driven by chemical potential gradients at the interfaces of the dispersed phase material, depend on particle size. The kinetics of these processes are "competitive," dictating that larger particles grow at the expense of smaller ones, overall leading to an increase of the average particle size. The classical treatment of phase coarsening was done by Todes, Lifshitz, and Slyozov, (TLS) in the limit of zero volume fraction, VV, of the dispersed phase. Since the publication of TLS theory there have been numerous investigations, many of which sought to describe the kinetic scaling behavior over a range of volume fractions. Some studies in the literature report that the relative increase in coarsening rate at low (but not zero) volume fractions compared to that predicted by TLS is proportional to, whereas others suggest. This issue has been resolved recently by simulation studies at low volume fractions in three dimensions by members of the Rensselaer/MSFC team. Our studies of ripening behavior using large-scale numerical simulations suggest that although there are different circumstances which can lead to either scaling law, the most important length scale at low volume fractions is the diffusional analog of the Debye screening length. The numerical simulations we employed exploit the use of a recently developed “snapshot” technique, and identifies the nature of the coarsening dynamics at various volume fractions. Preliminary results of numerical and experimental investigations, focused on the growth of finite particle clusters, provide important insight into the nature of the transition between the two scaling regimes. The companion microgravity experiment centers on the growth within finite particle clusters, and follows the temporal dynamics driving microstructural evolution, using holography.

10:15 AM BREAK

10:35 AM INVITED PAPER
SOLIDIFICATION PARAMETERS AND STRUCTURES IN THE COLUMNAR TO EQUIAXED TRANSITION IN LEAD-TIN ALLOYS: Alicia Esther Ares[2]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Faculty of Sciences, 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/UNaM, Faculty of Sciences, 1552 Azara St., Posadas, Misiones 3300 Argentina

Lead-Tin alloys were solidified directionally upwards under different thermal conditions and alloy compositions in order to study the columnar to equiaxed conditions for the transition as well as the solidification

parameters during both the columnar and equiaxed growth. The alloy compositions employed were Lead containing Tin from 2% to 40%. A number of relevant solidification parameters were determined from the measured temperatures such as temperature gradients, position of the liquid and solid fronts, solidification velocity, amount of heat extraction. In addition, the size of the columnar and equiaxed grains were determined. The results show that there are critical low values of gradients, heat flow and solidification speed associated with the transition. These values are independent of the columnar size and alloy composition. There is recalescence observed and associated with the transition which magnitude is measured. The speed of liquidus front determined from the temperature measurements show a sudden acceleration. The equiaxed structure obtained show a smaller grain size right after the transition. These results are presented, analized and compared with theory.

11:05 AM INVITED PAPER
DIRECTIONAL SOLIDIFICATION MICROSTRUCTURES: *R. Trivedi*[1]; [1]Iowa State University, Dept. of Mats. Sci. and Eng., Ames Laboratory US-DOE, Ames, IA 50011 USA

During directional solidification of alloys, the interface morphologies change from planar to cellular to dendritic as the velocity is increased. Theoretical and experimental results on the conditions for transitions in these morphologies along with the scaling laws for different length scales of microstructures for each morphology will be presented. Specific emphasis will be placed on the effect of convection on morphological transitions, and quantitative experimental results, in the Al-Cu system, on the effects of convection on microstructures will be discussed.

11:35 AM INVITED PAPER
PHYSICAL VAPOR TRANSPORT GROWTH OF MERCUROUS HALIDE CRYSTALS FOR ACOUSTO-OPTIC TUNABLE FILTERS: *N. B. Singh*[1]; *R. H. Hopkins*[1]; D. R. Suhre[1]; L. H. Taylor[1]; W. Rosch[1]; M. Gottlieb[1]; W. M. B. Duval[2]; M. E. Glicksman[3]; [1]Northrop Grumman Corporation, 1350 Beulah Rd., Pittsburgh, PA 15235 USA; [2]NASA Lewis Research Center, Brook Park Rd., Cleveland, OH 44135 USA; [3]R.P.I., Dept. of Mats. Sci. and Eng., Troy, NY 12180 USA

Mercurous halides are very exciting materials for imaging acousto-optic tunable filters because of very unusual combination of properties such as extremely low acoustic velocity, broad transparency range, large birefringence and large photoelastic constant. These properties result in a large acousto-optic figure of merit (2600 x quartz). We have developed a procedure to synthesize and purify large batches of Hg2Br2 source material for crystal growth. Cm size crystals were grown by physical vapor transport method in <110>, an orientation required for acousto-optic devices. The growth velocity in <001> was observed to be approximately two times faster than <110> orientation. Experimentally measured acoustic velocity is 2.73x104 cm/s for slow shear <110> wave mode. The measured value of acoustic attenuation in good quality crystal was 11.8 dB/microsGH2.We expect this value to be lower with further improvements in crystal quality. We thank NASA Microgravity Science and Applications Division Code UG for financial support.

11:55 AM
SOLIDIFICATION BEHAVIOR OF ORGANIC MATERIALS WITH LARGE ANISOTROPY IN S-L INTERFACE ENERGY: *Om Prakash Singh*[1]; N. B. Singh[2]; Martin E. Glicksman[3]; [1]K.N.Post-Graduate College, Gyanpur, Ravidas Nagar, UP India; [2]STC-ESSD, Northrop Grumman Corporation, 1350 Beulah Rd., Pittsburgh, PA 15235 USA; [3]R.P.I., Dept. of Mats. Sci. and Eng., Troy, NY 12180 USA

Solidification behavior of several organic materials with low entropy of fusion has been studied. We have chosen succinonitrile, camphene, pivalic acid and cyclohexanol for the detailed study. The solid-liquid interface anisotropy for these materials is very a lot. For example cyclohexanol has anisotropy an order of magnitude higher than succinonitrile and camphene. Predendritic and dendritic morphology, and difference in coarsenic behavior is studied for these materials. Our observations show that anisotropy play very important role in solidification and melting structures. The materials with larger anisotropy have larger tendency to coarsen faster and dendritic skelton collapses faster.

Tutorial Luncheon Lecture: "Damascus Steels"
Time: 12:00 Noon - 1:30 PM
Room: 16A
Location: San Diego Convention Center

Hume-Rothery Award Symposium
Time: 2:00 PM
Room: 14A
Location: San Diego Convention Center

11TH INTERNATIONAL SYMPOSIUM ON EXPERIMENTAL METHODS FOR MICROGRAVITY MATERIALS SCIENCE: Session I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Thermodynamics & Phase Equilibria Committee; NASA Microgravity Sciences
Program Organizer: R. A. Schiffman, R.S. Research, Inc., Crystal Lake, Barton, VT 05822 USA; C. Patuelli, Dipartimento di Fisica and Istituto Nazionale ci Fisica per la Materia, Alma Mater Studiorum, Berti Pichat 6/2, 40127 Bologna, Italy

Monday PM Room: 15B
March 1, 1999 Location: Convention Center

Session Chair: Hideki Minagawa, Hokkaido Nationa Industrial Research Institute, Mats. Div., Toyohira-ku, Sapporo 062-8517 Japan

2:00 PM INTRODUCTION AND WELCOME: R. A. Schiffman, R.S. Research, Inc., Crystal Lake, Barton, VT 05822

2:15 PM
GROWTH OF MERCUROUS HALIDES SINGLE CRYSTALS IN SPACE: Cestmir Barta[1]; *Martin Bernas*[1]; *Milos Klima*[1]; [1]BBT-Materials Processing, Doubicka 11, Prague 8, Czech Republic 184 00 Russia

The BBT-Materials Processing company has participated in numerous materials experiments in microgravity which have been performed on board MIR station during last 8 years. All these experiments were based upon the experimental facility CSK-1C manufactured in BBT-Materials Processing and facility TITUS manufactured also in BBT-Materials Processing in a cooperation with DLR-MUSC and Humboldt Univ. (Germany). Both facilities are still operational on board MIR orbital station. On the other hand we have developed an on-ground growing technology for the mercurous halides single crystals - optical materials with exceptional physical properties and high potential for technical applications. Some disadvantages of the on-ground growth are expected to be eliminated under microgravity conditions. From this point, the most critical issue seems to be a combination of very high density and plasticity of mercurous halides on one hand, and the convective disturbances on the other hand. Therefore, the growth of large crystals is strictly limited by the weight of particular crystal and some potential impacts on the final crystal quality could be derived. The paper summarises recent results in the field of mercurous halides single crystal growth and presents the proposed space experiments and their expected goals. The development of the original growing technology under microgravity conditions is the fundamental one.

2:35 PM
THE ROLE OF SURFACE STRUCTURES IN THE NUCLEATION PROCESSES OF AN ALUMINIUM MATRIX CONTAINING 23% OF ORIENTATED SiC WHISKERS: *C. Patuelli*[1]; R. Tognato[1]; [1]Dipartimento di Fisica and Istituto Nazionale ci Fisica per la Materia, Alma Mater Studiorum, Berti Pichat 6/2, Bolonga 40127 Italy

A model discussing the effects of SiC whiskers surface structures on the nucleation process in a liquid aluminium matrix is presented. The surfaces may be flat or stepped. The results are compared with experimental data.

2:55 PM
INFLUENCE OF CONVECTION ON ALIGNED COMPOSITE GROWTH IN HYPERMONOTECTIC Al-In ALLOYS: *L. J. Hayes*[1]; J. B. Andrews[1]; [1]University of Alabama at Birmingham, Mats. and Mech. Eng., Birmingham, AL 35294 USA

Aligned composite growth of In-rich fibers in an Al-rich matrix can be achieved in hypermonotectic Al-In alloys through directional solidification under interfacially stable conditions, i.e. a sufficiently high thermal gradient to growth rate ratio. During directional solidification, however, a solute depleted boundary layer is expected to develop at the solidification front. In the Al-In system and most other immiscible alloy systems, the solute depleted boundary layer results in an unfavorable density gradient with a more dense liquid above a less dense liquid. This convectively unstable situation is expected to lead to flow in the liquid in advance of the solidification front. The effect of this flow on compositional uniformity along the length of hypermonotectic Al-In samples will be presented. In addition, this presentation will address the effect of the flow on the ability to maintain an aligned composite morphology in these alloys.

3:15 PM
EXPERIMENTAL RETRIEVING OF GROWTH CHARACTERISTICS FROM THE MOTION OF AEROSOL CRYSTALS IN MICROGRAVITY: *A. A. Vedernikov*[1]; J. C. Legros[1]; O. Dupont[1]; C. Lockowandt[2]; [1]Universite Libre des Bruxelles, Chimie-Physique EP-MRC, Avenue F.D. Roosevelt, Brussels 50 CP 165, B-1050 Belgium; [2]Swedish Space Company, Solna Sweden

It was shown recently that even in a uniform aerosol, particles will acquire additional non-thermal motion in presence of heterogeneous reaction. This 'chemojet motion' may be much more intensive Brownian motion but still quite weak under normal gravity than conditions. The goals of this work are: 1) to analyze the relation between growth mechanisms and motion of the aerosol crystals, 2) to determine favorable investigation conditions, 3) to work out relevant experimental procedures and 4) to develop flight experimental facility. We have chosen growth rate fluctuations and surface nucleation intensity to be estimated by analyzing trajectories of urotropine crystals dispersed in a binary gas mixture of argon and oversaturated urotropine vapor. A new method of aerosol formation was proposed and tested in microgravity. An optical module was developed for determination of 3D-trajectories of aerosol particles and for discrimination between crystals and reference particles which are not subjected to the chemojet motion.

3:35 PM BREAK

3:55 PM
INFLUENCE OF CONVECTION INTERFACE STABILITY OF HYPERMONOTECTICS DURING DIRECTIONAL SOLIDIFICATION: *J. D. Barnes*[1]; J. B. Andrews[1]; S. R. Coriell[2]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., Birmingham, AL 35294 USA; [2]National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899 USA

This study is designed to permit direct observation of flows generated during the directional solidification of hypermonotectic alloys and the influence of these flows on interface stability. The transparent metal-analog system succinonitrile-glycerol is being used along with a transparent cell assembly to study the influence of convection on interface stability. A temperature gradient stage microscope with samples oriented vertically is being utilized to directionally solidify alloy compositions which result in convective instability. Experimentation is carried out to determine the effect of the flow on the morphology of the interface. The thickness of the sample cells are being varied to change the amount of damping on the fluid in order to control the flow velocity ahead of the solidification front. In addition, tracer particles are being used to determine the flow velocities and track the occurring flow patterns.

4:15 PM
POSSIBLE EFFECTS OF GRAVITY ON THE INTERFACIAL FREE ENERGY OF A SOLID CLUSTER EMBEDDED IN THE LIQUID PHASE: C. Patuelli[1]; *R. Tognato*[1]; [1]Dipartimento di Fisica and Istituto Nazionale ci Fisica per la Materia, Alma Mater Studiorum, Berti Pichat, Bolonga 40127 Italy

A model studying the effects of gravity on the interfacial free energy of a solid cluster containing 3x107 atoms and embedded in the liquid phase is discussed. It seems that as gravity decreases, there are limited decreases of the interfacial free energy.

4:35 PM
INFLUENCE OF G-JITTER ON DIFFUSION IN LIQUID METALS AND METALLOIDS: *Jose Robert*[1]; Reginald W. Smith[1]; [1]Queen's University, Dept. of Mats. & Metall. Eng., Kingston K7L 3N6 Canada

Long capillary liquid diffusion couples have been processed on the MIR Space Station using the QUELD II furnace facility, coupled to the Canadian Microgravity Isolation Mount (MIM). The MIM provides the opportunity for 1) exposing the diffusion couples to the ambient g-jitter of MIR, 2) isolating them from this and also 3) subjecting them to a forcing vibration superimposed on the isolating state. The results obtained will be presented and reviewed with respect to existing models of diffusion liquids.

4:55 PM
MICROSTRUCTURES IN GEOLOGY AND MATERIALS, SIMILARITIES AND DIFFERENCES: *Afina Lupulescu*[1]; Marian Lupulescu[2]; Martin E. Glicksman[1]; Paula J. Crawford[1]; [1]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., Troy, NY 12180 USA; [2]University of Bucharest, Dept. of Geology 70111 Romania

Materials and metallic minerals show interesting similarities and differences. The same processes, similar phase diagrams lead to comparable microstructures. Natural microstructures could take millions of years to complete, while the materials microstructures only have a few hours. Three types of microstructures will be discussed and exemplified: 1) The polycrystalline or equigranular microstructures are well distributed in nature, and laboratory. They take slow kinetics to develop, a big number of crystalline nucleii, and are characterized by triple junctions of the grain boundaries which form angles of 1200 (See FeS2 in nature, and pollycrystalline iron in laboratory). Many times these microstructures show twinned grains resulted from annealing in the laboratory (naval brass-alloy 46400), or natural twinned grains (sylvanite-AuAgTe4, bournonite-PbCuSbS3, Romania); 2) Very often in nature and laboratory are obtained the plastic deformation microstructures. Simple curly lamellar microstructure (pearlitic steel wire) or textured structures in different steels have also their natural correspondent [kink bands in biotite-K2 (Mg, Fe)6, (Fe, Al, Ti)0-2, [Al2-3, Si6-5, O20,] O0-2 (OH, F)4-2, deformed microstructures in PbS and ZnS, preferred orientations in metamorphic rocks]; 3) With increasing deformation the microstructures evolve toward microstructures with high density of dislocations. These microstructures occur upon annealing a cold-worked metal and are considered the beginning of the recovery microstructures (Fe-3Si). In nature, similar microstructures are named mylonitic, and respectively blasto-mylonitic (recrystallization) microstructures (Such microstructures created by PbS and ZnS. (Birsa Fierului, Romania).

5:15 PM
MAGNETIC DAMPING OF G-JITTER DRIVEN FLOWS IN MICROGRAVITY: 2-D AND 3-D CALCULATIONS: De-Yi Shang[1]; *Ben Q. Li*[1]; Henry C. de Groh[2]; [1]Washington State University, School of Mech. and Mats. Eng., Pullman, WA 99163 USA; [2]NASA Lewis Research Center, Cleveland, OH 44135 USA

This talk discusses the finite element model development for magnetic damping of g-jitter driven flows in microgravity. Formulations for both 2-D and 3-D models are presented and the difference in 2-D and 3-D modeling strategy described. Simulations are carried out for both synthetic g-jitter, which is characterized by a summation of Fourier series, and the real g-jitter taken from space flight, which is random and three dimensional in nature. Results are presented for a Bridgmann-Stockbarger system for the growth of Ga-doped germanium single crystals in microgravity with the presence of a DC magnetic field

ALUMINA AND BAUXITE: Specialty Alumina Products

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Joe Anjier, Kaiser Aluminum & Chemical Corporation, P.O. Box 3370, Gramercy, LA 70052 USA

Monday PM — Room: 6E
March 1, 1999 — Location: Convention Center

Session Chair: Dr. Jean Doucet, ALCAN International, Montreal, Quebec Canada

2:00 PM
HYDRATE QUALITY IMPROVEMENT: *Lynn L. Blankenship*[1]; [1]Kaiser Aluminum & Chemical Corp., P.O. Box 3370, Gramercy, LA 70052 USA

One of Kaiser Gramercy's products is alumina trihydrate (ATH). Certain impurities in ATH impart color to sodium aluminate solutions made from same. Kaiser Alumina Technical Services (KATS) has a project in progress to identify and remove these impurities. The approach was two-phased. First was to screen each additive by introducing each into the preparation of the sodium aluminate solution made from ultra-high purity (UHP) sodium hydroxide and UHP ATH, and measure the absorbance of the resulting solution. In this way the "color-imparting" additives could be ranked. Second, a series of cyclical digests simulating the Bayer process was performed to test each additive as it is used in the plant and observing the color imparted to the resultant liquors and products. Phase One yielded two (2) additives to be of consequence and Phase Two verified the impact of these two additives. This presentation discusses the results of this interesting study.

2:30 PM
DEVELOPMENT, PRODUCTION AND BUSINESS OF SPECIALTY ALPHA ALUMINA AT NALCO: *N. V. Badi*[1]; B. K. Satapathy[1]; S. K. Patnaik[1]; [1]National Aluminium Company, Ltd., NALCO Bhawan, P/1, Nayapalli, Bhubaneswar, Orissa 75013 India

With ever increasing demand from the industries to satisfy exacting chemical and physical properties of alumina-based chemicals, the continuous development of Chemical Grade Aluminas and Hydrates is at centre stage. Due to attractive prices, market stability and the capital investment involved in the development of these products, the technology is closely guarded by those in the business. It has been estimated that about 75-85% of the specialty alpha alumina is utilized in refractory and ceramic industries whose growth is estimated to be 10-12% during the coming years. Nalco, the largest alumina producer in India, has undertaken the development and production of a series of high quality alpha alumina products. The paper describes the development work from laboratory scale to pilot scale to produce high alpha aluminas. Several additives were tested during the course of this program. Process parameters were studied and the various products characterized by XRD, SEM, Sedigraph and BET to evaluate the variation in physical properties and product quality for the alumina produced.

3:00 PM BREAK

3:30 PM
ALUMINA TRIHYDRATE COLOR STUDY: *Marianne O. Kirkpatrick*[1]; [1]Kaiser Alumina Technical Services, P.O. Box 3370, Gramercy, LA 70052 USA

Reflectance is one of the many customer specifications for alumina trihydrate customers who grind the material for a variety of filler requirements, including flame retardants. This study was undertaken to better understand the relationship between color and reflectance. The effects of particle size distribution, trace metals, acid insolubles and caustic sediments on color and reflectance were investigated for the purpose of improving hydrate reflectance. It has long been recognized

that reflectance is a function of particle size. The study verified this fact and established a similar relationship between particle size and color. An important result of the study illustrated the impact of impurities on color. This paper presents the trends and key characteristics determined in the study.

4:00 PM

CHEMICAL PRODUCTS AND ALUMINA AS RESULT OF NON-BAUXITE RAW MATERIALS JOINT WASTEFREE: *G. Z. Nasyrov*[1]; V. V. Pivovarov[1]; S. Y. Dantzig[1]; V. A. Lipin[1]; [1]VAMI, 86 Sredny Pr, St. Petersburg 199026 Russia

From an ecological point of view, the best system to produce alumina results in the production of no waste streams. This can be accomplished when processing non-bauxite materials. The joint treatment of nepheline and alunite ores to produce alumina, potassium sulfate, cement, light weight aggregate and other materials is such a process. This paper covers the potential of utilizing alkaline alumosilicate and alunite raw material and the economics of these processes. Some flowsheets of alunite and alumosilicate processing and the techno-economic patameters of complete treatment are shown.

4:30 PM

INFLUENCE OF DIMENSIONS OF PRIMARY CRYSTALS OF CORUNDUM ON YOUNG'S MODULUS OF ALUMINA POWDER COMPACTS: *Mariusz A. Wójcik*[1]; Julian Plewa[2]; *Horst Altenburg*[2]; Andrzej Kwatera[1]; Viliam Figusch[3]; [1]Academy of Mining and Metallurgy, Faculty of Ceramics and Mats. Eng., Av. Mickiewicza 30, A-3, Cracow 30-059 Poland; [2]Fachhochschule Munster, Fachbereich Chemieingenieurwesen, Stegerwaldstrasse 39, Steinfurt, Munster D-48565 Germany; [3]Academia Istropolitana, Klariska Str.5, P.O. Box 217, Bratislava 1 81000 Slovak Republic

The influence of dimensions of primary crystals of corundum (d50) on Young's modulus (E) of alumina powder compacts was presented in this paper. Young's modulus has been determined from measurements of the propagation velocity of longitudal ultrasonic vibrations. The ultrasonic investigations allow to study the value of ununiformity and anisotropy of alumina powder compacts after grinding, pressing and sintering operations. Results shown that higher density of different starting alumina materials gave higher sound velocity as well as higher elastic constant E of alumina powder compacts.

ALUMINUM REDUCTION TECHNOLOGY: Materials Performance in Smelters

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Georges J. Kipouros, DalTech, Dalhousie University, Dept. of Mining & Met. Eng., Halifax, NS B3J2X4 Canada; Mark P. Taylor, Comalco Aluminium Limited, Brisbane, Queensland 4001 Australia

Monday PM Room: 6F
March 1, 1999 Location: Convention Center

Session Chair: Ron Barclay, Alumax, Research and Development, SC 29445 USA

2:00 PM

EROSION OF CATHODE BLOCKS IN 180 KA PREBAKE CELLS: *A. T. Tabereaux*[1]; J. H. Brown[1]; I. J. Eldridge[1]; T. R. Alcorn[1]; [1]Reynolds Metals Company, Smelter Technology Laboratory, 4276 Second St., Muscle Shoals, AL 35661 USA

Changes in the top surface level of cathode blocks due to erosion have been measured in 180 kA prebake cells for 3 consecutive years in order to determine the erosion rates for eight different types of cathode blocks. The results of the test program indicate significantly high, linear erosion rates for 100% graphite blocks with the rate slowing after 1600 days. Cathode blocks containing 30% graphite erode at a substantially lower rate. Erosion depths have been found to vary greatly within cathodes, and significantly higher erosion rates may occur at different locations under the same anode. These complex erosion patterns indicate that a more complicated, interrelated mechanism is involved than the simplistic abrasion-based mechanism suggested to date by researchers.

2:40 PM

INTERACTIONS OF ALKALI METAL WITH CATHODE CARBON: *T. Naas*[2]; H. A. Oye[1]; [1]The Norwegian University of Sciences and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway; [2]Norsk Hydro, A/S Research Centre, P.O. Box 2560,N-3901 Prosgrun, Norway

The effects on the performance of cathode carbons when LiF and KF are added to the electrolyte have been studied by laboratory scale electrolysis experiments. Alkali metal expansion (Rapoport test) data is reported for a semigraphitic cathode material electrolysed in an acidic bath ($SnMF/n_{ALF3}$ = 2.2; M = Li, Na, K) with up to 20 mol% LiF or KF from 870°C to 970°C. At 970°C, the cathode material expands 0.28%±0.03% in the reference melt without additions of LiF or KF. Substitution of 20 mol% LiF (»10 wt.%) for NaF reduces the expansion to 0.20%. A reduction of the electrolysis temperature causes a slight increase in the cathode expansion in LiF-modified melts. Addition of up to 5% KF does not seem to affect the cathode expansion, while larger additions give a moderate increase in the cathode expansion at 970°C. With 20 mol% (»20 wt.%) KF substituted for NaF, the expansion is doubled (0.60%). With 20 mol% KF at 870°C, however, the expansion is 5% and the cathode sample is cracked throughout. Trends similar to those found by alkali metal expansion measurements were also found by measuring the compressive strength of cathode samples in situ immediately after lab scale electrolysis.

3:05 PM

EROSION MECHANISMS IN SMELTERS EQUIPPED WITH GRAPHITE BLOCKS: A MATHEMATICAL MODELLING APPROACH: *J. M. Dreyfus*[1]; L. Joncourt[1]; [1]Carbone Savoie, R/D, 30 Rue Louis Jouvet, BP 16, Venissieux, Rhône 69631 France

Erosion patterns in cells, obtained from measurements made during operation and from autopsy observations, show a strong correlation with the current density distribution. Moreover, the speed of erosion increases with the current density. A 3D numerical model was developed to assess the current density distribution at the cathode-metal interface. The influence of the cathode-bar assembly characteristics on the current flow and on the voltage drop was investigated. These studies allow us to simulate the effects of the geometry, the contact resistance, the cathode block properties and the ledge. Calculations with eroded cathode allow to understand the behavior of the pot under aging. This calculations quantify the physical phenomenon of the current flow in the three geometric dimensions. A comparison of the model with experimental observations in cells equipped with graphite and graphitic cathodes provides an improved understanding of erosion mechanisms and allows us to propose alternative graphite cathode blocks.

3:30 PM

EXPERIMENTAL INVESTIGATION OF FROST HEAVING IN ALUMINUM REDUCTION CELLS USING A WATER MODEL: P. Pelletier[1]; *C. Allaire*[1]; [1]Ecole Polytechnique, CRIQ Campus, 8475 Christoph, Colomb St., Centreville, Montreal, Quebec H2M 2N9 Canada

In aluminum reduction cells, cathode blocks heaving is known to reduce cells' life. Many phenomena can cause cathode blocks heaving, one of them could be frost heaving. However, such a phenomenon, which is related to melt solidification in porous materials, has never been experimentally verified. In the present work, a water model is proposed based on melt solidification from highly concentrated aqueous salt solutions. Results obtained from the solidification of such solutions into alumina powder, under different cooling conditions and mechanical restrictions, are presented. It is suggested, based on these results, that frost heaving in a cell could intervene at temperatures above the melt solidus, and is more likely to occur in the bedding mix.

3:55 PM BREAK

4:15 PM
CHEMICAL RESISTANCE OF SIDELINING MATERIALS BASED ON SiC AND CARBON IN CRYOLITIC MELTS, A LABORATORY STUDY: *E. Skybakmoen*[1]; H. Gudbrandsen[1]; L. I. Stoen[1]; [1]SINTEF Materials Technology, Electrolysis Group, Process Metallurgy and Ceramics, Trondheim 7034 Norway

A polarised test cell was developed, in order to simulate the extremely corrosive conditions prevailing in industrial aluminium cells when no sideledge is present. The test cell allowed for simultaneous testing of four different materials, and the materials were exposed to cryolitic melt, its vapour (mainly $NaA1F_4$), CO_2/CO from the anode, and liquid aluminium for up to 120 h. The chemical resistance of pure silicon carbide, silicon nitride bonded silicon carbide, as well as different carbon qualities, was investigated. The chemical resistance of SiC - based materials depended on the type of material, the amount of CO_2 evolved at the anode, and the time of exposure. The oxidation resistance of SiC - based materials was tested in flowing air (950ϒC, 100 h), The chemical degradation mechanisms for SiC and carbon, as well as the correlation between oxidation resistance and chemical resistance for SiC - based materials, are discussed.

4:40 PM
FILLING ALUMINA SILOS BY USE OF ANTI SEGREGATION TUBES (ASTS): *M. Karlsen*[1]; A. Dyroy[2]; H. Kvande[3]; [1]Hydro Aluminium Metal Products, Technology Centre Ardal (R&D), Ovre Ardal N-5870 Norway; [2]Telemark Technological and Development Center, Dept. of POSTEC, Kjones Ring, Porsgrunn N-3914 Norway; [3]Hydro Aluminium Metal Products, N-1321, Stabekk Norway

Point feeding of alumina is common in modern alumina reduction cells. It is most often based on volumetrically measured alumina dumps, and such systems are vulnerable to the effects of segregation. In order to reduce the problem of alumina segregation during filling of silos, a device called an Anti Segregation Tube (AST) has been developed. With this device the alumina is filled into the tube instead of directly into the silo, which leads to air induced segregation. The AST has a specially designed inlet and is equipped with valves along its side walls. When multiple ASTs are used, it is essential that the tubes are arranged in a pattern which ensures that the alumina avalanche moves away from the discharging AST and into the heap formed by its neighbour. Using ASTs, the fines are distributed evenly in the silo, and hence they will be discharged likewise. Full scale tests have demonstrated a reduction in the coefficient of variation, COV (the standard deviation divided by the average value) of 54% in the minus 42 μm fraction after emptying of a secondary alumina silo. The coefficient of variation for the amount of fines fed to the electrolysis cells was then reduced by nearly 30%.

5:05 PM
CATHODE REFRACTORY MATERIALS FOR ALUMINIUM REDUCTION CELLS: *C. Schoning*[1]; T. Grande[2]; O.J. Siljan[3]; [1]SINTEF Materials Technology, N7034, Trondheim Norway; [2]Department of Inorganic Chemistry, Norwegian University of Science and Technology, N-7034, Trondheim, Norway; [3]Norsk Hydro ASA, Research Centre Porsgrunn, Norway

The paper presents a "state of the art" overview on dense bottom lining refractory materials in aluminum electrolysis cells. The performance of both traditional refractory linings and recently introduced dry barrier powder materials is summarized. The results and conclusions presented in the paper are based on evaluations of both physical and chemical properties of the refractory materials, and the influence of these properties on material performance in the cell. Special attention is given to the importance of the silica content in the refractories. The paper also presents the considerable progress in the understanding of the deterioration of the cathode lining materials during operation, which have been obtained during the last decade in the author's laboratory in collaboration with the Norwegian aluminum industry The improved understanding is based upon phase diagram studies; studies of melt properties, as well as field research through autopsies of shut down cells.

ANALYTICAL TECHNOLOGY IN THE MINERAL INDUSTRIES: Sampling and Classical Methods for Mineral Analysis

Sponsored by: Extraction & Processing Division, Process Mineralogy Committee; ASTM Subcommittee E01.02
Program Organizers: Louis J. Cabri, CANMET, Ottawa, Ontario K1A 0G1 Canada; Charles H. Bucknam, Newmont Metallurgical Services, Englewood, CO 80112 USA; Steven L. Chryssoulis, Amtel, London, Ontario N6G 4X8 Canada; Rebecca A. Miller, Minekeepers, Phoenix, AZ 85014 USA; Emil Milosavljevic, Lakewood, CO 80227 USA

Monday PM Room: 7A
March 1, 1999 Location: Convention Center

Session Chairs: Charles H. Bucknam, Newmont Metallurgical Services, Analytical Dept., Englewood, CO 80112 USA; Dr. Steve McCann, Alfred H. Knight, Spartanburg, SC 29304 USA

2:00 PM INTRODUCTION TO SESSION CHARLES H. BUCKNAM - SESSION CHAIRMAN

2:05 PM INTRODUCTION FOR SAMPLING PAPERS DR. STEVE MCCANN - CO-CHAIRMAN

2:10 PM
APPLICATION OF SIMPLIFIED QUANTITATIVE RISK ANALYSIS (SQRA) TO JUSTIFY OPTIMIZATION OF BLAST HOLE SAMPLING, SAMPLE PREPARATION AND ASSAYING FOR GOLD ORE CONTROL: *Charles H. Bucknam*[1]; [1]Newmont Metallurgical Services, Analytical, 10101 East Dry Creek Rd., Englewood, CO 80112 USA

A gold sampling optimization study was carried out in support of open pit gold mining operations for the Newmont Gold Company Bootstrap/Capstone deposit in Northeastern Nevada. Six bulk samples were tested using fire refining techniques at field particle size, to determine coarse particle heterogeneity of the ores, and by gravity concentration methods at minus 10 mesh particle size, to observe any particulate gold liberation. An overall sampling model is presented for the deposit based on the pooled test information. The sampling model is used to recommend procedures to control the variability of gold grade estimation due to the fundamental error (nugget effect) during sampling, sample preparation and assaying of the ores. The technique of simplified quantitative risk analysis (SQRA) is used to provide an economic justification for implementation of recommended improvements in field sampling practices.

2:30 PM INVITED PAPER
SAMPLING ERROR IN BLAST HOLE DRILLING AND WHAT CAN BE DONE TO IMPROVE SAMPLE QUALITY: *Harrison Cooper*[1]; Stephen Pack[2]; [1]Harrison R. Cooper Systems, Inc., 106 West Second North, Bountiful, UT 84010 USA; [2]Mining Consultant, Nykopingsvagen 31, Nykoping 611 50 Sweden

Data from samples taken during rotary air blast and reverse circulation drilling are crucial to properly assessing economics of ore deposits in the case of exploratory work, and are equally critical in accurately delineating ore and waste in execution of mine operation plans. Past studies of sampling procedures, comparing results of conventional splitting to precise extraction of representative samples, illustrate the significant degree of error frequently encountered. Economic impact of error in mine blast hole sampling work can be shown to result in theoretical losses valued at millions of dollars annually in operating large scale mining projects. Results of past drill sampling studies are reviewed. Drill sampling technique can be improved by collecting coarse and fine

particles together and extracting samples for assay from the complete flow of rock and dust simultaneously. This can be accomplished with a collection unit attached to the drill stem through which all except a small proportion of extreme fines are retained. The bulk mass can be static sampled as flow takes place through the collector when size reduction is not required. Alternately, collected mass with fines and coarse intermingled can be processed for increased accuracy through size reduction to obtain proportional samples.

2:50 PM INVITED PAPER

SAMPLING AND ANALYSIS OF COPPER ALLOY SLAGS REFINED IN A SUBMERGED ELECTRIC ARC FURNACE: *Jeffrey C. Morrow*[1]; [1]Colonial Metals Company, Non-Ferrous Metals, 217 Linden St., P.O. Box 311, Columbia, PA 17512-0311 USA

When copper alloys are melted, a slag containing oxides and metals is produced. As a co-product, these slags contain value which can be reclaimed through processing. As an end product, those produced by the melting of leaded alloys will fail the Toxicity Characteristic Leaching Procedure (TCLP) for lead, rendering them a toxic material in need of disposal. In the past, the furnace slags produced were sold to companies that recovered the value contained in these low grade materials. Today, you will find many of these metal recovery operations closed, unable to comply with current environmental regulations or already listed as a superfund site. Colonial Metals Company recognized this trend and in 1997 began the construction of a Submerged Electrical Arc Furnace (SEAF) to process the slags produced by our melting furnaces. The goal of the SEAF is to recover the metal content trapped in the slag and render the resultant slag nontoxic as defined by the TCLP. In order to maximize the efficiency of this process, chemical analysis is needed on the feed material and for melt control. In order to perform a slag analysis, the sample submitted to the lab must be representative of a relatively nonhomogenous material. Representative sampling of slag is critical to our SEAF process.

3:10 PM BREAK

3:40 PM INTRODUCTION FOR CLASSICAL METHODS PAPERS CHARLES H. BUCKNAM - SESSION CHAIRMAN

3:45 PM INVITED PAPER

THE CLASSICAL FIRE ASSAY: CURRENT STATE OF PRACTICE: *Joel A. Huffman*[1]; [1]Commercial Testing & Engineering Company, 5906 McIntire St., Bldg. #4, Golden, CO 80239 USA

Overview of the classical method of fire assay based on over twenty years of assay experience with exploration, metallurgical and settlement samples. Proper laboratory design, production and safety considerations used in laboratories designed in the U.S., Chile, Peru and Former Soviet Union. Brief comments on non-fire assayable gold and so called "magic fluxes" and special assay procedures.

4:05 PM INVITED PAPER

EFFECT OF ZINC IN CYANIDE HEAP LEACH PROCESSING SOLUTIONS: J. S. McPartland[1]; *John W. Langhans*[1]; [1]McClelland Laboratories, Inc., 1016 Greg St., Sparks, NV 89431 USA

The presence of zinc in cyanide solutions is generally thought to not have an adverse effect on precious metal recovery or recovery rate by heap leach processing. However, analytical interferences of zinc in solution can cause problems in accurately determining and controlling cyanide concentration in the leach solution, leading to increased cyanide consumption. Analytical methods and the chemistry of cyanide in the presence of zinc are reviewed. The effects of solution pH and zinc concentration on the free cyanide determination were evaluated in detail. Use of the various analytical techniques for controlling cyanide concentration during heap leaching were investigated experimentally. The practical and economic implications of cyanide heap leaching in the presence of cyanide soluble zinc minerals are discussed.

4:25 PM INVITED PAPER

PRELIMINARY ANALYSIS AND METHOD OPTIMIZATION BASED ON X-RAY FLUORESCENCE - APPLICATION TO PRECIOUS METALS: *Arnold M. Savolainen*[1]; David J. Kinneberg[1]; Barbara E. Mangion[1]; [1]Metalor USA Refining Corporation, P.O. Box 255, North Attleboro, MA 02761 USA

Analytical laboratories require a qualitative or semi-qualitative method of determining major and minor constituents in "unknown" samples to select optimal analytical procedures. This is exceedingly important in precious metal assay laboratories where the precision and accuracy requirements are exacting (routinely within 0.01%). This paper describes the use of an x-ray fluorescence spectrometer with a specially designed software package to select optimal analytical procedures and specify relevant analytical parameters. A wave-length dispersive XRF spectrometer is capable of quick and accurate analyses of properly prepared samples without extensive standardization. When coupled with a specially designed software package, this preliminary assay system is applicable to a wide variety of sample types including gold and silver bullion, karat gold and karat PGM alloys, high silver and high gold metallic samples as well as particulate samples of ores and sweeps. The system relieves the assayer of tedious calculations for silver inquartation and proofing, minimizes human error and standardizes selections over time. Most importantly, the system allows for independent verification of final assay results.

4:45 PM INVITED PAPER

APPLYING SIMULTANEOUS DSC/DTA-TG FOR HIGH TEMPERATURE CHARACTERIZATION OF MINERALS: *Bob Fidler*[1]; Jack Henderson[2]; E. Post[2]; J. Blumm[2]; [1]Netzsch Instruments, Inc., Thermal Analysis Division, P.O. Box 995, Huntersville, NC 28070-0995 USA; [2]NETZSCH-Geraetebau GmbH, P.O. Box 1460, Selb, Bavaria D-95088 Germany

Simultaneous thermal analysis (DSC/DTA-TG) of minerals and other inorganic materials can provide decisive data as to composition and suitability for processing. Using a high accuracy microbalance integrated directly with a DSC or DTA sensor can provide the ability to measure phases changes with their corresponding mass change and heat flow behavior. For example, during analysis of a complex mixture of aluminum oxide, quartz sand, and calcium carbonate, it is possible to measure mass change resulting from small amounts of CaCO3 as well as comparison of the phase transition enthalpy of the SiO2 component with the enthalpy of quartz sand, confirming the weight component of sand in the mixture. Over 1000°C, the measurement of the sintering behavior of the mixture is possible via analysis of the exothermal release of energy. The paper will examine various aspects of thermal analysis techniques applied to minerals and inorganic materials.

5:05 PM QUESTIONS

AUTOMOTIVE ALLOYS III: Session II — Fundamental Studies

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh Das, ARCO Aluminum Company, P.O. Box 32860, Louisville, KY 40232 USA

Monday PM Room: 3
March 1, 1999 Location: Convention Center

Session Chairs: Subodh K Das, ARCO Aluminum, Inc., Louisville, KY 40232 USA; Dr. Subi Dinde, Chrysler Corporation, Madison Hts., MI 48071 USA

2:00 PM

THE EFFECT OF POROSITY SIZE AND DISTRIBUTION ON THE FATIGUE PROPERTIES IN CAST 319 Al: *J. M. Boileau*[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Research Laboratories, MD 3182 SRL, Dearborn, MI 48121-2053 USA

As the automotive industry increases its use of cast aluminum components, the need for more detailed information relating the effect of casting practice on fatigue behavior also increases. One of the key factors influencing the fatigue of cast aluminum is porosity. Therefore,

a study characterizing the influence of solidification time on the microstructure and fatigue properties in a cast 319 Al alloy was conducted. Multiple fatigue tests were conducted on a cast 319 Al alloy (T6 and T7 heat-treatments) at selected stress levels so that valid statistical comparisons could be made. Extensive metallographic and fractographic characterization was performed to understand the influence of pore size and distribution on fatigue life. In general, microporosity was associated with all of the fatigue failures and was located at or near the specimen surface. Also, as solidification time increased, the average initiating pore diameter increased and the number of samples having multiple initiating sites tended to increase. Multiple initiating sites were observed in several samples and were observed to have an effect on the fatigue. Quantitative measurements of microporosity found that conventional metallographic techniques substantially underreport the maximum pore size present in the W319 alloy.

2:20 PM

DUCTILITY AND FORMABILITY OF AUTOMOTIVE Al ALLOY SHEET: *T. R. G. Kutty*[1]; [1]Canada

The sheet forming behaviour of a range of automotive Al alloys is known to be degraded by the presence of iron-based intermetallic phases. We have therefore studied two alloys, AA6111 and AA7574, for which experimental heats have been made containing a wide range of iron contents (0.06 - 0.7 wt%). The mechanical behaviour of these has been investigated experimentally using a combination of tensile testing (using both uniform and notched specimens) and bending tests. The addition of Fe has a complex effect due to the interplay between inclusion content and grain size. Moreover, the inclusion phases is damage-resistant in that very few damaged particles are seen prior to the onset of necking. However, tensile instability is followed rapidly by fracture. This presentation will discuss the role of inclusion content of ductility in a range of stress states.

2:40 PM

EFFECTS OF Si AND Mn ON THE AGEING BEHAVIOUR AND FORMABILITY OF ALLOYS BASED ON AA6016A: *S. M. Hirth*[1]; [1]Alcan International, Ltd., Banbury Laboratories, Southam Rd., Banbury, Oxon OX16 7SP England

The heat treatable 6XXX series (Al-Mg-Si-(Cu) aluminum alloys are finding increasing use in automotive skin panel applications where relatively high formability and in-service strength for dent resistance are major requirements. In Europe, the alloy of choice for such applications is currently the low Cu-containing alloy AA6016A, which derives its strength from the precipitation hardening phase, Mg2Si. The volume fraction of Mg2Si is, in turn, affected primarily through the level of Mg within the alloy, although the Si content is also important. The level of Si within the alloy influences the solution heat treated (T4) strength and the subsequent ageing response of the 6XXX series alloys, again predominantly through its effect on the volume fraction of Mg2 Si. In this paper, the effects of Si content on the ageing behaviour and mechanical properties (formability) of alloys based on the AA6016A composition, containing 0.4 wt.% Mg, are described. In addition the effect of Mn on the microstructure and mechanical properties of alloys based on the AA6016A composition is discussed. In this respect, Mn is often added to the 6XXX series alloys to provide grain size control, but it is shown that it may also provide a component of solid solution strengthening and affect general formability.

3:00 PM

MICROSTRUCTURAL STRENGTHENING IN ALUMINUM AUTOMOTIVE BUMPER ALLOYS: *Jan Anders Saeter*[1]; Jin Huang[1]; Grethe Waterloo[2]; Warren James Poole[1]; [1]The University of British Columbia, Dept. of Metals and Mats. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Hydro-Raufoss Aluminium, Hydro Raufoss Automotive Research Centre, P.O. Box 41, Raufoss N-2631 Norway

The purpose of the present paper is to elucidate on the strengthening mechanisms of relevance for aluminum alloys used in automotive bumpers. These bumpers are manufactured by stretch bending of 7000 series aluminum alloys, followed by age-hardening. The final strength of the bumper arises from a contribution of work hardening and precipitation strengthening. An important aspect of the stretch bending process is that the level of deformation varies spatially in the bumper. This will influence on the local strength contribution from work hardening but also the age hardening process, as dislocations may act as heterogeneous nucleation sites for precipitates and accelerate the growth/coarsening of precipitates due to pipe diffusion. In the present work, a 7108-alloy and a 7030-alloy have been predeformed in the strain range of 0 - 1.2, followed by conventional two-step age hardening. For the various predeformations, the strength has been measured as function of ageing time by conducting tensile tests and also by monitoring changes in electrical resistivity. Observations from TEM demonstrate that variations in dislocation structure and particle sizes when the processing conditions are changed. The experimental results will be interpreted in terms of the two hardening mechanisms mentioned above.

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THE ROLE OF NATURAL AGING ON SUBSEQUENT PRECIPITATION DURING THE ARTIFICIAL AGING IN AA6111 ALUMINUM ALLOY: Shahrzad Esmaeili[1]; Jan Anders Saeter[1]; *Warren James Poole*[1]; David J. Lloyd[2]; [1]The University of British Columbia, Dept. of Metals and Mats. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International, Kingston Research and Development Centre, P.O. Box 8400, Kingston, Ontario K7L 5L9 Canada

The aging behaviour of AA6111 aluminum alloy has been studied using tensile testing and electrical resistivity measurements. This alloy is of particular interest due to its increasing use in automotive sheet applications in North America. In these applications the aluminum sheet is usually formed in the solution treated and naturally aged T4 condition. The final strength of the component is attained during the paint bake cycle which is usually simulated by 30 to 60 minutes at 180ϒC in laboratory experiments. The final yield strength obtained from this processing route is substantially lower than the amount that would be obtained if the as-quenched supersaturated solid solution had been immediately aged at 180ϒC. This difference is due to the formation of solute-vacancy clusters, i.e. initial zones, during the natural aging period which affects the subsequent precipitation processes during artificial aging of the alloy. In the present investigation, tensile tests were performed at different stages of natural aging and artificial aging at both room temperature and -196ϒC. The natural aging response of the alloy and its effect on the strength after artificial aging at 180ϒC were determined. By examining the temperature dependence of the yield stress as a function of artificial aging time it was possible to obtain information regarding the evolution in the nature of the obstacles to dislocation motion. Further information on the evolution in structure was obtained from electrical resistivity measurements at -196°C taken during both natural aging and artificial aging. These results in conjunction transmission electron microscopy and differential scanning calorimetry results can be combined to develop an understanding of the precipitation processes which occur and the age hardening response of the alloy.

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4:00 PM

A PROCESS MODEL FOR THE AGE HARDENING OF A 319-TYPE ALUMINUM ALLOY: *Carla A. Cloutier*[1]; Paula M. Reeber[1]; J. Wayne Jones[2]; [1]Ford Motor Company/University of Michigan, Dept. of Mats. Sci. and Eng., MD 3182 SRL, P.O. Box 2053, Dearborn, MI 48121-2053 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI USA

The age hardening response of a 319-type aluminum alloy was studied by examining the variation of yield strength and proportional limit with aging time and temperature. Aging curves were constructed for cast materials produced by two different solidification rates. Aging temperatures ranged from 130-305ϒC for periods up to 1000 hours. The aging curves follow conventional diffusion controlled precipitation hardening behavior. Using this data, a process model was developed that is based on an approach suggested by Shercliff and Ashby [H. R. Shercliff and M. F. Ashby, Acta metall. mater 38, 1789 (1990)]. The process model predicts the changes in yield strength and proportional limit that result from isothermal aging. The components of the model are outlined, and the deviation of measured behavior from that predicted by the modified Shercliff-Ashby model is discussed for both solidification rates. The results show that the aging process of 319 can be reasonably predicted by

the age-hardening process model. A variant of the model describing the effects of thermal exposure on aging behavior will also be discussed.

4:20 PM
OBSERVATION OF THROUGH-THICKNESS DEFORMATION BANDS IN AN AL 6111 ALLOY DEFORMED IN PLANE STRAIN DEFORMATION: *Paul Seungyong Lee*[1]; G. Jarvis[1]; A. D. Rollett[1]; H. R. Piehler[1]; B. L. Adams[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In the past century, efforts have been made to quantify through-thickness deformation bands, but only the local view of sample scale through-thickness deformation bands was obtained by unreproducible chemical etching techniques. Acquisition of sample scale bands in complete form with the indication of local deformation and orientation information was infeasible. A recently developed technique, orientation imaging microscopy (OIM), was applied to the characterization of through-thickness deformation bands. 6111-T4 commercial aluminum alloy was deformed in plane strain tension, and the strain inhomogeneity in the cross-section along with texture inhomogeneity was observed. The wavelength and orientation of the bands were quantified by calculating the power spectral density and the auto-correlation function of the image quality maps measured by OIM.

4:40 PM
TEXTURE EVOLUTION OF STRIP CAST AA5754 ALUMINUM ALLOYS DURING ANNEALING AND COLD ROLLING: *Y. Liu*[1]; Y. L. Liu[1]; J. Qui[1]; G. Liao[1]; J.G. Morris[1]; [1]University of Kentucky, Dept. of Chem. and Matls. Eng., Light Metals Research Laboratories, 177 Anderson Hall, Lexington, KY 40506 USA

AA5754 aluminum alloy is considered for application in the automobile industry. Understanding the texture evolution of the alloy is important in order to control its formability. In the present work, annealing textures of the hot band are determined by the electron back scattering pattern (EBSP) technique. A new concept, texture continuity, which describes the distribution of grains with similar orientations in the two dimension microstructure is proposed. Cold rolling textures of the alloy are measured by the X-ray pole figure method. The relationship between texture, texture continuity and mechanical properties of the material is discussed.

5:00 PM
MICROSTRUCTURAL EVOLUTION OF AN Al-Si-Cu-Mg ALLOY DURING SOLIDIFICATION: *Jacob W. Zindel*[1]; Jon W. Hangas[1]; William T. Donlon[1]; Larry A. Godlewski[1]; [1]Ford Motor Company, Ford Research Laboratory, MD3182 SRL, P.O. Box 2053, Dearborn, MI 48121-2053 USA

Al-Si-Cu-Mg alloys, commonly referred to as 319, are widely used for the production of castings for automobile powertrain components. Micromodels are being developed to predict as-cast microstructures since these microstructures control the subsequent mechanical and physical properties of the casting. In order to provide input data and to verify the micromodels, a quenching technique was developed to characterize the microstructural evolution during solidification. The quenching technique consists of taking samples of the molten alloy from a furnace and controlling the solidification time to be consistent with industrial casting processes. At various temperatures during solidification, the partially solidified samples were quenched. Solid phases at the time of the quench were characterized by optical metallography and electron microprobe analysis. The dendrite arms were observed to coarsen rapidly from the liquidus temperature to the Al-Si eutectic temperature. The Cu concentration of the dendrites was found to remain constant while the Si concentration increased as the dendrites coarsened during solidification. Precipitation of three Fe containing phases were also observed prior to the Al-Si eutectic reaction.

5:15 PM
IMPROVEMENT OF HOT DUCTILITY OF Al-Mg BASE ALLOYS BY SMALL AMOUNTS OF ADDITIONAL ELEMENTS: *Keitaro Horikawa*[1]; Shigeru Kuramoto[1]; Motohiro Kanno[1]; [1]The University of Tokyo, School of Eng., Dept. of Matls. Sci., 7-3-1, Hongo, Bunkyo-ku, Tokyo 113 Japan

An Al-5.5mol%Mg alloy containing traces of sodium, calcium or strontium as impurities shows high temperature embrittlement based on intergranular fracture at around 300°C. The authors have also reported that high temperature embrittlement disappears when sodium content is lowered to 0.01ppm. However, it is considered to be difficult to reduce those impurity contents to below 1ppm for practical purposes. In the present study, effect of small amounts of additional elements on hot ductility of an Al-Mg alloy containing traces of sodium or strontium was examined at elevated temperatures. Al-5.5%Mg-2molppmNa alloys with and without additional antimony and Al-5.5mol%Mg-2molppmSr alloys with and without additional silicon were melted and cast in argon. Aluminum of 99.999% purity and magnesium of 99.98% purity were used. A high purity graphite crucible was used to avoid impurity contamination. These ingots were homogenized and cold-swaged by 70%. Round tensile test pieces were machined from the swaged rods and annealed at 510°C. Tensile tests were made at temperatures ranging from R.T. to 400°C and at a strain rate of 8.3x10-4 s-1. Polished surfaces of the specimens were observed with an optical microscope and analyzedby an energy dispersive X-ray spectroscope. Fracture surfaces of these specimens were observed with a scanning electron microscope. High temperature embrittlement caused by sodium of 2ppm is suppressed by the addition of antimony of more than 2ppm, and that by strontium of 2ppm is suppressed by the addition of 940ppm silicon. The Al-Mg-Na and Al-Mg-Sr alloy showed almost intergranular fracture surface at 300°C, while the Al-Mg-Na alloy containing 2ppm antimony and the Al-Mg-Sr alloy containing 940ppm silicon transgranular one. Energy dispersive X-ray analysis revealed that Na-Sb compounds were formed in the Al-Mg-Na-Sb alloy, and Si-bearing compounds trap strontium in the Al-Mg-Sr-Si alloy. Thus, it is concluded that antimony and silicon can scavenge sodium and strontium from grain boundaries through the formation of compounds, respectively.

CARBON TECHNOLOGY: Anode Quality & Reactivity

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: C. Dreyer, Aluminium Pechiney, St. Jean De Maurienne 73303 France

Monday PM — Room: 6D
March 1, 1999 — Location: Convention Center

Session Chair: Jaffer Ghuloom Ameeri, Aluminium Bahrain, Manama, Bahrain

2:00 PM SESSION CHAIRMAN INTRODUCTION

2:05 PM
DUST GENERATION AND ACCUMULATION FOR CHANGING ANODE QUALITY IN CELLS WITH DIFFERENT DESIGNS: *Raymond C. Perruchoud*[1]; Kirstine Hulse[1]; Werner K. Fischer[1]; Wolfgang Schmidt-Hatting[2]; Ueli Heinzmann[3]; [1]R&D Carbon, Ltd., P.O. Box 362, Sierre CH-3960 Switzerland; [2]International Standardization Switzerland; [3]Alusisse, Technology & Management, Ltd. Switzerland

The amount of dust that is general by the anodes in the cells is estimated by using a models taking into account. - the synergetic effect of the anode permeability and reactivity - the enhancement of the CO2 burn near the end of the anode life time by previous air burn on the anode top - the exponential increase of dusting propensity close to the end of the anode cycle - the impact of the current density and anode cycle days as well as the anode density. The model validated in various smelters using different technologies.

2:30 PM
ASSESSMENT OF EFFECTS OF SODIUM IN HALL-HEROULT CELL ANODES: *David L. Belitskus*[1]; H. A. Simon[2]; E. F. Bart[3]; [1]Carbon Consultant, 2361 Meadow Rd., New Kensington, PA 15068 USA;

[2]Allied Signal, Inc., Carbon Mats. & Tech., 1104 Radford Dr., Russell, KY 41169 USA; [3]Allied Signal, Inc., Carbon Material & Technologies, 101, Columbia Rd., Morristown, NJ 07962 USA

Sodium has long been recognized as a catalyst for reactions of carbon with air and with carbon dioxide. As Hall-Heroult cell anode technology becomes more sophisticated, considerable scrutiny has been placed on effects of anode sodium content on on excess anode consumption. Numerous technical papers have addressed this issue, with variations in anode sodium content caused by different levels in binder pitch, calcined petroleum coke, and recycled anode butts, by additions of sodium compounds to the paste prior to anode production, or by way of impregnation of baked anodes with dissolved sodium compounds. This paper reviews, compares, and contrasts various results to provide a more comprehensive understanding of the effects of sodium origins and levels in anodes.

2:55 PM

THE EFFECT OF SODIUM ON BINDER COKE REACTIVITY: *Trygve Eidet*[1]; Morten Sorlie[1]; Stein Yngve Larsen[2]; [1]Elkem ASA, Research, P.O. Box. 8040 Vagsbygd, Kristiansand N-4602 Norway; [2]Norwegian University of Science and Technology, Dept. of Inorganic Chem., Trondheim N-7034 Norway

Coal tar pitch used as a binder in carbon anodes for aluminium electrolysis, contains sodium impurities. In pitch production, Na is often added to the coal tar feed as Na2CO3 or NaOH to neutralise the HCI content, and thereby reduce the still corrosion. Sodium is a strong catalyst to airburn and carboxy reactivity, but it has been claimed that only the excess caustic content in the resulting binder coke, due to overtitration of the HCI, significantly increase the reactivity. To investigate this more closely, and to exclude effects of other impurities, binder cokes were made from clean precursors with controlled additions of Na2CO3, or NaCI in high pressure laboratory coker. The effects from the dopants on air carboxy reactivity were measured, end surface investigations were performed.

3:20 PM

PREFERENTIAL OXIDATION PROCESS IN CARBON AND SILICON CARBIDE USED IN HALL HEROULT CELLS: *J. A. Sekhar*[1]; J. Liu[1]; V. de Nora[2]; [1]University of Cincinnati, Dept. of Mats. Sci. & Eng., International Center for Micropyretics, P.O. Box 210012, Cincinnati, OH 45221 USA; [2]Moltech S. A., 9 Route de Troinex, 1227 Carouge, Geneva Switzerland

The poor oxidation resistance of the carbonaceous materials in the Hall-Heroult cell leads to an increase in the cost of aluminum production. During the oxidation process, a preferential or selective oxidation of the binders phase is noted for all pitch bonded carbonaceous materials. This preferential oxidation is related to the transformed pitch morphology and structure. The preferential oxidation also results in accelerated strength loss as the oxidation progresses. The poor oxidation resistance of pitch bonded carbonaceous materials may be improved by special treatments which are discussed. Conversely, inadequate oxidation prevention treatments may increase the preferential oxidation and result in early failures. Similar preferential oxidation phenomena also occurs in bonded silicon carbide which is also discussed in the article.

CAST SHOP TECHNOLOGY: Molten Metal Processing/Grain Refining I

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Y. Sahai, The Ohio State University, Dept. of Mats. Sci. and Eng., Columbus, Ohio 43210-1179 USA; James O'Donnell, Commonwealth Aluminum, Dept. of Eng., Louisville, KY 40202 USA

Monday PM Room: 6C
March 1, 1999 Location: Convention Center

Session Chair: Joseph Tessandori, Commonwealth Aluminum Corp., Lewisport, KY 423541 USA

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THE GRAIN REFINEMENT OF Al7SiMg ALLOYS WITH BORON CONTAINING REFINERS: *John Anthony Spittle*[1]; Jennifer M. Keeble[1]; [1]University of Wales Swansea, IRC in Materials for High Performance Applications, Singleton Park, Swansea SA2 8PP UK

Considerable confusion exists in the aluminium foundry industry regarding the type and level of refiner addition that is most suitable for the refinement of primary crystals in hypoeutectic Al-Si alloys. Factors causing this confusion include the poisoning effect of Si when using traditional AlTiB refiners i.e. Ti: B ratios greater than the stoichiometric 2.2: 1 TiB2 ratio, reports that AlTiB refiners with sub-stoichiometric ratios or binary AlB refiners are superior, residual Ti levels in secondary alloys, casting cooling rate effects and nucleant settling with melt holding. Grain refinement of a binary Al7Si alloy has been studied, using commercial/non commercial refiners to produce varying Ti: B ratios, at two boron addition levels 0.006 and 0.02% and as a function of holding time. An influence of the Ti: B ratio is demonstrated at the low B level. The concentration, type, size and density of the nucleants determine the influence of melt holding time on refinement.

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GRAIN REFINING OF Al-7wt%Si ALLOYS: *Christian J. Simensen*[1]; [1]SINTEF Material Technology, Forskningsveien 1, P.O. Box 124, Blindern, Oslo N-0314 Norway

This is an investigation of a series of Al-7wt%Si alloys cast with a cooling rate of 1*C/s. Different amount of Al-1.6wt%Ti-1.6wt% B and Al-10wt%Ti rods were added to the melt making alloys in the range 0.01-0.l8wt%Ti and 0.003-0.01wt%B. The cooling curves showed that initially the grains started to grow at a certain undercooling. Then they grew with a nearly constant temperature. This growth temperature increased with increasing amount of titanium in the melt: T growth =613.2*C + 30.2wt%Ti. The grain size of the alloys was reduced from about 0.2mm to 250 m when the content of titanium increased from 0.01 wt% to 0.12wt%. The amount of boron (borides) was kept constant in these experiments. The best results were obtained when Al3(Ti,Si) were nucleated on TiB2 -particles during cooling. Then aluminium grains are formed on the Al3(Ti,Si)-particles yielding the fine-grained material.

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THE EFFECTS OF GROWTH RESTRICTION AND EFFECTIVE NUCLEANT POTENCY ON GRAIN SIZE AND MORPHOLOGY IN Al-Si AND Al-Cu ALLOYS: James E. C. Hutt[1]; Young C. Lee[1]; *Arne K. Dahle*[1]; David H. St.John[1]; [1]The University of Queensland, Dept. of Mining, Minerals and Mats. Eng., CRC for Alloy and Solidification Technology, Brisbane, Qld 4072 Australia

The effects of increased solute content on dendrite morphology and grain size in both Al-Si and Al-Cu alloys have been investigated. The results show that a morphological transition occurs in both alloys as the solute level is increased towards the eutectic point. Although this change in morphology is accompanied by an increase in grain size in the Al-Si

system, this effect is not observed in Al-Cu. Instead, the grain size decreases rapidly with initial solute additions and then levels out as the eutectic composition is approached. The behaviour in the Al-Cu system can be explained by the effect of increased growth restriction with increased solute content, which promotes a successively decreasing grain size. The results for the Al-Si alloys indicate that the increase in growth restriction occurs while there is a concomitant decrease in apparent nucleant potency. Hence the grain size is first reduced to a minimum, then increased as the 'poisoning' effect of silicon begins to dominate. The effect of grain refinement additions and cooling rate on the grain size was subsequently examined in the Al-Si system to investigate the characteristics and confirm the mechanisms responsible for the transition in grain size.

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GRAIN REFINEMENT OF HYPOEUTECTIC Al-Si FOUNDRY ALLOYS WITH TIBLOY: *Eivind Bondhus*[1]; Trond Sagstad[1]; Nora Dahle[1]; [1]Hydelko, Saheimsveien, Rjukan 3660 Norway

TiBloy is a grain refiner developed by Hydelko and Aluminium Rheinfelden, and is optimally adapted to hypoeutectic AlSi foundry alloys. The extreme small mixed borides (Al,Ti)B~ in TiBloy have proved to have a high grain refining efficiency on AlSi alloys, and do not interfere with the modification of the eutectic phase. Nucleation and grain growth in aluminium alloys will be discussed in general and with reference to hypo eutectic aluminium-silicon alloys in particular. Also a theory explaining the mechanism and advantage of TiBloy when applied to aluminium -silicon alloys will be present. The presentation covers grain refining of an A356 and an AlSi7 alloy containing 3 wt% Cu and/or 3 wt% Zn. A comparison between TiBloy and conventional AlTiB refiners containing TiB2 and Al3Ti will be presented. Also trials have been carried out with different Ti amounts. Both basis material with and without Ti were used. Based upon laboratory test results, optimum addition rates of Ti and TiBloy were determined and applied in full scale trials on AluRheinfelden, Germany and alloy A380 at Gland Rapids Aluminum Castings Practical results from full scale production of both alloys will be present.

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HYDLOY-A NEW ALLOY AND METHOD FOR GRAIN REFINING OF ALUMINUM: *Trond Sagstad*[1]; Eivind Bondhus[1]; [1]Hydelko, Sahoimsveien, Rjukan 3660 Norway

Hydelko has developed a new grain refiner alloy called (trademarked) I-Hydloy. The alloy is produced with the attention to substitute all other known grain refiner alloy compositions with a Ti/B-ratio > 2, 2. Hydloy is optimized with regards to remelting, TiB2-agglomeration, particle size and grain refining efficiency, compared to TiB grain refiners in use (5/1, 3/1 etc.), the application of Hydloy has demonstrated a significant improvement of the above mentioned quality criteria. Based on laboratory trials and full scale production tests, the use of Hydloy indicates a potential reduction of boron addition in the form of TiB2 with up to 80%, without any increase in grain size. Consequently, application of Hydloy will be associated with quality enhancement combined with cost reduction. A description of Hydloy and a comparison to conventional AlTi5B1 will be presented. The paper will also present results from laboratory tests, production trials and full scale testing.

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STUDIES ON THE DISSOLUTION OF Al-Ti-C MASTER ALLOY USING LIMCA II: *Einar Asbjornsson*[1]; Graham McCartney[2]; Thorsteinn Sigfusson[3]; Throstur Gudmundsson[4]; Dave Bristow[5]; [1]University of Nottingham & University of Iceland, Dept. of Mats. Sci. & Sci. Institute, University Park & Dunhagi 3, Nottingham & Reykjavik NG7 2RD & 107 England & Iceland; [2]University of Nottingham, Dept. of Matls. Sci., University Park, Nottingham NG7 2RD UK; [3]University of Iceland, Science Institute, Dunhagi 3, Reykjavik 107 Iceland; [4]Alusuisse Technology & Management, Technology Center, Chippis CH-3965 Switzerland; [5]London & Scandinavian Metallurgical Company, Ltd., Technical Center, Fullerton Rd., Rotherham, South Yorkshire S60 1DL England

The dynamics of grain refiners dissolution and agglomeration in aluminium melt is under investigation. The main emphasis is on gaining better understanding of the dissolution and dispersion of grain refiner particles added to molten aluminium. An important part of the project is to evaluate the quantity of insoluble particles larger than 20 micron released from different types of master alloys when added to molten aluminium. To test this, small pieces of the master alloy are dissolved in a crucible, and the increase in the number of particles is monitored with a LiMCA II device. With the LiMCA device it is also possible to observe agglomeration and the conservation of the volume of the insoluble particles. Results from these experiments with an Al-Ti-C master alloy are described.

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A KINETIC STUDY OF DEMAGGING OF MOLTEN ALUMINUM BY THE USE OF SIO2 SUBMERGED POWDER INJECTION: *A. Flores-Valdes*[1]; A. Puente-Amador[2]; [1]Centro de Investigacion, y de Estudio Avanzados del 1PN, Unidad Saltillo, P.O. Box 663, Coahuila 25000 Mexico; [2]Praxair, S.A. de C.V, Av de la Juventud, 614 Nic., Fracc, Nogalar, 66840 San Nicolas, De los Garza, N.I. Mexico

The demagging of molten aluminum is a well known industrial practice that has been carried out by different means, e.g. chlorinating, electrolysis, and by solid reactive fluxing. The common solid fluxes include AlF3, Na2SiF6, KAIF3, K2SiF6, etc., which apart to be expensive, they have the inconvenience to generate toxic fumes based on volatile fluorides. The use of SiO2 has emerged as a suitable alternative, owing to its low cost and its great availability. This work reports the results obtained after using this compound for the demagging of Al-Mg commercial alloys obtained from scrap, using submerged powder injection. The effects of temperature, powder size, the reactive powder flow rate-to-carrier gas flow rate ratio, initial magnesium content, and injection time were studied. Promising results were obtained, as the rate of demagging was measured as a function of the parameters written above, being the magnesium removal efficiencies attained close to 85%.

CREEP BEHAVIOR OF ADVANCED MATERIALS FOR THE 21ST CENTURY: Microstructure and Mechanisms II

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

Program Organizers: Rajiv S. Mishra, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA 95616 USA; Amiya K. Mukherjee, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA 95616 USA; K. Linga Murty, North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA

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March 1, 1999 — Location: Convention Center

Session Chair: R.S. Mishra, University of California, Davis; G. Eggeler, Institut fur Werkstoffe-Werkstoffwissenschaft, Bochum

2:00 PM COMPOSITES

2:00 PM INVITED PAPER

RECENT DEVELOPMENTS IN THE CREEP OF METAL MATRIX COMPOSITES: Yong Li[1]; *Terence G. Langdon*[1]; [1]University of Southern California, Dept. of Mats. Sci. and Mech. Eng., Los Angeles, CA 90089-1453 USA

There have been several recent reports of the creep of metal matrix composites at elevated temperatures and this has led to advances in interpreting the nature of the creep processes. This paper examines

these recent developments with reference to (i) measuring and evaluating the magnitudes of the threshold stresses, (ii) the possibility of incorporating a temperature-dependent load transfer into the analysis and (iii) the division of the deformation behavior into two classes designated class A and class M.

2:25 PM INVITED PAPER

CREEP AND STRENGTHENING IN POWDER METALLURGY SiC-Al COMPOSITES: *Farghalli A. Mohamed*[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Mats. Sci., Irvine, CA 92697-2575 USA

The Creep behavior of particulate Si-Al composites and their unreinforced Al matrices that were prepared by powder metallurgy has been investigated under similar experimental conditions. The experimental data, which cover several orders of magnitude of strain rate, are discussed with special emphasis on the following: (a) major similarities and differences in creep characteristics between the composites and their matrices, (b) interpretation of creep behavior in terms of a threshold stress, (c) steady-state deformation processes, and (d) the loss of strengthening at high strain rates.

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CREEP BEHAVIOR OF ODS ALUMINUM REINFORCED BY SILICON CARBIDE PARTICULATES - AN ODS Al-30SiC$_p$ COMPOSITE: *J. Cadek*[1]; S. J. Zhu[2]; K. Milicka[1]; [1]Academy of Sciences of the Czech Republic, Institute of Physics of Materials, Brno 616 62 Czech Republic; [2]Dalian University of Technology, Dept. of Mats. Eng., Dalian 116023 China

Results of an investigation of creep behavior in ODS aluminum reinforced by silicon carbide particulates - an ODS Al-30SiC$_p$ composite - are reported. The minimum tensile creep strain rates were measured at temperatures 623, 673 and 723 K and applied stresses ranging from 2.77 ∞ 10^{-3} G to 7.74 ∞ 10^{-3} G, where G is the shear modulus of aluminum. The creep in the composite is associated with a relatively high true threshold stress which decreases with increasing temperature more strongly than the shear modulus. The true threshold stress is suggested to originate predominantly from an attractive dislocation/fine alumina particles interaction, the presence of SiC particulates does not seem to contribute to it significantly. The minimum creep strain rate is matrix lattice diffusion controlled and the true stress exponent of this strain rate is close to 5. In this respect, the creep behavior of the ODS Al-30SiC$_p$ composite is similar to that of Al-30SiC$_p$ composite. Depending on the conditions of applied stress and temperature, the minimum creep strain rate in the ODS Al-30SiC$_p$ composite is up to 8 orders of magnitude lower than that in the Al-30SiCp composite. This effect of strengthening of aluminum matrix by fine alumina particles is largely, but no entirely, due to the higher threshold stress in the ODS Al-30SiC$_p$ composite. Some other possible contributions to this effect are discussed, but the load transfer is not considered to play any significant role in the heavily alumina particle strengthened Al-30SiC$_p$ composite.

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CREEP DEFORMATION BEHAVIOR OF SiC PARTICULATE REINFORCED ALUMINUM COMPOSITE: *S. B. Biner*[1]; [1]Iowa State University, Ames Laboratory, 208 Metals Development, Ames, IA 50011 USA

In this study the creep deformation and creep rupture behavior of 20 vol% SiC particulate reinforced 2014 aluminum alloy and 1100 aluminum alloy were studied. The results indicate that the stress enhancement resulting from grain sliding, grain rotations in the matrix and interface cavitation at the reinforcements play a significant role in the creep deformation behavior of the composite as predicted from the numerical analysis. When these effects and the threshold stress concepts are considered together, the creep data for the composite unify with the creep data that are seen for the 1100 aluminum alloy. This work was supported by USDOE, Office of Basic Energy Sciences, Div. of Materials Science under contract no. W-7405-ENG-82.

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CREEP ANISOTROPY OF ALUMINIUM ALLOY-BASE SHORT FIBRE REINFORCED MMC: *A. DlouhΩ*[1]; K. Kucharova[1]; T. Horkel[1]; [1]Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Zizkova 22, Brno 616 62 Czech Republic

Tensile and compression creep was performed using specimens the axis of which was either perpendicular to or parallel with the planar fibre texture of the original MMC block. The composite matrix was represented by the Al-Si7-Cu3-Mg1 alloy, which was reinforced by the 15vol% of Al2O3 Saffile short fibres. Results obtained at the temperature 623K and in the applied stress range 20-70MPa strongly suggest that the minimum creep rate changes considerably with the orientation of the specimen axis and also with the loading mode used (tension vers. compression). A careful metallographic investigation showed that the damage accumulation kinetics in terms of fibre breakage is sensitively dependent on both, the loading mode and the direction of the specimen axis with respect to the plane characterising the preferential orientation of fibres. The observed anisotropy effects are interpreted on the basis of a micromechanical model which links processes of hardening, recovery and damage accumulation in MMCs.

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HIGH-TEMPERATURE RUPTURE OF PARTICULATE REINFORCED AND UNREINFORCED 2124 AL UNDER MULTIAXIAL STRESS STATES: Ahmadali Yousefiani[1]; Farghalli A. Mohamed[1]; *James C. Earthman*[1]; [1]University of California at Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA

High-temperature deformation and rupture behavior of powder metallurgy (PM) 2124 Al has been studied under uniaxial, biaxial, and triaxial stress states. Tests were conducted at 648K, over four orders of magnitude of strain rate. Results from the uniaxial creep tests reveal a high and variable apparent stress exponent that decreases with increasing stress levels. It was found that this anomalous behavior can be interpreted in terms of a threshold stress for creep, and deformation in the alloy is driven by an effective stress, which incorporates this threshold stress. Rupture times for the different stress states are compared with respect to four different mechanistic multiaxial stress parameters, which are each linked to a particular physical mechanism controlling the creep rupture process. The results indicate that Cane's representative stress parameter successfully correlates the data over the entire stress range investigated. This suggests that the creep rupture process is constrained by creep deformation in the matrix, and the driving force for cavity growth is determined by the rate of dislocation creep in the matrix. Furthermore, the results obtained in this investigation are compared with those obtained earlier for a particulate reinforced PM 2124 Al alloy. Similarities in their rupture behavior emphasize the critical role of the matrix PM alloy during creep deformation.

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CREEP BEHAVIOR OF MAGNESIUM CONTAINING HIGH VOLUME FRACTIONS OF OXIDE DISPERSOIDS: *B. Q. Han*[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Material Science & Eng., 2225 N. Campus Dr., Rm. 2036, Evanston, IL 60201 USA

This paper examines the creep properties of oxide-dispersion-strengthened magnesium composites fabricated by melt infiltration of 0.3 µm Y2O3 particle with unalloyed magnesium. The composites contained 30 vol.% particles and exhibited a very fine grain size of 0.5 µm due to recrystallization upon extrusion. Compression creep experiments were performed between 300ϒC and 450ϒC for stresses between 7 MPa and 150 MPa. Creep is characterized by two distinct regions. At low stresses, the stress exponent is about n = 2, indicating that deformation is controlled by diffusional mechanisms. At high stresses, the stress exponent is about n = 10, indicative that deformation is dominated by power-law creep mechanisms with a threshold stress. The origin of this threshold stress is discussed in the light of existing models.

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ROLE OF SiC REINFORCEMENTS ON HIGH TEMPERATURE CREEP BEHAVIOR OF SiC/Al METAL MATRIX COMPOSITES: Ho J. Ryu[1]; *Soon H. Hong*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. & Eng., 373-1 Kusung-dong, Yusung-gu 305-701 Korea

The role of SiC particles and whiskers on high temperature creep deformation of SiC/2124Al composites were investigated by constant stress creep tests at 300℃. The volume fraction of SiC particle in SiCp/2124Al varied from 10 to 30% to investigate the effect of volume fraction of SiC particles on creep deformation of SiCp/2124Al composites. The extrusion ratio of 20 vol.% SiCw/2124Al varied from 10:1 to 25:1 to investigate the effect of aspect ratio and misorientation angle of whiskers on creep deformation of SiCw/2124Al composites. The minimum creep rate decreased with increasing the volume fraction of SiC particles in SiCp/2124Al metal matrix composite. The SiCw/2124Al composite showed the lowest minimum creep rate when extruded 15:1. The shear lag model was modified to analyze the effect of volume fraction of SiC particles and the aspect ratio and misorientation angle of SiC whiskers on creep behavior of SiC/Al composite. A new concept of effective aspect ratio was proposed, combining the effects of aspect ratio and alignment of reinforcement, to calculate the effective stress on 2124Al matrix. The minimum creep rates of SiC/2124Al composites with different volume fraction of SiC particles and with different aspect ratio and alignment of SiC whiskers were found to be similar under an identical effective stress. It is suggested that the creep deformation of composite proceed by the deformation of matrix and the role of SiC reinforcement is to reduce the effective stress acting on the matrix.

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AN INVESTIGATION OF THE CREEP BEHAVIOR OF A MAGNESIUM-BASED COMPOSITE: *Yong Li*[1]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Mats. Sci. & Mech. Eng. Los Angeles, CA 90089-1453 USA

An investigation was conducted to evaluate the creep behavior of an AZ91 alloy (Mg-9% Al-1% Zn) strengthened with 20 volume per cent of Saffil (Al_2O_3) reinforcement over the temperature range from 473 to 673 K. All creep tests were conducted in air using specimens machined into a double-shear configuration. This paper describes the experimental results and and presents an analysis of the data in terms of the true stress exponent and the true activation energy.

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ROLE OF INTERFACIAL CREEP DURING DEFORMATION OF FIBROUS AND LAYERED METAL-MATRIX COMPOSITES: *Nagarajan Rajagopalan*[1]; Indranath Dutta[1]; [1]Naval Postgraduate School, Dept. of Mech. Eng., Center for Materials Science and Engineering, Monterey, CA 93943 USA

The creep response of the matrix/reinforcement interface in metal-matrix composites (MMCs) has been studied on a model single Ni fiber reinforced Pb-matrix composite and Pb/Ni multi-layered composite. A constitutive law for interfacial creep has been derived based on single fiber pushdown test. The results suggest that the interface slides by diffusional creep with a threshold stress (Bingham flow). The activation energy associated with interfacial sliding suggests that the interface acts as a high diffusivity path with the mechanism being interface-diffusion-controlled interfacial creep. To get a fundamental insight on the role of interfacial creep in fiber reinforced composites, constant load tensile creep experiments were performed on a model diffusion bonded Pb/Ni composites. The results indicate that the interface slides near the free surfaces at the ends of the fiber where the interfacial shear stress developed is maximum. This results in divergence of the matrix and fiber strains, which are measured independently, producing strain incompatibility at the fiber ends. The extent of the sliding depends on the aspect ratio of the fiber and the gauge length of the sample. Similar studies have been conducted on three layered Pb/Ni system to understand the influence of the interfacial creep in multi-layered systems. A unidimensional micro-mechanical model for creep deformation for fibrous and layered metal-matrix composites has been proposed by incorporating the constitutive law for interfacial creep and thermal strain history of the sample. These results demonstrate the importance of interfacial creep in metal-matrix composites and thin film coating which are being considered for both structural and non-structural applications.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Solidification and Casting: Modeling and Numerical Methods

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee; Jt. Extraction & Processing Division and Materials Processing and Manufacturing Division, Synthesis, Control, and Analysis in Materials Processing Committee; Light Metals Division

Program Organizers: Nagy El-Kaddah, University of Alabama, Dept. of Met. & Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Stein Tore Johansen, SINTEF Materials Technology, Process Metallurgy & Ceramics, Trondheim, NTH N-7034 Norway; David G. Robertson, University of Missouri-Rolla, Dept. of Metall. Eng., Rolla, MO 65409-1460 USA; Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Minneapolis, MN 55414-2196 USA

Monday PM Room: 2
March 1, 1999 Location: Convention Center

Session Chairs: Vaughan R. Voller, University of Minnesota, Minneapolis, MN 55414 USA; Christoph Beckermann, University of Iowa, Dept. of Mech. Eng., Iowa City, IA 52242 USA

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MODELLING THE CASTING PROCESS: A REVIEW OF WHERE IT STANDS FROM THE FOUNDRY PERSPECTIVE: *Mark Jolly*[1]; [1]The University of Birmingham, Process Modelling Group, IRC in Materials, Edgbaston, Birmingham B15 2TT UK

Over the last ten years or so many man hours have been dedicated to developing methods for simulating the casting process. The majority of the methods developed have been devised by a combination of computer, mathematics and materials specialists with little or no knowledge of foundries and foundrymen. As a result of this it would appear that although there are many software packages for the foundryman to use there still exist fundamental misunderstandings as to their usefulness and suitability within the foundry. The paper aims to identify how well current software packages perform. The relationships between physical phenomena, practical defects and software capability are presented. Some discussion of the use of criterion functions is also presented. Finally issues arising from the post-processing of results are discussed as the presentation of the results to the lay-person in simulation techniques is possibly one of the most important aspects in influencing the adoption of this type of software in the foundry community.

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THE EVOLUTION OF A SOLIDIFICATION MODEL: *Vaughan R. Voller*[1]; [1]University of Minnesota, St. Anthony Falls Laboratory, Mississippi River at 3rd Ave. S.E., Minneapolis, MN 55414 USA

The objective of this paper is to highlight key components in a solidification model. Towards this end a test system involving the unidirectional solidification of an aluminum alloy is considered. In the first place, a basic conduction model is proposed. In subsequent sections, additional features are added to the model. In each case, appropriate model equations and an associated numerical solution approach are presented. In this development focus is placed on the nature and effect of (i) fluid flow, (ii) solid movement, (iii) microsegregation, and (iv) the coupling of solutal and thermal fields.

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SIMULATION OF CONVECTION AND MACROSEGREGATION IN A LARGE STEEL INGOT: *Christoph Beckermann*[1]; J. P. Gu[1]; [1]University of Iowa, Mech. Eng., 2412 EB, Iowa City, IA 52242 USA

Melt convection and macrosegregation in casting of a large steel ingot is numerically simulated using a previously developed model for

coupled transport phenomena in the liquid, mush, and solid. Heat transfer in the mold and insulation materials, as well as the formation of a shrinkage cavity at the top are taken into account. The predicted variation of the macrosegregation of carbon and sulfur along the vertical centerline is compared with measurements from an industrial steel ingot that was sectioned and analyzed. Although generally good agreement is obtained, the neglect of settling of free equiaxed grains prevents the prediction of the zone of negative macrosegregation observed in the lower part of the ingot. It is also shown that the variation of the final solidification temperature due to macrosegregation is important in obtaining good agreement between the predictions and measurements.

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NATURAL CONVECTION IN ALUMINIUM INGOTS: O. Paireau[1]; *P. A. Davidson*[1]; T. Alboussière[1]; [1]University of Cambridge, Eng. Dept., Trumpington St., Cambridge CB2 1PZ UK

We have designed an experiment to simulate natural convection in a direct-chill aluminium ingot. In this experiment we induce natural convection within a hemisphere filled with liquid-metal (mercury). The device consists of a copper hemispherical bowl closed by a copper plate. The top is maintained at superheat temperature while the boundary is cooled. While the temperature measurements were found using thermocouples, the velocities were obtained using an original technical procedure that relies on MHD properties. By applying a steady local magnetic field, we measure the velocity. The distribution of temperature and velocity can be separated into a thermally stratified core bounded by thermal wall jets. These wall jets greatly influence the degree of macrosegregation in ingots.

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EFFECT OF THERMOCAPILLARY CONVECTION ON MELTING OF DROPLETS: *J. M. Khodadadi*[1]; Y. Zhang[1]; [1]Auburn University, Dept. of Mech. Eng., 201 Ross Hall, Auburn, AL 36849-5341 USA

Melting and solidification of materials are important processes which are encountered in nature and numerous industrial applications, such as spray casting, space radiators, rapid solidification, purification of materials, containerless processing, etc. Specifically, the effect of thermocapillary convection on the evolution of the phase change within droplets has not been addressed at all. Only a few authors have studied the transport phenomena without phase change in spherical droplets. In view of the nonexistence of any previous work on transport phenomena during the thermocapillary-assisted melting of spherical droplets, this study was initiated to elucidate the main features of the phenomenon. A computational study of the effect of thermocapillary convection on melting of a droplet under the influence of an incident uniform heat flux is presented. The computations are based on an terative, finite-volume numerical procedure using primitive dependent variables, whereby the time-dependent continuity, momentum and energy equations in the spherical coordinate system are solved. During the early periods of the melting process, conduction mode of heat transfer is dominant. As the thermocapillary convection is strengthened due to the growth of the melt zone, melting on the side of the droplet is observed to be much faster in comparison to the conduction-only case. Due to the skewedness of the temperature field, a new recirculating vortex is created which promotes melting within the droplet. For a greater Pr-number fluid, it is observed that the molten zone increases very fast along the surface and reaches the unheated side, thus enclosing a solid inner core.

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A ROBUST IMPLICIT INTERFACE TRACKING METHOD FOR NUMERICAL MODELLING OF FAST SOLIDIFICATION PROCESSES: S. P. Wang[1]; G. X. Wang[1]; *Eric F. Matthys*[1]; [1]University of California, Dept. of Mech. and Enviro. Eng., Room 2330, Engineering II, Santa Barbara, CA 93106-5070 USA

A robust implicit interface-tracking method was developed for numerical modelling of fast solidification processes. This new method is based on our previous interface-tracking with element subdivision scheme. This previous scheme suffers from numerical instability in cases where the moving interface velocity becomes extremely high. In addition, numerical instabilities also arise for rapidly-solidifying materials with large latent heat, high density and low thermal conductivity. A new iteration scheme was therefore developed to determine implicitly the interface parameters. The stability of the new scheme is discussed in light of Patankar's positive coefficient principle. To demonstrate the validity of the present scheme, four test cases under splat cooling conditions were investigated. A comparison of the two methods shows that the new method gives stable solutions in all cases, whereas the previous method is only conditionally stable in special cases.

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PROJECTION METHODS FOR COMPUTATION OF INTERDENDRITIC FLOWS: A. S. Subau[1]; *S. Viswanathan*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Bldg. 4508, MS 6083, Oak Ridge, TN 37831-6083 USA

Liquid contraction during solidification plays an important role in phenomena such as macrosegregation, shrinkage, and microporosity. As a first step towards developing numerical methods for full numerical simulations of transport phenomena in materials processing, this study presents two-step projection methods for the numerical simulation of shrinkage induced flows during solidification. Methods are presented for solving the full mass and momentum conservation equations for variable liquid fractions and densities in solidifying alloy. An implicit treatment of the Darcy's and Forchheimer's terms in the momentum equation is used to remove the severe time step restriction due to the explicit or semi-implicit discretization of these terms. Numerical examples for shrinkage-induced flow during the solidification of an Al-4.5% Cu alloy bar are used to illustrate the effectiveness of the proposed algorithm.

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DEVELOPMENT OF STRESS IN A DENDRITIC NETWORK DUE TO SHRINKAGE INDUCED FLOW: *Haavard J. Thevik*[1]; Arne Kristian Dahle[2]; [1]SINTEF, Materials Technology, P.O. Box 124, Blindern, Oslo N-0314 Norway; [2]The University of Queensland, Dept. of Mining, Minerals and Mats. Eng., CRC for Alloy and Solidification Technology (CAST), Brisbane, Queensland Qld 4072 Australia

A mathematical model that calculates the stress development in a coherent and stationary dendritic network due to interdendritic flow is proposed. The fluid flow is assumed to be related to the solidification shrinkage, and under the assumption of no porosity formation and unidirectional solidification, the shrinkage-induced flow within the mushy zone is calculated analytically. The interdendritic melt flow exerts a force on the solid network and an analytical expression for the stress build-up in the network is developed. Parameter studies illustrating the influence of cooling conditions, mushy zone length, dendrite coherency fraction solid, volume fraction of eutectic, and solidification shrinkage upon the stress build-up within the solid network are presented. Comparing the magnitude of the calculated stresses to measurements of the strength in equiaxed mushy zones shows that it is possible for the stresses to exceed the strength, thereby resulting in reorientation or collapse of the dendritic network.

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COMPUTATIONS OF STRUCTURES FORMED BY THE SOLIDIFICATION OF IMPINGING MOLTEN METAL DROPS: *Judy Che*[1]; Gretar Tryggvason[1]; Steven L. Ceccio[1]; [1]University of Michigan, Dept. of Mech. Eng. and Applied Mechanics, Ann Arbor, MI 48109-2121 USA

Layered manufacturing techniques, which deposit 20-100 mm diameter droplets of molten metal, are a means of producing prototypes rapidly. These slowly moving drops, with velocities on the order of 5 m/s, do not disintegrate on impact to form finger-like lamellae as in splat quenching techniques, instead these drops remain intact and build upon one another. The ability to computationally predict final drop shape and cooling rate will help manufacturers select parameters to produce parts with desired shape and mechanical properties. To assist the development of such predictive methods, the solidification of molten metal drops impinging on a cold substrate to form coherent structures is studied numerically. Axisymmetric and fully three-dimensional results of towers built by the deposition of multiple drops are presented. A parameter study demonstrates the effect of Weber number, Peclet number, and drop deposition frequency on the final form of the structure. Examples

of the complex interaction of multiple drops falling at oblique angles are shown in three-dimensions. In these computations, fluid flow, heat transfer, and phase change are fully coupled throughout the drop deformation and solidification process. Heat transfer is characterized primarily by conduction of heat through the solid. Both latent heat of fusion and thermal contact resistance effects are included. The solution method is based on a single set of conservation equations for all phases simultaneously. The phase boundaries are treated as imbedded interfaces by adding the appropriate source terms to the conservation equations. Each of these phase boundaries is tracked explicitly using a lower dimensional moving grid. This method has been validated by comparison with experiments and grid independence studies.

GENERAL ABSTRACTS: Session 3 - Mechanical Properties II

Sponsored by: TMS

Program Organizers: Garry W. Warren, University of Alabama, Dept. of Met. and Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Ray D. Peterson, IMCO Recycling, Inc., Irving, TX 75039 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Monday PM Room: 12
March 1, 1999 Location: Convention Center

Session Chairs: Michael Miles, American National Can Company, Chicago, IL 60631 USA; John Vetrano, Pacific Northwest National Laboratory, Richland, WA 99352 USA

2:00 PM

COMPARISON OF SOLUTIONIZING EFFECT OF 7075 ALUMINUM ALLOYS ON AGING WITH 6061 AND 2014 ALUMINUM ALLOYS AND COMPOSITES: *Erika V. Esquivel*[1]; Shailendra K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metall. and Mats. Eng., El Paso, TX 79968-0520 USA

An effect of solutionizing time on the age hardening curves of 7075 aluminum alloy has been determined. The results have been compared with those previously reported on 6061 and 2014 aluminum alloys and composites reinforced with various volume fractions of alumina particles. The time required to get the peak hardness values during aging has been compared in these materials. The grain growth law (square of grain diameter as a function of solutionizing time) has been tested and its influence on the aging response has been evaluated. Microstructural evolution during aging will be characterized by transmission electron microscopy (TEM) and correlated to the hardness values in the underaged, peak hardened and overaged conditions.

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THE EFFECTS OF TRACE ELEMENT ADDITIONS AND THERMAL-MECHANICAL PROCESSES ON THE HIGH TEMPERATURE MECHANICAL BEHAVIOR OF PM U720 FORGED ALLOY: *Chih-An Yin*[1]; Bruce Ewing[1]; Sushil Jain[1]; [1]Allison Engine Company, P.O. Box 420, S-52, Indianapolis, IN 46204-0420 USA

Although considerable work has been done on the effects of various processing parameters on the structural and mechanical properties of conventional cast-wrought U720 alloy, little is known about the effects in PNI U720 forged alloy. This study was undertaken to investigate the structural changes and mechanical behavior induced in PM U720 forged alloy by thermal-mechanical processes and/or trace element modifications. Tensile, creep-rupture, low cycle fatigue and fatigue crack growth testing as well as optical and electronic microscopy were used to evaluate composition/process modified material. By comparison to conventional cast-wrought U720 alloy, it was found that grain size variations and trace element modifications have resulted in significant improvements in strain controlled LCF and load controlled FCGR when both were tested at 1200F. Analysis of microstructure and creep-rupture properties indicated that the grain size variation was a dominant factor as compared to the influence of trace element additions. Finally, the thermal-mechanical processes employed did not change 1200F tensile strength when compared to the conventional cast-wrought U720 alloy; however, a slight reduction in 0.2% yielding strength was observed as compared to conventional cast-wrought U720 alloy.

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ELEVATED TEMPERATURE CREEP PROPERTIES OF THE 54Fe-29Ni-17Co "KOVAR" ALLOY: *John J. Stephens*[1]; Jerome A. Rejent[1]; David T. Schmale[1]; [1]Sandia National Laboratories, Dept. 1833, MS0367, P.O. Box 5800, Albuquerque, NM 87185-0367 USA

Despite the widespread use of the Kovar alloy in metal/ceramic brazing and glass-to-metal sealing applications, very limited elevated temperature mechanical properties data exist for this material in the literature. This study was motivated by a need to identify the optimum stress/ temperature/time parameters for a diffusion bonding application of Kovar sheet. Compressive stress-strain properties (24ϒC-900ϒC) as well as creep tests (750-900ϒC) were obtained from 1/4 inch diameter bar material conforming to the ASTM F1466 specification. The minimum creep rate data for Kovar alloy at elevated temperatures was found to obey a classical power law equation with a stress exponent of 4.9 and an activation energy of 52.7 kcal/mole. The effect of elevated temperature on grain growth in Kovar alloy was also documented and will be compared to a previous study on grain growth of 0.020 inch diameter wire material. *Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U. S. Dept. of Energy under Contract DE-AC04-94AL85000.

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CREEP BEHAVIOR OF POWDER METALLURGY SiC_p-2014 Al AND 2014 Al: *Zhigang Lin*[1]; Farghalli A. Mohamed[1]; [1]University of California at Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA

The creep behavior of powder metallurgy (PM) 10% silicon particulate reinforced 2014 aluminum alloy (SiC_p-2014 Al) and its matrix, PM 2014 Al, was investigated over six orders of magnitude of strain rate and in the temperatures range of 618-678K. The results are examined in the light of recent data for the creep characteristics of discontinuous SiC-Al composites and their matrices.

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EFFECT OF HYDRIDE ORIENTATION ON THE DUCTILITY LOSS OF ZIRCALOY CLADDING: *Jen-hung Chen*[1]; N. K. Sungyu[1]; [1]Institute of Nuclear Energy Research, Nuclear Fuel and Materials, P.O. Box 3-14 Lung-Tan, Taoyuan 325 Taiwan

The effect of zirconium hydride orientation on the mechanical properties of Zircaloy-4 cladding was investigated. It was found that the stress reorientation of hydrides in the cladding occurred as the hoop stress larger than a critical value. And the reorientation of hydride from circumferential to radial can be affected by the hoop stress, solution time and thermal cycling applied. Tension test was conducted at room temperature on the reoriented cladding specimens to evaluate the effect of radial hydride. Two types of specimens were tested. One is uniaxial tension test specimen that is in a plane stress loading state, and the other one is slotted arc tension specimen under a plane strain condition approximating the realistic operation in reactors. The results showed that radial hydrides significantly reduce the elongation of the slotted arc tension specimens, but there was no effect to be revealed on the uniaxial tension specimens.

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THE EFFECT OF OXIDE PARTICLES ON THE CREEP BEHAVIOR: *Zhigang Lin*[1]; Sammy L. I. Chan[2]; Farghalli A. Mohamed[1]; [1]University of California at Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA; [2]National Taiwan University, Institute of Mats. Sci. and Eng., Taipei 10617 Taiwan

The creep behavior of two grades of 2014 Al, which were prepared by powder metallurgy (PM) and whose oxygen content was different

(0.3wt% and 1.0wt%), was investigated over six orders of magnitude of strain rate. The results show that the creep characteristics of PM 2014 containing 0.3% oxygen are similar to those of PM 2014 containing 1.0% oxygen with regard to the variation in both the apparent stress exponent and the apparent activation energy for creep with applied stress. However, a direct comparison between the creep data of these two alloys at low strain rates reveals that, for constant temperature, PM 2014 Al with an oxygen content of 1.0% is more creep resistant than PM 10% SiC-2014 Al with an oxygen content of 0.3%. The above characteristics are discussed in terms of a threshold stress for creep that is attributed to the interaction between moving dislocations and oxide particles.

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FERRITE REFINEMENT BY HEAVY DEFORMATION IN PLAIN CARBON STEELS: *Sang Woo Lee*[1]; [1]POSCO Technical Lab, P.O. Box 36, Pohang Korea

As a basic research for making ultrafine-grained steel, the ferrite refinement by heavy deformation was studied. Using hot deformation simulator, uniaxial compression was performed on O.15C-1.1Mn- O.25Si steel. Experiments were carried out by varying deformation temperature, reduction, cooling rate and strain rate. When a specimen was deformed by 80% just above Ar3 temperature ferrite grain size of about 2 um was obtained. This fine grain size may be obtained through deformation induced transformation. Microstructures of specimens were analyzed from the viewpoint of ferriite refinement mechanisms. In addition, the ferrite transformation behavior during and after deformation will be discussed.

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THE TEMPERING BEHAVIOR OF HIGH STRENGTH 14 COBALT/ 10 NICKEL SECONDARY HARDENING STEELS: *Luana E. Iorio*[1]; James L. Maloney[2]; Warren M. Garison[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. & Eng., Pittsburgh, PA 15213 USA; [2]Latrobe Steel Company

For the past 25 years new ultra high strength steels have largely been extensions of the approach Speich and coworkers used to develop HY180, which suggested that HY180 achieves excellent toughness after tempering at 510ϒC, in part, because of the absence of coarse intra-lath cementite. To achieve higher strengths in this system the primary approach has been to increase the carbon content. AF1410 and AerMet 100 have carbon levels of 0.16 and 0.24 wt.% respectively, compared to 0.10 wt.% carbon in HY180. The effects of molybdenum and chromium contents on tempering response and cementite precipitation have been investigated as a function of carbon content in AF1410 and AF1410-type steels. The carbon content varies from 0.16 to 0.25 wt.% and the molybdenum and chromium levels vary from 1.25 to 1.75 wt.% and from 2.0 to 2.5 wt.%, respectively. Increasing the molybdenum content was found to increase the peak hardness, while increased chromium levels led to an increased rate of overaging. The extent of cementiteprecipitation appeared to be controlled by the carbon level of the alloy. This work was funded by the National Science Foundation and the Latrobe Steel Company.

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EFFECT OF ALLOYING ELEMENTS ON STRESS RELAXATION BEHAVIOR OF EUTECTOID STEELS AT ROOM TEMPERATURE: *Kyung-Tae Park*[1]; [1]POSCO Tech., P.O. Box 36, Res. Lab., Pohang Korea

A series of room temperature stress relaxation text was carried out on several eutectic steels containing different alloying element content. Stress relaxation behavior of these steels was analyzed by converting load - time data into stress - true plastic strain rate and by constructing plots of true stress against true plastic strain rate in double logarithmic scale. The results exhibited that, under the present experimental conditions, the lines in the plot were straight unlike BCC and FCC metals. Of alloying elements, Si, Cr and Mn were effective on enhancing stress relaxation resistance in the order. However, Cu and Ni had little effect on stress relaxation behavior. Discussion was made in light of the effect of alloying elements on interlamellar spacing of these pearlitic eutectoid steals.

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SPRAY ATOMIZATION AND DEPOSITION PROCESSING OF AN Al-Cu-Mg-Ag ALLOY: *Linda Y. Del Castillo*[1]; Ahmadali Yousefiani[1]; Farghalli A. Mohamed[1]; Enrique J. Lavernia[1]; [1]University of California at Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., 916 Eng.Tower, Irvine, CA 92697 USA

A new series of precipitation hardenable alloys based on the Al-Cu-Mg-Ag system has been developed for high strength and toughness at both room and elevated temperatures. The addition of Ag in Al-Cu-Mg alloys with high Cu/Mg ratios leads to the formation of Ω, a secondary phase which reveals good thermal stability up to 180°C. Concurrent precipitation of θ', Ω, and dispersion strengthening phases in Al-Cu-Mg-Ag alloys has led to an excellent combination of room and elevated temperature mechanical properties. The use of spray deposition processing for the synthesis of the aforementioned alloy system can improve material properties by providing a uniform distribution of alloying elements, increasing the solid solubility of transition elements, and refining the grain size. To that end, the primary objective of the present research program has been to investigate the effects of spray deposition processing on the room and elevated temperature mechanical properties and precipitation kinetics of t he experimental Al-Cu-Mg-Ag alloy C415 (Al-5.0Cu-0.8Mg-0.6Mn-0.5Ag). C415 was synthesized using spray atomization and deposition. In view of the previously discussed objectives, the present investigation involved kinetic studies, microstructural characterization, and mechanical testing of both spray deposited and ingot cast Mats.. Experimentation included preliminary identification and characterization of secondary phases as well as determination of room and elevated temperature tensile properties, creep behavior, and fracture toughness.

HIGH-TEMPERATURE SUPERCONDUCTORS: SYNTHESIS, FABRICATION AND APPLICATION: Tl, Hg, & Bi-Based Systems

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Superconducting Materials Committee
Program Organizers: U.Balu Balachandran, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 212, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, 450 Old Niskayuna Rd., Latham, NY 12110 USA; Chandra Pande, Naval Research Lab, Mats. Sci. & Tech. Div., Washington, D.C. 20375-5000 USA

Monday PM Room: 18
March 1, 1999 Location: Convention Center

Session Chairs: Winnie Wong-Ng, National Institute of Standards & Technology, Ceramics, Gaithersburg, MD 20899 USA; Peter Majewski, Max-Planck-Institute, PML, Stuttgart 70569 Germany

2:00 PM

PHASE DIAGRAM STUDIES IN THE SYSTEM Tl2O3-BaO-CaO-CuO-Ag: *Peter Majewski*[1]; Artur Jalowiecki[1]; Fritz Aldinger[1]; [1]Max-Planck-Institut für Metallforschung, PML, Heisenbergstr. 5, Stuttgart 70569 Germany

The phase relations in the system Tl2O3-BaO-CaO-CuO-Ag are studied with emphasis on the phases TlBa2Ca2Cu3O8.5 (1223) and Tl2Ba2Ca2Cu3O10 (2223). The samples were prepared in quartz container filled with pure oxygen at 890ϒC. The samples consist of about 80 vol% of silver in order to fir the conditions during the experiments with those during tape processing. 1223 and TlBa2CaCu2O6.5 (1212) are stable only at low Tl contents. With increasing Tl concentrations 2223 and Tl2Ba2CaCu2O8 (2212) are dominant. An equilibrium between 1223 and a liquid phase has not been found.

2:20 PM INVITED PAPER

PROGRESS ON Ti-OXIDE WIRE AND TAPE DEVELOPMENT: *R. D. Blaugher*[1]; R. N. Bhattacharya[1]; Z. F. Ren[2]; J. H. Wang[2]; [1]National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA; [2]State University of New York at Buffalo, Buffalo, NY 14214 USA

The electrodeposition method for depositing thick film Tl-Bi-Ba-Sr-Ca-Cu-O precursors currently presents the most promising technique for fabricating superconducting Tl-oxide wire or tape. Thick-film processing using electrodeposition on textured nickel substrates has shown J_c above 105 A/cm^2 at 77K in zero magnetic field. The magnetic field dependence within the liquid nitrogen range should be suitable for practical applications such as transmission cables and transformers if long lengths with comparable properties are produced. This paper will review the recent progress for Tl-oxide wire and tape processing employing the electrodeposition technique with and without Tl in the precursor. The prospects for a "long length" thick-film process using a continuous electrodeposition method will also be discussed.

2:40 PM INVITED PAPER

PREPARATION OF THALLIUM CUPRATE THIN FILMS ON LARGE AREA SUBSTRATES: *Norbert Reschauer*[1]; Helmut Kinder[1]; [1]Technische Universität München, Physik Dept. E10, James-Franck-Str. 1, Garching 85747 Germany

For microwave applications thallium cuprate films are required because of their low surface resistances. Due to the high volatization of the thallium oxides such films are difficult to prepare reproducably on large area substrates. We have developed a new thallination technique, which allows the reproducible, simultaneos preparation of 10 thallium cuprate films with diameters of 3''. The precursor films were put in a container and annealed in a tube furnace at temperatures of 700-850ϒC, respectively to the requested compound. First results show that the new method represents a cost-effective, easy handling technique to prepare large area thallium cuprate films suitable for microwave applications.

3:00 PM INVITED PAPER

SYNTHESIS, PHASE STABILITY, AND SUPERCONDUCTIVITY OF DOPED HG1223 SUPERCONDUCTORS: *P.V.P.S.S. Sastry*[1]*;* Justin Schwartz[1]; [1]National High Magnetic Field Laboratory, Mag. Sci. and Tech., 1800 East Paul Dirac Dr., Magnet Lab, Tallahassee, FL 32310 USA

The Hg1223 superconductor has the highest Tc (135K) among all the high temperature superconducting cuprates. The stability and superconducting properties of Hg1223 can be improved by partial substitution of Hg by Re, Bi, and Pb. There are, however, no studies reported on the affect of various dopants on the phase stability and superconducting properties of Hg1223. We synthesized a large number of HgX1223 (X=Bi, Pb, and Re) samples and conducted a systematic study on the phase stability and superconductivity. Bulk HgX1223 samples were synthesized from commercial BaCaCuO precursor powder using CaHgO2 as the external Hg source. Phase composition and superconducting properties of several samples, stored in a desiccator or in ambient atmosphere, were followed over a period of one year. It was observed that the stability depends on the phase purity of HgX1223 samples. Pure HgX1223 samples did not show any noticeable degradation. Results of microstructural investigations and superconductor property measurements are discussed.

3:20 PM INVITED PAPER

MICROSTRUCTURAL AND PHYSICAL INVESTIGATIONS OF ELECTROCHEMICALLY DEPOSITED Bi-BASED 2212 CERAMICS ON SILVER SUBSTRATES: *Marcel Ausloos*[1]; Paulette Clippe[1]; Hassan Bougrine[2]; Jean-Philippe Dumont[3]; Rene Linard[3]; *Rudi Cloots*[3]; [1]University of Liege, SUPRAS, Institute of Physics, B5, Liege B-4000 Belgium; [2]University of Liege, SUPRAS, Montefiore Electricity Inst., B28; [3]University of Liege, SUPRAS, Institute of Chemistry, B6

Bi-based 2212 superconducting ceramics on silver substrates have been synthesized by using an appropriate electrochemical deposition process. Different types of ionic concentrations have been considered in order to control the stoichiometry of the deposited phase. An optimization of the thermal process needed for oxidation and sintering of the expected superconducting phase is presented. Microstructural and electrical transport properties investigations have been performed and correlated to the chemical composition of the electrolytic solution.

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ROLE OF MELTING EQUILIBRIA IN THE PROCESSING OF HIGH Tc SUPERCONDUCTORS IN THE Bi-Pb-Sr-Ca-Cu-O SYSTEM: *Winnie Wong-Ng*[1]; Lawrence P. Cook[1]; [1]National Institute of Standards and Technology, Ceramics, A256 , NIST, Dept. of Mats, Gaithersburg, MD 20899 USA

Melts play a significant role in materials processing, especially for high Tc superconductors in the (Bi,Pb)-Sr-Ca-Cu-O (BSCCO) system. Characterization of melts is therefore of central importance to the design and optimization of successful processing paths. Since both the Pb-doped and Pb-free high Tc superconductors are important in the superconducting wire and tape industry, investigation of melts associated with these compounds have been conducted extensively in our laboratory. In this paper, different types of melts and their roles during material processing will be discussed.

4:10 PM INVITED PAPER

CRYSTAL GROWTH OF Bi2212 PHASE IN HIGH MAGNETIC FIELD: *M. Maeda*[1]; W. P. Chen[1]; K. Kakimoto[1]; M. Kikuchi[1]; K. Watanabe[1]; M. Motokawa[1]; H. Kumakura[2]; K. Itoh[2]; [1]Tohoku University, Institute for Materials Research, Katahira 2-1-1, Aoba-ku, Sendai-shi 980-8577 Japan; [2]National Research Institute for Metals, Tsukuba Japan

Recently, we have found that high magnetic field applied during crystal growth enhances the Bi2212 grain alignment. To examine this behavior in detail we prepared dip-coated Ag/Bi2212 composite tapes and Bi2212 bulks, with and without Pb content up to 0.6, with the partial melt-solidification process (PMSP) in magnetic fields up to 9T. For the samples we measured superconducting properties, mainly magnetic J_c at 4.2 to 77K, and investigated morphology and grain alignment of Bi2212 phase and on what precipitates and defects appear in the oxide core using XRD, SEM, and TEM. The formation of Pb doped Bi2212 phase is enhanced with decreasing the cooling rate in PMSP. For all the samples the c axis of the Bi2212 crystals are highly aligned along the magnetic field and the alignment increases with increasing the magnetic field. The J_c values strongly depend on the grain alignment.

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CONTROL OF HOLE CONCENTRATION AND SUPERCONDUCTIVITY IN THE Bi2Sr2(Ca1-xYx)Cu2O8+d SYSTEM: *Ru-Shi Liu*[1]; I. J. Hsu[1]; J. M. Chen[2]; [1]National Taiwan University, Dept. of Chem., Roosevelt Rd., Section 4, Taipei, Taiwan ROC.; [2]Synchrotron Radiation Research Center (SRRC), Hsinchu, Taiwan ROC

The Bi2Sr2(Ca1-xYx)Cu2O8+d system demonstrates a parabolic variation of Tc with hole concentration, there being a minimum and a maximum hole concentration for superconductivity. Tuning the chemical content between Ca2+ and Y3+, the overdoped, optimal doped and underdoped states in the Bi2Sr2(Ca1-xYx)Cu2O8+d system can be controlled. The hole concentration within the CuO2 planes which is responsible for superconductivity in the Bi2Sr2(Ca1-xYx)Cu2O8+d system can be observed by high-resolution O K-edge x-ray-absorption near-edge-structure spectra. Moreover, the correlation between hole concentration and critical current density in the Bi2Sr2(Ca1-xYx)Cu2O8+d system will be also reported.

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AN ENHANCEMENT OF T_C IN $(Pb_{0.59}Cd_{0.5})Sr_2(Y_{0.67}Ca_{0.4})Cu_20_{7-8}$ BY $NANO_3$ ADDITION: *B. Latha*[1]; T. S. Sampath Kumar[1]; [1]University of Madras, Dept. of Nuclear Phys. & Mat. Sci. Centre, Guindy Campus, Chennia 600 025 India

We report the first case of a substantial T_c enhancement in (Pb,Cd)-1212 superconductor by $NaNO_3$ addition. The $NaNO_3$ has been found to have the high oxygen-potential and its decomposition products seem to induce liquid phase sintering resulting in a denser structure with an increase in critical current density of 123 superconductor. The 1212 type superconductors are of special interest due to their close structural relationship with other superconductors having T_c of > 90K and its chemi-

cal flexibility provides a means to tailor new superconducting materials with improved flux pinning properties. High purity $NaNO_3$ (3-8 mol.%) was added during the final sintering of $(Pb_{0.5}Cd_{0.5})Sr_2(Y_{0.67}Ca_{0.4})Cu_2O_{7-8}$ at 850ϒC in flowing O_2 atmosphere. The composite formed with 5 mol.% additive exhibits an enhancement of T_c to 10K (as measured by electric al resistivity measurement) with Tc,onset at 55K while the undoped superconductor exhibits $T_{c,onset}$ at 45K. The addition also leads to the formation of 1212 phase without $SrCuO_2$ impurity. The lattice constants and cell volume of the superconducting phase in the composite show a marginal increase compared to the pure superconductor. Melting point of the superconductor was found to be lowest at 974ϒC with 5 mol.% $NaNO_3$ addition. To establish that $NaNO_3$ may be a key element in 1212 superconductor fabrication, further characterization of the composites are under progress.

HIGH TEMPERATURE COATINGS III: Thermal Barrier Coatings - II

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee; Jt. ASM International: Materials Science Critical Technology Sector/TMS Structural Materials Division, Corrosion and Environmental Effects Committee

Program Organizers: Janet Hampikian, Georgia Tech, School of Mats. Sci. & Eng., Atlanta, GA 30332-0245 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Ctr. for Laser Applications, Tullahoma, TN 37388 USA

Monday PM Room: 19
March 1, 1999 Location: Convention Center

Session Chairs: James A. Nesbitt, NASA Lewis Research Center, Mats. Div., Cleveland, OH 44135 USA; Woo Y. Lee, Stevens Institute of Technology, Mats. Sci. and Eng., Hoboken, NJ 07030 USA

2:00 PM INVITED PAPER

MECHANISMS FOR THE FAILURE OF ELECTION BEAM PHYSICAL VAPOR DEPOSITED THERMAL BARRIER COATINGS INDUCED BY HIGH TEMPERATURE OXIDATION: M. S. Stiger[1]; N. M. Yanar[1]; *F. S. Pettit*[1]; G. H. Meier[1]; [1]University of Pittsburgh, Dept. of Mats. Sci. and Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Yttria stabilized zirconia (YSZ) thermal barrier coatings (TBCs) fabricated via electron beam physical vapor deposition (EBPVD) provide some unique properties for aerofoil applications. Such coatings are usually deposited on diffusion aluminide or MCrAlY bond coats on superalloy substrates. During deposition of the YSZ-TBC on the bond coat, a thermally grown oxide (TGO) consisting primarily of α-Al_2O_3 is formed between the YSZ-TBC and the bond coat. The lives of these TBCs in oxidizing environments is determined by the interplay of the stored elastic energy driving spallation versus the interfacial toughness of the TGO at the TGO-bond coat interface since failure of these TBCs occurs at this interface. The microstructures of EBPVD-TBCs in the as processed and exposed conditions have been documented using a variety of techniques to attempt to determine and describe the failure mechanisms of EBPVD-TBCs. Results will be presented to show that some failure processes proposed previously do not cause TBC failure. It will also be shown that the failure mechanisms for TBCs are dependent upon the bond coat fabrication procedure.

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SPALLING FAILURE OF A THERMAL BARRIER COATING ASSOCIATED WITH ALUMINUM DEPLETION IN THE BOND-COAT: E. A. G. Shillington[1]; *D. R. Clarke*[1]; [1]University of California, Mats. Dept., Santa Barbara, CA 93106-5050 USA

A plasma-sprayed thermal barrier coating is observed to spall after oxidation at 1121ϒC from a CoNiCrAlY bond-coated superalloy at the interface between the thermally grown oxide and the zirconia thermal barrier coating (TBC). Phase characterization by photostimulated luminescence and X-ray diffraction, as well as microstructural characterization by scanning electron microscopy indicates that the spalling is associated with the conversion of the initially formed α-alumina thermally grown oxide to α-chromia and a (Co,Ni)(Cr,Al) spinel. It is proposed that this occurs as a result of a three-step process: First, depletion of aluminum from the bond-coat alloy; second, cracking of the convoluted alumina scale driven by thermal stresses associated with thermal cycling; third, oxygen penetrate through the alumina scale causing the continued enrichment of the oxide by Cr, Co and Ni with oxidation. On the basis of these observations, it is proposed that photostimulated luminescence will be a viable non-destructive tool for evaluating the progress of the reaction and determining when alumina formation is superceded by the onset of the detrimental chromia formation.

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HOT CORROSION OF NICKEL-BASE ALLOYS IN A BIOMASS DERIVED FUEL SIMULATED ATMOSPHERE: *Christoph Leyens*[1]; Bruce A. Pint[1]; I. G. Wright[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6158 USA

Biomass derived fuels as a renewable source of energy are of increasing interest for future power generation gas turbines. Based on its origin, biomass derived fuel contains impurities which are different from fuels derived from coal, oil or natural gases. Whereas numerous studies have addressed the corrosion attack of high temperature resistant materials for gas turbines by conventional fuels, the materials problems arising with impurities from biomass derived fuels are not yet well studied. Therefore, an initial assessment of nickel-base superalloys with and without coatings and cast versions of standard aluminide, platinum aluminide and MCrAlY is being conducted. Specimens were exposed in a standard 1h cyclic hot corrosion test at 950ϒC with a conventional salt composition and with modified salts relevant to biomass fuels. The objective is to assess the extent and the mechanisms of biomass-related hot corrosion attack relative to conventional hot corrosion.

3:00 PM

THE EFFECT OF SALT CHEMISTRY ON SUPERALLOY CORROSION: *John G. Smeggil*[1]; [1]United Technologies Research Center, 411 Silver Lane, East Hartford, CT 06108 USA

In service, corrosive salt deposits can deposit onto hot section turbine hardware. Once there and if the temperatures are sufficiently high, the salts can become molten to then produce hot corrosion. Two sources of the molten salts have been proposed. According to one mechanism, sodium sulfate forms in combustor sections and then subsequently condenses onto cooler turbine hardware surfaces. A second mechanism suggests that deposits shed from compressor sections impact these surfaces to form deposits there. Results of experiments involving the elevated temperature exposure of two superalloys (IN792 and PWA1484) both to sodium sulfate and to salt chemistries simulating compressor deposits at a variety of temperatures will be compared and contrasted. This work was funded by the Office of Naval Research, Contract No. N00014-97-C-0180.

3:20 PM BREAK

3:40 PM INVITED PAPER

THE AGTSR CONSORTIUM: TBC RESEARCH HIGHLIGHTS: *D. B. Fant*[1]; [1]Clemson University, South Carolina Energy R&D Center, 386-2 College Ave., Clemson, SC 29634 USA

The Advanced Gas Turbine Systems Research (AGTSR) Consortium currently has 95 performing member institutions, representing 37 states, and eight cost-sharing gas turbine manufacturers/ users as industrial members. This presentation will briefly discuss the mission of AGTSR and describe the various research and educational opportunities available through the Consortium. The second part of the presentation will focus on highlighting the active research projects being supported by AGTSR in the area of Thermal Barrier Coatings (TBC's). Since 1993, AGTSR has awarded eleven research contracts in TBC's - seven of which are still active. For these on-going projects, the issues and results to be discussed pertain to TBC bond strength and stress measurements, chemical and

mechanical instabilities at TBC interfaces, advanced coating techniques, NDI for TBC's, and life prediction. In addition, a summary of the AGTSR Metallic Coatings Specialty Workshop will be provided. This Workshop was held in April, 1998 at the Stevens Institute of Technology, and focused on the long-term pre-competitive R&D challenges associated with aluminide and platinum aluminide coatings for aircraft and industrial gas turbines. Finally, the new TBC projects selected by AGTSR from the 98RFP announcement will be described. It is anticipated that these new research efforts will begin work in February, 1999.

4:00 PM

FORMATION OF GRAIN BOUNDARIES AND DEVELOPMENT OF TEXTURE IN A DIFFUSION NiAl COATING PRODUCED BY CHEMICAL VAPOR DEPOSITION: *W. Y. Lee*[1]; G. Y. Kim[1]; J. A. Haynes[2]; [1]Stevens Institute of Technology, Dept. of Mats. Sci. and Eng., Castle Point on Hudson, Hoboken, NJ 07030 USA; [2]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6063 USA

Recent isothermal and cyclic oxidation results show that scale spallation initiates preferentially along grain boundaries in the diffusion NiAl and (Ni,Pt)Al coatings produced by chemical vapor deposition (CVD). This observation implies that the ultimate performance of these diffusion coatings for use as bond coats in thermal barrier coating applications may be limited by the presence of grain boundaries. In this study, morphological changes occurring at the (100) surface of a single crystal Ni-based alloy during the very early stage of the NiAl growth were examined to elucidate the mechanism of the formation of grain boundaries and to follow the development of texture in the substrate-coating interface region. The prospect of proactively tailoring the microstructure of the diffusion aluminide coatings by procedural modifications of the CVD aluminizing process will be discussed. Research sponsored by Advanced Gas Turbine Systems Program, DOE Office of Industrial Technologies, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

4:20 PM INVITED PAPER

DEGRADATION OF ELECTRON BEAM PHYSICALLY VAPOR DEPOSITED THERMAL BARRIER COATINGS: THE ROLE OF BOND COAT: *S. Bose*[1]; [1]Pratt & Whitney, M.S. # 114-45, 400 Main St., East Hartford, CT 06108 USA

Thermal Barrier Coatings (TBC) are used in gas turbine engines to reduce surface temperatures of high-pressure turbine components. In the TBCs used by Pratt & Whitney for blade or vane application, the substrate is a nickel or cobalt base single crystal superalloy. The bond coat has a general composition of NiCoCrAlY with small amounts of other oxygen active elements. The ceramic consists of partially stabilized zirconia, deposited by Electron Beam Physical Vapor Deposition (EB-PVD). The spallation life of the TBC is controlled primarily by the oxidative and mechanical behavior of the bond coat. During processing, a thin, thermally grown oxide (TGO) scale, predominantly alumina, forms at the interface between the bond coat and the ceramic layer. This GTO rides on the bond coat, which is prone to creep at every transient excursion of the engine or rig test cycle. Due to the thermal exposure, significant level of time dependent residual stress develops at the vicinity of the TGO. The residual stress, changes in the chemistry and segregation within the oxide scale and TGO/metal interface play important roles in the de-cohesion of the TGO from the creeping bond coat. Test data from rig and field experience support this mechanism.

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EFFECT OF Al_2O_3 DIFFUSION BARRIER ON MICROSTRUCTURE AND OXIDATION BEHAVIOR OF THERMAL BARRIER COATING PREPARED BY DETONATION-GUN SPRAYING: *Young-Mok Rhyim*[1]; Jing Hong Kim[1]; Hyun-Woo Jin[1]; Chan-Gyung Park[1]; Moon-Chul Kim[2]; [1]Pohang University of Science and Technology, Center for Advanced Aerospace Materials, San 31 Hyoja-dong Nam-ku, Pohang, Kyungbuk 790-784 Korea; [2]Research Institute of Industrial Science and Technology, Dept. of New Mats., San 32 Hyoja-dong Nam-Ku, Pohang, Kyungbuk 790-600 Korea

Many studies have indicated the severity of oxidation of the bond coat is the dominant degradation mechanism of the thermal barrier coating (TBC) performing at elevated temperature. The oxidation of bond layer is generally caused by oxygen diffusion through ZrO_2 ceramic layer, therefore, several methods applying Al_2O_3 diffusion barrier have been proposed to reduce the oxygen permeation. In this study, the two types of Al_2O_3 diffusion barrier were applied between MiCrAlY bond coat and partially stabilized zirconia (PSZ) top coat layer: 1) Al_2O_3 oxide scale was formed on the surface of MCrAlY bond coat by pre-heat treatment at 1000°C before deposition of PSZ ceramic layer, 2) the intermediate Al_2O_3 layer was deposited by detonation-gun spraying with a thickness of 50, 150 and 250 μm. The TEM microstructure observation revealed that the duplex TBC prepared by detonation gun method was consisted of three layers, those are PSZ layer, PSZ+NiCrAlY layer and NiCrAlY layer. And the Al-rich amorphous was found in the PSZ+NiCrAlY layer. As the pre-heat treatment time increased, lateral and vertical cracks were developed in the PSZ layer after the spraying by detonation-gun. The intermediate Al_2O_3 layer reduced effectively the oxidation of bond coat, however, it spalled in the early stage of the cyclic oxidation test. The variation of interfacial microstructure and oxidation behaviors depending on the two different Al_2O_3 diffusion barriers has also been discussed.

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OXIDATION RESISTANCE OF CERAMIC COATINGS TREATED BY IMPULSE PLASMA FLUXES: *S. F. Korablev*[1]; T. R. Korableva[2]; A. R. Kopan[2]; [1]National Technical University, 11 Polyarnaya St., Apartment 61, Kiev-201 254201 Ukraine; [2]Instute of Problems of Materials Science NAS, Kiev Ukraine

The resistance to oxidation of two-layer coatings from NiCrAlY heat-resistant alloy with ZrO_2-Y_2O_3 thermal barrier layer was studied in air at 1273 K. The coatings were sprayed by gaseous thermal method. Then these coatings were treated by impulse plasma fluxes. A plasma injector provided a power density of 10^5 to 10^7 W cm^2, which is sufficient for to melt the ZrO_2 surface layer. It was established that the oxidation process after an impulse plasma treatment is decelerated by 1.5 to 1.7 times. This is due to the reduction of the open porosity of the surface ceramic layer treated. During an impulse plasma treatment, ZrO_2 large crystals with horizontal-plane structure were formed and residual stresses were reduced. The destruction of coatings resulted from the formation of (Ni, Cr, Al, Y)$_x$O$_y$ scale on the boundary of heat-resistant alloy - thermal barrier layer. The growth rates of the scale were 2 x 10^{-8} m/h and 3.1 x 10^{-8} m/h for treated and untreated coatings respectively. After 160 h exposure, full destruction of the untreated coatings occurred, while coatings treated by impulse plasma fluxes survived after 240 h exposure.

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MECHANICAL PROPERTY CHANGES IN ZrO_2 IMPLANTED WITH Y IONS: *Jizhong Zhang*[1]; Xiaoyan Ye[2]; Chong Guan[1]; [1]Tsinghua University, Dept. of Mats. Sci. and Eng., Beijing 100084 China; [2]Tsinghua University, Dept. of Chem., Beijing 100084 China

Zirconia ceramics were modified by Y-ion implantation in a MEVVA (Metal Vapor Vacuum Arc) implanter. The influence of implantation parameters was investigated by varying the ion dose over a wide range. The samples were implanted with 140 KeV Y ions with doses from $8x10^{16}$ to $1.8x10^{18}$ Y/cm^2. The experimental results suggested that the implantation dose strongly influences the microhardness, flexural strength, and fracture toughness of zirconia. With increasing ion dose, the microhardness and flexural strength of as-implanted zirconia samples increased at first, and then decreased at high doses (typically greater than $2x10^{17}$ Y/cm^2). As comparing with the hardness and flexural strength, the relative change of fracture toughness of as-implanted zirconia samples showed quite the opposite status with increasing ion dose.

HUME ROTHERY SYMPOSIUM TO HONOR M. HILLERT; ALLOY EFFECTS ON MIGRATING INTERFACES: Session I

Sponsored by: Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division, Alloy Phases Committee; ASM International: Materials Science Critical Technology Sector,Thermodynamic Activities & Phase Equilibria Committee
Program Organizers: Y. Austin Chang, University of Wisconsin, Dept. of Mats. Sci. & Eng., Madison, WI 53706-1595 USA; Ray Y. Lin, University of Cincinnati, Dept. of Mats. Sci. & Eng., Cincinnati, OH 45221-0012 USA

Monday PM Room: 14A
March 1, 1999 Location: Convention Center

Session Chairs: Y. A. Chang, University of Wisconsin, Dept. of Mats. Sci. & Eng., Madison, WI 53706-1595 USA; J. K. Lee, Michigan Technological University, Houghton, MI 49931-1295 USA

2:00 PM OPENING REMARKS - Y. A. Chang, University of Wisconsin, Dept. of Mats. Sci. & Eng., Madison, WI 53706-1595 USA

2:05 PM KEYNOTE
ALLOY EFFECTS ON MIGRATING INTERFACES: *Mats Hillert*[1]; [1]KTH (Royal Institute of Technology), Dept. of Mats. Sci. and Eng., Stockholm SE-10044 Sweden

Interactions of alloying elements with migrating interfaces are of considerable importance for many phenomena in alloys. Important examples are solute drag in one-phase materials, massive transformation, alloy effects in the gamma to alpha transformation in Fe-C-X alloys, trapping in solidification and diffusionless solidification, DIGM, liquid film migration and discontinuous precipitation. The physical basis of these effects are closely related and should be regarded as a single topic of theoretical work. The lecture will review the theories that have been developed in these various areas and similarities will be emphasized.

2:50 PM INVITED PAPER
ON THE INFLUENCE OF ALLOYING ELEMENTS UPON THE KINETICS OF THE PROEUTECTOID FERRITE REACTION: *H. I. Aaronson*[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., Pittsburgh, PA 15213 USA

The orders of magnitude differences in the diffusivities of carbon and of substitutional alloying elements, X, in austenite at temperatures of interest have evoked a rich theoretical literature to compose these differences into a coherent picture of the influence of X upon the growth kinetics of proeutectoid ferrite. Prof. Mats Hillert has long been the leader of these efforts. Two principal models of these effects have survived: the orthoequilibrium one, requiring local equilibrium of both C and X at a:g boundaries, and the paraequilibrium one, involving only carbon equilibrium across these boundaries. Three types of experimental test have been made of these models, all of which were conducted on grain boundary ferrite allotriomorphs. Except under rarely duplicated circumstances, the orthoequilibrium model has been repeatedly found to be inappropriate. The paraequilibrium model yields improved comparisons with experiment but also reveals disquieting discrepancies in respect of growth kinetics. A growing consensus now tends to ascribe these discrepancies to a solute drag-like effect (SDLE). Experimental and theoretical progress recently made in characterizing and describing this effect will be recounted in some detail.

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3:40 PM INVITED PAPER
SIMULATION OF THE SOLUTE DRAG EFFECT IN MULTICOMPONENT SYSTEMS: *Bo Sundman*[1]; [1]Royal Institute of Technology, Stokholm Sweden

The mobility of grain boundaries and phase interfaces may depend strongly on alloying additions, also in so minor amounts that they can be considered as impurities. An attempt to quantify this effect was proposed with the solute drag theory. In this theory the segregation to the interface was assumed to move with the interface and create a "drag" making the interface less mobile. The theory also predicted that at high driving forces the interface may "break away" from the segregated atoms and the interface may then move at high speed. The theoretical treatment has been limited to binary systems in all cases so far but recently new software has been developed to treat the combined effects of two or more alloying elements. Some calculations for typical steels, Fe-Mn-C, Fe-Cr-C and Fe-Cr-Mn-C will be presented and compared to experimental data. The calculations make use of a database for the thermodynamic description of the systems. The influence of the diffusion coefficient in the interface on the solute drag will also be discussed.

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SOLUTE DRAG EFFECTS DURING PHASE TRANSFORMATIONS IN MULTICOMPONENT ALLOYS: *John Agren*[1]; [1]Royal Institute of Technology, Dept. of Mats. Sci. and Eng., Stockholm S-100 44 Sweden

The solute drag theory was developed in detail for binary systems more than 20 years ago and has been applied to grain boundary motion as well as the motion of phase interfaces during phase transformations. Hillert and Sundman showed that the solute drag effect actually promotes the transition to a partitionless transformation. In this presentation the theoretical aspects of solute drag, in ternary and higher order systems, will be discussed and compared with experimental observations. The multicomponent diffusion coupling effects, and the deviation from local equilibrium at the migrating phase interface will be analyzed. In particular the hypothesized paraequilibrium growth mode of ferrite during cooling after austenitization of steels will be analyzed.

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Alloy, Microstructure and Process Design

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging & Interconnection Materials Committee
Program Organizers: Gautam Ghosh, Northwestern University, Dept. of Mats. Sci., Evanston, IL 60208-3108 USA; Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; Rao Mahidhara, Cypress Semiconductor Corporation San Jose, CA 95134 USA; Ephraim Suhir, Bell Labs., Murray Hill, NJ 07974 USA

Monday PM Room: 17A
March 1, 1999 Location: Convention Center

Session Chairs: S. Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; S. K. Lahiri, National University of Singapore

2:00 PM INVITED PAPER
THERMODYNAMIC DATABASE FOR PHASE DIAGRAMS FOR MICRO-SOLDERING ALLOYS: I. Ohnuma[1]; X. J. Liu[1]; H. Ohtani[1]; *K. Ishida*[1]; [1]Tohoku University, Dept. of Mats. Sci., Graduate School of Eng., Aoba-yama 02, Sendai 980-8579 Japan

Thermodynamic database for micro-soldering alloys systems, which consist of the elements Pb, Bi, Sn, Sb, Cu, Ag, and Zn has been developed by CALPHAD method. The thermodynamic parameters for describing experimental data on phase boundary compositions and thermochemical properties such as activity, heat of mixing and enthalpy of forma-

tion. The database provides the liquidus and solidus surfaces, isothermal and vertical section diagrams, the mole fraction of phase constituents etc. for multi-component soldering alloys. The surface tension and viscosity of the liquid phase can also be calculated. The database will be very useful for developing Pb-bearing and Pb-free solders.

2:30 PM INVITED PAPER

ALLOY DESIGN AND INVESTIGATION ON THE PHASE EQUILIBRIUM IN LOW-TEMPERATURE SOLDER, Sn-Bi-In SYSTEM: *Hyuck-Mo Lee*[1]; Choong-Un Kim[2]; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. and Eng., Kusung-Dong 373-1, Taejon 305-701 Korea; [2]The University of Texas at Arlington, Materials Science and Engineering, Arlington, TX USA 76019-0031

This study investigates on the phase equilibrium in the low-temperature solder, Sn-Bi-In ternary alloy system, performed both by theoretical and experimental methods. Following the regular solution model and a standard thermo-chemical calculation, theoretical evaluation of the phase equilibrium in the entire ternary system is conducted. The thermodynamical parameters required for the calculation are initially obtained by fitting the model into existing data available from the prior studies. The theoretical results are then validated and further improved by experimental work in which alloys with several critical compositions are chosen and examined. In the experimental work, the differential scanning calorimetry (DSC), the X-ray diffraction (XRD) and the energy dispersion spectrometry (EDX) are jointly used to identify the type of the phase present in the microstructure and the transition temperatures. The resulted phase diagram agrees well both to the existing data and to the data from the current experiment. However, different from previous findings, this study finds non-binary nature of the Sn-BiIn and Sn-$BiIn_2$ quasi-binaries, and seven invariant reactions, one eutectic and six peritectic reactions. The phase reaction scheme (scheil diagram), the ternary liquid projection and the phase diagram, covering entire compositional ranges, are established. Suggestion for the potential solder alloys are also made based on the phase diagram.

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DESIGN OF IMPROVED SOLDER ALLOYS THROUGH MICROSTRUCTURE CONTROL: *S. Jin*[1]; H. Mavoori[1]; [1]Bell Laboratories, Lucent Technologies, 700 Mountain Ave., Murray Hill, NJ 07974 USA

The properties of solder alloys and reliability of solder joints are significantly affected by microstructural features such as grain size, phase distribution, and precipitate morphology. A finer grain size, smaller precipitates, and a uniformity in phase, precipitate and grain size distribution are often beneficial for improving the strength and ductility as well as the resistance to fatigue and creep failures. The control of microstructure is accomplished by modifications in alloy chemistry and processing conditions. Several examples of microstructural control in lead-containing and lead free solder systems will be discussed. More forward-looking approaches for further control of microstructure in solder alloys and composites, e.g., by distribution of nano-scale dispersoid particles will also be discussed.

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Zn-Al-Mg-Ga ALLOY AS Pb-FREE SOLDER FOR DIE-ATTACHING USE: *T. Shimizu*[1]; H. Ishikawa[1]; I. Ohnuma[2]; K. Ishida[2]; [1]Sumitomo Metal Mining Company, Ltd., Research and Development Center, Electronics Division, Suehiro-cho 1-6-1, Ohme-shi 198-8601 Japan; [2]Dept. of Mats. Sci., Graduate School of Engineering, Tohoku University, Aobayama 02, Sendai 980-8579 Japan

Zn-based alloys have been investigated to replace Pb-5%Sn solder for die-attaching use. We have found that a Zn-4%Al-3%Mg-3%Ga alloy has 310°C of solidus, and 350°C of liquidus. This alloy has also twice the thermal conductivity and two-third of the thermal expansion rate of Pb-5%Sn solder. A die-attaching test was done with 0.2 mm thick preforms of this alloy, Ag-plated lead-frames, and Au-plated dummy dies. Good die-attaching with a small amount of voids can be achieved at 320°C or higher. In a subsequent heat cycle test, no failure was observed until 1000 cycles between -65°C and 150°C. The Zn-4%Al-3%Mg-3%Ga alloy shows poor workability at room temperature, and it is difficult to obtain thin plates or wires of this alloy. How to apply this alloy to actual die-attaching systems is a problem to be solved.

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MICROSTRUCTURAL ENGINEERING OF SOLDERS: *K. N. Subramanian*[1]; Thomas R. Bieler[1]; James P. Lucas[1]; [1]Michigan State University, Dept. Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

The reliability of solders, especially in severe service conditions, is strongly influenced by their behavior under creep, thermomechanical fatigue, creep-fatigue interactions and the effects of aging on these properties. Among the various methods used to improve reliability, one that holds significant promise is incorporation of in-situ, compatible reinforcements. The elemental composition of in-situ incorporated reinforcements is similar to phases typically present at the solder/substrate interface, and therefore is deemed compatible with the solder alloy system. By suitable methodology, solder composites with uniformly distributed reinforcements have been fabricated as solder preforms. Upon melt-reflow in time scales typical of manufacturing processes, solder joints that have the in-situ reinforcements retard aging and stabilize mechanical properties. The presence of these reinforcements homogenizes deformation across the joint, resulting in a lower apparent work hardening rate, enhanced ultimate strength at lower strain rates, and improved ductility of the solder by as much as a factor of two. Creep strain rates at lower temperatures were reduced by a factor of 100-1000. Thus with the composite solder, creep resistance is gained while improving ductility and retaining comparable flow stress characteristics present in the non-composite solder. The reinforcements also retard the growth of the intermetallic layer at the solder/substrate interface, and this tends to improve mechanical fatigue behavior. These composite solders have similar wetting behavior as the corresponding non-composite ones and therefore should not pose any additional concerns in the manufacture of the electronic systems.

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SOLIDIFICATION MODELLING OF EUTECTIC Sn-Ag SOLDER WITH AND WITHOUT INTENTIONALLY INCORPORATED Cu6Sn5 REINFORCEMENTS: *Alan W. Gibson*[1]; K. N. Subramanian[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Solidification of eutectic Sn-Ag solder, with and without Cu_6Sn_5 reinforcements, on copper substrates, was investigated at various cooling rates. The morphology, size and the amount of the constituents were examined as a function of cooling rate and distance from the solder/substrate interface. Cu_6Sn_5 intermetallics present within the solder matrix, either as occasionally observed extensions from the interface layer or as intentionally added component, were found to act as nucleating sites. Such a process tends to break up the normally observed dendritic morphology in rapidly solidified solders, commonly encountered in electronic interconnects. Observed features are explained on the basis of micro- and macroscopic solidification models.

5:00 PM INVITED PAPER

LOW THERMAL EXPANSION COPPER COMPOSITES WITH NEGATIVE CTE METALLIC ELEMENTS: *H. Mavoori*[1]; S. Jin[1]; [1]Bell Laboratories, Lucent Technologies, 700 Mountain Ave., Murray Hill, NJ 07974 USA

In high power dissipation electronic packages, heat sinks are essential for preventing thermal damage to heat-sensitive components on the silicon chip. However, the heat sink materials commonly used today such as Cu and Al alloys have much higher coefficient of thermal expansion (CTE) than Si. CTE mismatch between the various materials in an electronic package can lead to stresses that can trigger complex failure mechanisms seriously degrading the device reliability and lifetime. Therefore, it is highly desirable to minimize the CTE mismatch by developing new heat sink materials with CTEs close to that of Si. In this work, low thermal expansion copper composites with CTE as low as 4 ppm/ °C have been fabriated by employing a negative thermal expansion alloy (equiatomic Ti-Ni with CTE ≅ -21 ppm/ °C). The use of negative CTE elements, especially those with very large CTE values offers an attractive route for controlling the thermal expansion behavior of various metallic and non -metallic materials.

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SINTERING OF HIGH TEMPERATURE SOLDER MATERIALS: *Mark A. Palmer*[1]; [1]Virginia Commonwealth University, Mech. Eng., 601 West Main St., P.O. Box 843015, Richmond, VA 23284-3015 USA

Eutectic lead-tin has been the solder of choice throughout the history of the electronics industry. Alternatives to this material are being considered because of environmental concerns, as well as the strength and temperature limitations of eutectic lead-tin. Most alternative alloys have significantly higher melting temperatures. This means that if molten solder is to be used in the manufacture of electronics assemblies the processing temperature will have to significantly increase. This in turn will require the replacement of much of the manufacturing infrastructure. It is proposed to use sintering as a means of forming solder joints, from a higher temperature solder, yet without increasing the processing temperature. The processing of eutectic tin-silver (T_m=221ϒC) solder joints prepared by sintering will be discussed. The mechanical properties of these joints will be presented. Finally, the implications for the electronics industry will be reviewed.

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Twinning in Titanium Alloys

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

Program Organizers: S. Ankem, University of Maryland, Dept. of Mats. & Nuclear Eng. College Park, MD 20742-2115 USA; Chandra Pande, Naval Research Lab, Mats. Sci. & Tech. Div., Washington, D.C. 20375-5000 USA

Monday PM Room: 17B
March 1, 1999 Location: Convention Center

Session Chairs: Bruce A. MacDonald, National Science Foundation, Mats. Research Div., Arlington, VA 22230 USA; Sreeramamurthy Ankem, University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742-2115 USA

2:00 PM INVITED PAPER
RECENT DEVELOPMENTS IN GROWTH KINETICS OF DEFORMATION TWINS IN BULK METALLIC MATERIALS: *Sreeramamurthy Ankem*[1]; Charles A. Greene[1]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742-2115 USA

Generally it is believed that the growth rates of deformation twins in bulk metallic materials are very high, i.e., in the vicinity of the speed of sound. However, recently it has been observed that the growth rates can be very much lower. This time dependent twinning phenomenon was observed in both hcp and bcc titanium alloys. The slow growth of the twins was shown to be a significant factor in giving rise to room temperature creep where the creep test was conducted at a stress level of 95% yield stress. The mode of time dependent twinning in compression was found to be different from that in tension in alpha titanium. It has been further shown that the extent of time dependent twinning decreases with decrease in grain size. In this investigation, these developments will be critically reviewed and an attempt will be made to rationalize these practically important and scientifically interesting observations. This work is being supported by the Office of Naval Research under grant No. N000149610819.

2:35 PM INVITED PAPER
DISLOCATION CORE NUCLEATION OF TWINS IN HCP METALS: *James R. Morris*[1]; K.-Y. Chen[1]; Kai-Ming Ho[1]; *Man H. Yoo*[2]; [1]Iowa State University, Ames Laboratory, A524 Physics, Ames, IA 50011-3020 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

Secondary deformation modes in hcp metals play a crucial role in determining the ductility of the metals. These modes are similar to many complex deformation phenomena in intermetallics. There is a competition between twinning and slip modes with common crystallographic elements, and the Burgers vector associated with the slip mode is much larger than typical interatomic distances, leading to a complex "synchroshear" slip process. Core phenomena play a critical role in the process. We have examined the dislocation core structures with b=<c+a> using large scale atomistic simulations with many-body potentials. We will present results on several dislocation line orientations. Our results show that a sessile basal plane splitting of the edge dislocation associated with {11-22} slip results in the generation of a twin nucleus near the dislocation core, which grows under tensile stress along the c-axis.

3:10 PM INVITED PAPER
THE INFLUENCE OF INTERSTITIAL CONTENT, TEMPERATURE, AND STRAIN RATE ON DEFORMATION TWIN FORMATION: *George T. Gray*[1]; George C Kaschner[1]; Shuh-Rong Chen[1]; Thomas Mason[2]; [1]Los Alamos National Lab, MST-8, MailStop G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Lab, MST-6, MailStop G755, Los Alamos, NM 87545 USA

In this study, the influence of oxygen content, temperature, and strain rate on the propensity for deformation twin formation and texture evolution in two grades of α-titanium and α-zirconium is presented. The influence of strain rate, temperature, and deformation on twin formation and texture evolution of high-purity (low-interstitial) Ti and commercial-grade (high-oxygen) Ti and Zr was probed utilizing quasi-static and Hopkinson-Bar loading. Suppression of deformation twin formation in the high-oxygen contents Ti and Zr is seen to simultaneously correlate with higher yield strengths and lower Stage-II work-hardening rates. The influence of interstitial content on the kinetics of twin formation, texture evolution, and work hardening is contrasted in light of previous literature studies. The influence of deformation twinning on the overall mechanical behavior and constitutive modeling description of low-symmetry metals and alloys is discussed. Work performed under the auspices of the U.S. Dept. of Energy.

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AN ELECTRON MICROSCOPE STUDY OF TWIN PROPAGATION IN TWO-PHASE TITANIUM: *Fritz Appel*[1]; [1]GKSS Research Center, Institute for Materials Research, Max-Planck-Str., Geesthacht D-21502 Germany

The importance of twinning has long been recognized as deformation mechanism of gamma-TiAl. However, many aspects of the process are not yet solved. This concerns in particular the nucleation and growth of twins and interactions with glide obstacles. The paper presents an electronmicroscope study of twin propagation in two-phase titanium aluminide alloys. The generation of mechanical twins was found to be closely related to the structural features of lamellar interfaces present in the material. These are dense arrangements of mismatch structures and high coherency stresses. Misfit dislocations with a favourable core configuration apparently can easily be rearranged into twin embryos. The interaction of deformation twins with perovskite precipitates was characterized in order to assess the potential of precipitation hardening for improving the high temperature strength of the material.

4:20 PM INVITED PAPER
THE INFLUENCE OF DEFORMATION TWINNING ON THE HIGH-STRAIN-RATE RESPONSE OF ALPHA-TITANIUM: D. R. Chichili[2]; *K. T. Ramesh*[1]; K. J. Hemker[1]; [1]The Johns Hopkins University, Dept. of Mech. Eng., 122 Latrobe Hall, 3400 Charles St., Baltimore, MD 21218-4316 USA; [2]Currently at Fermi National Accelerator Laboratory, P.O. Box 500, M.S. 316, Batavia, IL 60510 USA

The influence of deformation twins on the high-strain-rate mechanical response of alpha-titanium is examined. The dynamic mechanical behavior of alpha-titanium has been evaluated using high-strain-rate (10^2 to $7x10^3$ s^{-1}) compression, torsion and tension testing with compression, torsion and tension Kolsky bars, as well as very high-rate (10^4 to 10^5 s^{-1}) shearing under pressure using the pressure-shear plate impact technique. At the macroscopic level, alpha-titanium displays

substantial rate sensitivity of the flow stress and pronounced strain hardening. At the microscopic level, both dislocations and twins are observed; the density of twins increases with both strain and strain rate and is shown to be a unique function of the flow stress. It is demonstrated that the degree of twinning does not determine the flow stress of the material at high rates. Although dislocation motion accounts for the majority of plastic deformation, twin-dislocation interactions play an important role in strain hardening. The development of deformation twinning is also shown to be a function of the multiaxial stress state, and as a consequence the plastic flow of the material cannot be described in terms of conventional J_2-flow theory.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Fundamentals

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Synthesis & Processing, Structural Materials Committee, Titanium Committee
Program Organizers: Young-Won Kim, UES, Inc., Mats. & Proc. Div., Dayton, OH 45432-1805 USA; Dennis M. Dimiduk, Wright-Patterson AFB, WL/MD, WPAFB, OH 45433 USA; Michael H. Loretto, University of Birmingham, IRC, Birmingham B15 2TT UK

Monday PM Room: 8
March 1, 1999 Location: Convention Center

Session Chairs: David P. Pope, University of Pennsylvania, Dept. of Mats. Sci. and Eng., Philadelphia, PA 19104-6272 USA; Shigehisa Naka, ONERA, BP 72, Chatillon Cedex 92322 France

2:00 PM INVITED PAPER
THE INFLUENCE OF SOLID SOLUTIONS ON FLOW BEHAVIOR IN GAMMA-TiAl:*Christopher Woodward*[1]; Scott A. Kajihara[1]; Satish I. Rao[1]; Dennis M. Dimiduk[2]; [1]UES, Inc., Mats. Research Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Mats. and Manuf. Directorate, MLLM Bldg. 655, 2230 Tenth St., Ste 1, Wright Patterson AFB, OH 45433-7817 USA

Modifications of alloy chemistry are often used to tailor the intrinsic flow behavior of structural materials. Models of solution strengthening, high temperature yield stress and creep must relate the effects of chemistry to the mechanisms which influence these material properties. In ordered alloys, additional information regarding the crystallographic site occupancy of ternary elements is required. 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 γ-TiAl. Size and modulus misfit parameters are calculated and the interaction of these defects with a dissociated ordinary screw dislocation evaluated within anisotropic elasticity theory. The derived interaction strength is then related to solid solution strengthening for these defect centers. Predicted solid solution effects are in good agreement with experimental observations for the binary alloy.

2:30 PM
BOND ORDER POTENTIAL FOR ATOMISTIC SIMULATION OF TiAl: *Stefan Znam*[1]; *Duc Nguyen-Manh*[2]; *Vasek Vitek*[1]; *David G. Pettifor*[2]; [1]University of Pennsylvania, Dept. of Mats. Sci. and Eng., 3231 Walnut St., Philadelphia, PA PA 19104-6272 USA; [2]Oxford University, Dept. of Mats., Parks Rd., Oxford, Oxfordshire OX1 3PH UK;

A new bond-order potential for Ti-Al alloys has been constructed in the framework of a Tight-Binding description of the binding energy. In this scheme the energy consists of three parts: the bond part that comprises the 3d (Ti) and 3p (Al) electron contributions to bonding, the central-force many-body part that reflects the environmental dependence of overlap repulsion and a pair-wise contribution. A {\it transferable} set of the tight-binding parameters entering the bond part has been generated by first-principles calculations and incorporates the angular character of bonding arising from Ti-Al hybridization effects. The {\it negative Cauchy pressures} of gamma-TiAl are adequately reproduced owing to inclusion of the environmentally dependent term. The potential is tested and compared with central-force potentials of the Finnis-Sinclair type by examining the mechanical stability of the $L1_{0}$ lattice with respect to a variety of large deformations. It is applied in a study of dislocation behaviour in TiAl.

2:50 PM INVITED PAPER
DISLOCATION INTERACTIONS AND DEFORMATION MECHANISMS IN TWO-PHASE ALLOYS BASED ON TiAl: *Hamish L. Fraser*[1]; *Jorg Michael Wiezorek*[2]; Xiao-Dong Zhang[1]; *Michael J. Mills*[1]; [1]Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]University of Pittsburgh, Dept. of Mats. Sci. and Eng.; 848 Benedum Hall, Pittsburgh, PA 15261 USA

Two-phase TiAl based intermetallics with the lamellar microstructure exhibit attractive sets of properties. The anisotropic mechanical properties associated with the lamellar microstructure are very well documented in the literature and so-called "soft" and "hard" deformation modes can be distinguished. Lamellar interfaces between adjacent g-TiAl lamellae and also between adjacent lamellae of a2-Ti3Al and g-TiAl are thought to be obstacles for dislocation motion and so defect interactions with lamellar interfaces become the basis for models describing the strength of lamellar TiAl alloys. In the present study, the details of slip transmission across lamellar boundaries and the occurrence of stress-induced activation of slip from sources at, for example, interfaces have been assessed. Of particular interest is the activation of slip in the phase a2-Ti3Al. While it is possible to activate dislocations with Burgers vecots, b, given by b=<a> by a process of slip transmission, it is not as easy to activate c-component dislocations, which would be required if reasonable ductility were to be exhibited. The roles of lamellar interfaces and the activation of slip in a2-Ti3Al on the strength and ductility of two-phase TiAl have been assessed. This work has been supported by a grant from the National Science Foundation with Dr. Bruce MacDonald as program manager.

3:20 PM
THE EFFECT OF MISFIT ON THE CHARACTER OF STEPS ON GAMMA/ALPHA-2 INTERFACES IN THE LAMELLAR MICROSTRUCTURE: Ping Shang[1]; Tai-Tsui Cheng[1]; *Mark Aindow*[1]; [1]The University of Birmingham, School of Metall. and Mats. & IRC in Mats. for High Performance Applications, Elms Rd., Edgbaston, Birmingham, W. Midlands B15 2TT UK

The structure of gamma/alpha-2 interfaces plays a significant role in determining the mechanical properties in lamellar TiAl-based alloys. It is usually accepted that there are two types of defects present in these interfaces; perfect dislocations with Burgers vectors 1/2<110> which accommodate the lattice mismatch and partial dislocations with Burgers vectors 1/6<112> which are associated with interfacial steps two atomic layers high. In our work we have studied lamellar gamma/alpha-2 interfaces in a series of TiAl-based alloys by analysing HREM images using Pond's topological theory of interfacial defects. It was found that the range of step heights and Burgers vectors is more diverse than has been reported previously and that these are not consistent with a partial dislocation description for the ledges. In this paper we will review these observations highlighting the effects of misfit on the defect character and discussing the consequences for the phase transformation and slip transmission mechanisms.

3:40 PM
DEFORMATION OF POLYSYNTHETICALLY TWINNED TiAl SINGLE CRYSTALS WITH "NEAR-HARD" ORIENTATIONS: Min-Chul Kim[1]; Lei Lu[1]; M. Nomura[1]; Vasek Vitek[1]; *David P. Pope*[1]; [1]University of Pennsylvania, Dept. of Mats. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104-6272 USA

The deformation modes observed in polysynthetically twinned TiAl single crystals are dramatically affected by the lamellar boundaries. Crystals having the "easy" orientation deform by slip on {111} planes

parallel to the boundaries. Deformation of crystals with "hard" orientations takes place in two different ways, depending upon the orientation of the applied uniaxial stress. When the stress axis is parallel to the boundaries, the lamellae deform such that the net slip vector is parallel to the boundary plane, i.e., slip does not cross the boundaries. In contrast, if the stress axis is perpendicular to the boundary plane, the slip is forced to cross the lamellar boundaries. In the latter case the flow stress is substantially higher and thus it represents the most difficult step in deformation of the polysynthetically twinned TiAl. We have performed compression tests on samples having near-hard orientations to determine the circumstances under which slip crosses the boundary planes, both in terms of the orientation of the crystals and the details of the slip process itself. The results provide a measure of the amount of strengthening provided by the boundaries when dislocations are forced to cross them. In parallel, we have carried out atomistic simulations of the interaction of ordinary dislocations impinging on the boundaries. The goal is to reveal the atomic level mechanisms of the slip transmission and/or slip reflection leading to the overall slip direction parallel to the interface. This research was supported by the NSF Grant No. DMR96-26344.

4:00 PM

PROPERTIES OF ORDINARY DISLOCATIONS IN Al-RICH GAMMA-TiAl SINGLE CRYSTALS DEFORMED IN SINGLE SLIP: *Patrick Veyssiere*[1]; Fabienne Gregori[1]; [1]LEM, CNRS-ONERA, BP 72, Chatillon Cedex, Hauts de Seine 92322 France

Several statistical measurements of temperature dependence of the density of pinning points on ordinary dislocations have served to model the flow stress anomaly of Al-rich gamma-TiAl. The underlying mechanism is the propensity of these dislocations towards self-locking by thermally activated cross-slip. We report on a TEM investigation of the organization of these, carried out on single crystals oriented for single slip to minimize ambiguities resulting from forest interaction. The cusping obeys rules that appear to correlate with the chronology of formation of dislocation bundles, which are profuse in foils cut parallel to the primary {111} slip plane. The distribution of pinning points is studied and the density of these measured. These measurements are compared to data available in the literature and discussed in terms of the flow stress anomaly. Factors other than intrinsic to the dislocation core, in particular the possible influence of a short-range Ti3Al5 ordered phase, are discussed.

4:20 PM INVITED PAPER

PROGRESS IN THE UNDERSTANDING OF PHASE TRANSFORMATIONS IN GAMMA TITANIUM ALUMINIDES: *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Mats. Sci. and Eng., Cincinnati, OH 45221-0012 USA

It is well known that the mechanical properties of gamma-TiAl alloys depend strongly on microstructure, which, in turn, is governed principally by the phase transformations the alloys experience. In the past decade, considerable advances have been made in the understanding of these phase transformations and associated phase equilibria, and in the definition of pathways through control of chemistry, alloying additions and thermal/thermomechanical parameters to obtain microstructures with desirable properties. In the first part of this talk, progress made in the understanding of the effects of cooling rate on the decomposition modes of the high temperature alpha phase will be presented. Emphasis will be given to the development of non-equilibrium transformation modes, particularly, the occurrence of the massive transformation, and the associated kinetics, thermodynamics and nucleation and growth mechanisms of this transformation will be discussed. In the second part of this talk, the effects of grain-refining alloying additions in the form of beta-phase stablizers and boron on phase transformations and microstructure evolution will be presented. When these additions are present, the lamellar transformation kinetics are found to be significantly altered and other competing transformation modes involving the high-temperature beta phase may begin to dominate. The sequence of decomposition modes, the associated kinetics and mechanisms will be discussed. Attainment of fine-grained fully-lamellar microstructures in the multicomponent alloys hinges on careful control of the levels of the various elements, heat treatment temperature and cooling rate. Finally, areas requiring further research will be highlighted.

4:50 PM

INFLUENCE OF Si ADDITION ON THE PHASE EQUILIBRIA OF Ti(40-51)at.%Al2at.%Cr ALLOYS: Choong Yeol Lee[1]; *Joong Keun Park*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. and Eng., 373-1 Kusong-Dong Yusong-Gu, Taejon 305-701 S.Korea

The influence of small amount of Si addition on the phase equilibria of Ti (40-51) Al2Cr (all in at.%) alloys at temperature lower than 1330ϒC has been studied using optical microscope, scanning electron microscope in back scattered electron image mode, X-ray diffractometer, and differential scanning calorimetry. Ti_5Si_3 phase was present at all temperatures and compositions with more than 0.5at.%Si investigated. The addition of Si has an effect to stabilize the β phase in the presence of Cr. Thus the phase sequence of Ti45Al2Cr alloy during cooling from 1330ϒC alters from α->α+γ to α->α+γ->α+β+γ -> β+γ as 1at.%Si is added. In addition, Si addition raises Tα temperature and extends the γ-phase field toward lower Al composition, thereby enlarging the γ-phase field in both the composition and temperature scale. This is consistent with the observation that Si atom substitutes Al atom in γ-phase. This substitution leads to an increase of c/a ratio from 1.0116 to 1.0133 despite the fact that the atomic size of Si is smaller than that of Al. A pseudo-binary phase diagram of Ti (40-51) Al2Cr1Si alloys has been experimentally determined at temperatures lower than 1330ϒC from the present investigation.

5:10 PM

NANOMETER LAMELLAR MICROSTRUCTURE AND TRANSFORMATION MECHANISM IN A GAMMA TiAl ALLOY: *Yong-Qian Sun*[1]; [1]University of Illinois, Dept. of Mats. Sci. and Eng., 1304 West Green St., Urbana, IL 61801 USA

Fully-lamellar microstructure on the nanometer scale can be produced by a suitable combination of heat-treatment routines. This paper reports the formation mechanism of the nanometer fully-lamellar microstructure and its effect on the mechanical properties. Aging treatment produced an ultra fine nanometer-scale fully lamellar microstructure, ranging from 8 nm in thickness at 600ϒC to 17 nm at 700ϒC. Optical microscopy observations show surface relief in prepolished samples, indicating the displacive nature of the phase transformation that leads to the fully-lamellar structure. The formation of lamellae is shown to be a nucleation-controlled process, characterized by fast growth of the individual lamellae. The lamellae nucleate preferentially at grain boundaries and grow into the grain interior. The fine lamellar microstructure produced a large strengthening effect, consistent with the Hall-Petch type relation between yield strength and lamellar thickness.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Poster Session I – 6:00 to 10:00 PM

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Synthesis & Processing, Structural Materials Committee, Titanium Committee
Program Organizers: Young-Won Kim, UES, Inc., Mats. & Proc. Div., Dayton, OH 45432-1805 USA; Dennis M. Dimiduk, Wright-Patterson AFB, WL/MD, WPAFB, OH 45433 USA; Michael H. Loretto, University of Birmingham, IRC, Birmingham B15 2TT UK

Monday PM Room: San Diego Ballroom A & B
March 1, 1999 Location: Marriott, North Tower

Session Chairs: Dongliang Lin, Shangahi Jiao Tong University, Inst. of Mats. Sci. & Eng., Shanghai 200030 China; Thomas R. Bieler, Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA; Alan Partridge, DERA Farnborough, Struc.Mats. Center, Farnborough, Hants GU14 0LX UK

MECHANICAL PROPERTIES OF HEAT-TREATED GAMMA-TiAl SHEET MATERIAL: *Anita Chatterjee*[1]; Hartmut Baur[2]; Rainer Joos[2]; Helmut Clemens[3]; Heinrich Kestler[4]; Arno Bartels[5]; Fritz Appel[6]; [1]Max-Planck-Institut fuer Metallforschung, Seestrasse 92, Stuttgart D-70174 Germany; [2]Daimler Benz AG, Forschung und Technologie, Ulm D-89013 Germany; [3]Universitaet Stuttgart, Institut fuer Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany; [4]Plansee AG, Technology Center, Reutte A-6600 Austria; [5]Technical University Hamburg-Harburg, Physics and Technology of Materials, Hamburg D-21071 Germany; [6]GKSS Research Center, Institute for Materials Research, Max-Planck-Strasse, Geesthacht D-21502 Germany

The mechanical properties involving tensile strength, fatigue and creep of Ti-46.5 at% Al- 4 at% (Cr,Nb,Ta,B) sheet material have been investigated at room temperature and elevated temperatures. The sheets were hot-rolled from HIPed prealloyed powder compacts. After rolling within the (alpha + gamma) phase field the sheets exhibit a fine-grained near gamma microstructure with a modified cube texture, i.e. the c-axes of the tetragonal unit cells are preferentially aligned in the sheet plane transversely to the rolling direction, which leads to a temperature dependent anisotropy of the mechanical properties. Prior to testing the sheets were subjected to four different heat treatments in order to establish different microstructures, containing fine-grained near gamma, coarse-grained fully lamellar and pseudo-lamellar microstructures with varying lamellar spacings as well as to alter the sheet texture. In order to characterize the lamellar constituents of the materials in detail TEM investigations were conducted.

THE CREEP AND TENSILE BEHAVIOUR OF A CARBON CONTAINING EXTRUDED TiAl ALLOY: *Alan Partridge*[1]; Jonathan J. Colvin[1]; Johnathan D.G. Paul[1]; [1]DERA, Structural Materials Centre, Ively Rd., Farnborough, Hants GU14 0LX UK

A number of recent studies have shown that high temperature extrusion can lead to significant improvements in the mechanical properties of g-TiAl alloys. As part of a larger programme studying the effects of wrought processing on the microstructure and properties of g-TiAl alloys, a carbon containing alloy has been plasma melted and extruded at 1200ϒC to produce an alloy with a highly refined grain structure. An assessment of the mechanical properties of this alloy show a good balance of properties, combining tensile strengths in excess of 700MPa with ductilities of the order of 3%. The origins of these improved properties will be discussed. The effect of intermediate temperature precipitation heat treatments on the tensile and creep strength of the extruded material has been evaluated and will also be discussed.

COMPARATIVE ANALYSIS OF THE DEFORMATION BEHAVIOR AND THE DISLOCATION STRUCTURE OF CuAu AND TiAl ALLOYS: *Bella Aleksandrovna Greenberg*[1]; Olga Vladimirovna Antonova[1]; Alex Yurievich Volkov[1]; [1]Institute of Metal Physics, Ural Branch of Russian Academy of Sciences, 18 S.Kovalevskaya St., Ekaterinburg GSP-170, Sverdlovsk Region 620219 Russia

Polycrystals of the ordered CuAu alloy having grains (5-10 microns) without lamellar structure were produced by a special thermomechanical treatment (see Intermetallics v.5, 1997, p.297). Plastic deformation and the microstructure of these polycrystals were studied over the temperature interval from -196 to 300° C. The yield stress and the plasticity were found to vary nonmonotonically with temperature. The yield stress was a minimum (~115 MPa) at room temperature and increased (~200 MPa) on approaching the limits of the temperature interval studied. On the contrary, the plasticity was a maximum (~18%) at room temperature and dropped at other temperatures, the drop being especially severe (down to 3%) at T > 250ϒC. It is remarkable that if the sample, which underwent such high-temperature deformation, was subsequently tested at room temperature, it recovered the initial plasticity. The TEM examination revealed the presence of superdislocations, ordinary dislocations and microtwins. The variations in the microstructure of the CuAu alloy deformed at various temperatures were compared with our earlier results on the evolution of the TiAl microstructure. Different models, such as disordering of the alloy at elevated temperatures, possible alteration of the state of grain boundaries, blocking of superdislocations and alteration of the deformation modes, have been discussed to account for specific features of the deformation behavior of CuAu.

MICROSTRUCTURAL CHANGES DUE TO PRIMARY CREEP OF A (-BASED TiAl ALLOY: *Birger Karlsson*[1]; Maria Kuntson-Wedel[1]; Dennis Lundstrom[1]; [1]Chalmers University of Technology, Dept. of Eng. Metals, Goteborg SE-412 96 Sweden

High and medium temperature alloys used in e.g. gas turbines suffer from severe creep during service. To control and increase the creep resistance, such as lowered primary creep strain and steady state creep rate, increased understanding of the deformation mechanisms is necessary. To study these mechanisms, TEM-studies of dislocations and twins have been performed. The investigated (-TiAl based alloy Ti-48Al-2W-0.5Si (at%) was produced by investment casting, followed by hot isostatic pressing and two successive heat treatments. The microstructure and defect structures were investigated both before and after creep testing using a Zeiss 912 equipped with an OMEGA energy filter. The creep tests were performed in air at constant load, and the applied stresses were 225, 275 and 325 MPa at 750ϒC. The materials studied were taken from specimens interrupted at 1% creep strain by decreasing temperature without unloading in order to retain the actual dislocation structure during the creep.

CHARACTERISATION OF THERMALLY EXPOSED TiAlCrNb ALLOYS: *Dawei Hu*[1]; A B Godfrey[1]; M H Loretto[1]; [1]University of Birmingham, IRC, Edgbaston, Birmingham, West Midlands B15 2TT UK

TiAl-based alloys with 47~49at%Al, 2at%Cr, 2at%Nb and 1at%B have been exposed to 700C for 1000h or 800C up to 3000h. C14 type Ti(AlCr)2 type Laves phase was observed in the alloys after thermal exposure. Alpha2 phase was found to be thermally unstable in the alloys with high Al concentration and decomposes during exposure via the reaction of alpha2-alpha2+gamma. Possible effects of Al concentration on alpha2 decompositon and Laves precipitation are disscussed.

THE EFFECT OF BORON ADDITION ON BRITLE-TO-DUCTILE TRANSITION TEMPERATURE AND ITS STRAIN RATE SENSITIVITY IN GAMMA TITANIUM ALUMINIDE: *Dongliang Lin*[1]; Wang Yu[1]; [1]Shanghai Jiao Tong University, Institute of Mats. Sci. & Eng., 1954 Huashan Rd., Shanghai 200030 PR China

Tensile properties and fracture mode of two gamma titanium aluminides, Ti-47Al-2Mn-2Nb and Ti-47Al-2Mn-2Nb-0.8TiB2, were investigated in a temperature range from 77 to 1373K and strain rate range from 10-5 to 10-1s-1. Brittle-to-ductile transition (BDT), which was accompanied by a transition in fracture mode, was manifested in the investigated alloys. Brittle-to-ductile transition temperatures (BDTT)

of both alloys were determined under different strain rates and, based on the strain rate dependence of the determined BDTTs, apparent BDT activation energies were determined using Zener-Hollomon factor. It was found that the BDTT of either alloy increases sharply with the strain rate and that the minor addition of 0.8TiB2 reduced BDTT by about 100K under the same strain rate. The TiB2 addition also decreases apparent BDT activation energy from 324 to 256 kJ/mol. But BDT activation energies of the two alloys approximate to self- and inter-diffusion of Ti and Al atoms in TiAl phase. The approximation, fractography analysis and theoretical calculation using the Nabarro Model add up to the speculation that BDT of the investigated alloys is controlled by dislocation climbing.

ORIGIN OF DISPLACIVE PHASE TRANSFORMATIONS IN TiAl-Nb ALLOYS: A FIRST-PRINCIPLES STUDY: *Duc Nguyen-Manh*[1]; *David G. Pettifor*[1]; [1]Oxford University, Dept. of Mats., Parks Rd., Oxford, Oxfordshire OX1 3PH UK

New ductile intermetallic alloys with desirable mechanichal properties have recently been developed within the β/B2 (+orthohombic) TiAl-Nb ternary system. Unfortunatery, however, occasional omega phase formation may drastically embrittle the parent B2 phase during cooling. We present a first-principle study of the stability conditions under which these metastable orthorhombic and omega phases are likely to occur. In general, the orthorhombic and omega displacive phase transformations involve not only homogeneous strains but also shuffles and chemical ordering. Our studies show a strong correlation between these phase transformations and mechanical instabilities in the related B2 alloys at low temperature. Interestingly, we also find that the B2 high temperature phase of TiAl-Nb alloys may transform to low temperature orthorhombic phase by homogeneous twinning deformation. The structural relationships between these phases are analysed, the theoretical results strongly supporting the observed microstructural evolution in the advanced intermetallic TiAl-Nb alloys.

THE STRESS DEPENDENCE OF SUBSTRUCTURE DURING CREEP OF GAMMA-BASED TITANIUM ALUMINIDES: *Eric A. Ott*[1]; Tresa M. Pollock[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The creep deformation of gamma-based titanium aluminide alloys has been shown to involve a number of mechanisms including ordinary and super dislocation activity, deformation twinning, recrystallization, and grain boundary sliding. Minimum creep rates are strongly dependent upon applied stress at high stress levels and moderately dependent at lower stresses. Overall trends in the stress dependence of minimum creep rate for the majority of typical microstructures suggests that similar deformation mechanisms may occur in equiaxed, duplex, and fully lamellar structures. The results of creep tests at 760ϒC for stresses between 50 and 400 MPa will be presented. Deformation-induced substructures were studied in detail for equiaxed and duplex microstructures by transmission electron microscopy for low strain tests performed at high and at low stresses. These substructures will be compared to those obtained by high temperature tensile testing at higher strain rates.

THE MECHANICAL PROPERTIES OF NIOBIUM ALLOYED GAMMA-TITANIUM ALUMINIDES: *Fritz Appel*[1]; Jonathan H. Paul[2]; Michael Oehring[1]; [1]GKSS, Institute for Materials Research, Max-Planck-Str., Geesthacht D-21502 Germany; [2]DERA, Structural Materials Centre, Griffith Building (A7), Hampshire, England GU14 0LX UK

The underlying mechanisms behind the high strength of titanium aluminide alloys containing a large addition of niobium have been investigated by mechanical testing and detailed TEM investigations. It has been found that alloys such as Ti-45Al-(5-10)Nb(at.%) have flow stresses in excess of 900 MPa at room temperature and higher than 500 MPa at 900ϒC. At the same time moderate tensile elongations of about 1% plastic strain can be achieved at room temperature. The TEM observations indicate that these appreciable strength properties can mainly be ascribed to a microstructural refinement which occurs as a consequence of the site occupation of Nb in gamma (TiAl). Hot working routes for the processing of these alloys will be discussed with respect to engineering applications.

SOLID SOLUTION STRENGTHENING MECHANISM OF TiAl ALLOY: *Fu-Sheng Sun*[1]; C. X. Cao[2]; M. G. Yan[2]; Y. T. Lee[3]; S. E. Kim[3]; [1]Beijing Institute of Aeronautical Materials, Lab. of Titanium Alloys, P.O. Box 81-15, Beijing 100095 China; [2]Beijing Institute of Aeronautical Materials, Lab.15, P.O. Box 81, Beijing, 100095 China; [3]High Temperature Materials Lab, Korea Institute of Machinery & Materials, 66, Sangnam-Dong, Changwon, Kyungnam Korea

The effect of beta stabilizers, such as Fe, Cr, V, and Nb on the microstructures and phase constituents of Ti52Al48-xM (x=0, 1.0, 2.0, 4.0 6.0at%) alloys was studied. The dependence of tensile properties and creep rupture property of TiAl alloys on the alloying elements was investigated. Fe is the strongest B2 phase stabilizer, Cr is the second one, V is an intermediate B2 phase stabilizer, while Nb is the weakest one. The composition partitioning of Fe, Cr, V, and Nb in (phase is affected by the formation of B2 phase. The peaks of the tensile strengths at 25 and 900¡æ, and creep rupture life of Ti52Al48-xM alloys generally occur at the maximum solid solution of these elements in (phase, which is just before the formation of B2 phase. The improvement of tensile strengths and creep resistance with the increase of Fe, Cr, V, and Nb content is chiefly attributed to the solid solution strengthening of these elements in (phase. The appearance of B2 phase deteriorates the creep resistances, tensile strengths and ductilities. With respect to the maximum solid solution strengthening, an empirical equation of Cr equivalent [Cr] is suggested as following: [Cr]= Cr+Mn+3/5V+3/8Nb+3/2(W+Mo) +3Fe=1.5-3.0.

A PRELIMINARY STUDY OF HOT CORROSION BY NaCl ON Ti-48Al-2Cr-2Nb: *Gregory T. Dowling*[1]; Thomas R. Bieler[1]; [1]Michigan State Univerisity, Mats. Sci. and Mech., 3536 Eng. Bldg., E. Lansing, MI 48824-1226 USA

A TiAl alloy (nominal composition in at%, Ti-48Al-2Cr-2Nb) was subjected to hot corrosion by application of small amounts of simulated sea salt by aqueous means. Bare ground specimens of the alloy were sprayed with sea water solution, dried, and exposed in an air furnace at 760°C for times between 0.1 and 20 ks. Obvious attack occurred in as little as 0.3 ks in regions where clumped salt crystals resulted from dried salt water spots. In such regions, oxide growth was accelerated and non-adhesive. Regions below accelerated growth were pitted heterogeneously in a manner that indicated that the alpha-2 phase was preferentially attacked, particularly in lamellar regions. Despite the visible damage, mechanical strength as measured by 4-point bending experiments always exceeded the yield strength. A consistent reduction of fracture strength was observed only for specimens with the longest exposure. In an effort to examine the potential of salt to damage TiAl in conditions relevant to heat engines, pre-oxidiz ed specimens have been exposed in similar ways using more realistic salt application methods, and no apparent accelerated oxide growth was observed after times up to about 1 ks, though Na and Cl can be identified with EDS on the oxide surface in low concentrations. DSC and DTA analysis of powdered salt-TiAl mixtures in Ar or N2 indicate that exothermic reactions occur at temperatures near 700 and 750°C. These results are discussed with the goal of identifying critical conditions where salt attack in a relevant manner to heat engines may be quantified in future work.

DEFORMATION AND MICROSTRUCTURE OF Ti-47Al-2Cr-0.2Si ROLLED SHEETS: *Gopal Das*[1]; [1]Pratt & Whitney, P.O. Box 109600, West Palm Beach, FL 33410-9600 USA

The tensile behavior of a near-gamma titanium aluminide alloy sheet (Ti-47Al-2Cr-0.2Si) was determined in the primary annealed condition with an initial grain size of 12-14 microns at RT-800ϒC. The strain rate sensitivity parameter, m, was evaluated by conducting tensile tests at various strain rates ranging from 10-3 to 10-5 sec-1 at 1000 -1100ϒC. Gas forming of rolled sheets was performed at elevated temperature in order to assess the potential of superplastic forming of rolled sheets. Additionally, hot forming of rolled gamma sheets was successfully accomplished. Microstructural changes such as recrystallization and cavitation, and failure modes including texture behavior before and after forming and residual strength following gas forming will be presented.

MECHANISMS OF FATIGUE CRACK PROPAGATION IN GAMMA TITANIUM ALUMINIDES: *Gilbert Henaff*[1]; Catherine

Mabru[1]; [1]ENSMA, LMPM, Teleport2, Futuroscope Cedex, Vienne 86960 France

In view of the introduction of gamma based alloys into structural components the damage tolerance of these materials must be assessed. However, as for conventional engineering alloys, their fatigue crack propagation resistance result from a complex balance between processes of different nature which is not fully understood. The present study is tackling the issue of identifying the micromechanisms involved in the crack growth process and quantifying their respective influence by conducting series of tests on a quaternary alloy. A special attention is paid to the role crack tip shielding by closure as to the environmental enhancement with respect to temperature. On the basis of measurements and in-situ observations the main source of closure appears to be due to premature recontact between asperities on cracked surfaces, even at elevated temperatures. Besides the severe environmental enhancement observed in ambient air does not seem to be temperature dependent. Potential involved mechanisms are discussed with the support of microfractographic observations.

FORMATION OF FINE-GRAINED LAMELLAR STRUCTURE IN GAMMA TITANIUM ALUMINIDES BY MEANS OF RAPID HEATING: *Gennady Salishchev*[1]; Renat Imayev[1]; Andrey Kuznetsov[1]; Marat Shagiev[1]; Oleg Senkov[2]; Francis Froes[2]; [1]Institute for Metals Superplasticity Problems, Khalturina Str. 39, Ufa 450001 Russia; [2]University of Idaho, Institute for Materials and Advanced Processes, Mines Building, Room 321, Moscow, ID 83844-3026 USA

Poor ductility of gamma titanium aluminides with a lamellar structure at room temperature (RT) impedes their application. In the present study the methods of rapid heating of preliminary refined materials were employed in order to improve the RT ductility. Specimens of Ti-48Al-2Cr-2Nb and Ti-46Al alloys with initial microcrystalline structure were heated by electro-pulse and induction methods up to temperatures higher than alpha-transus with a subsequent cooling in the air. As a result, the lamellar structure with a colony size less than 30-40 mkm was obtained in both the alloys considered. Mechanical tests has confirmed already known fact that decrease in the colony size leads to considerable increasing of both the RT ductility and strength of gamma titanium aluminides. Thus the rapid heatingof these materials was found to be effective for forming the fine-grained lamellar structure.

INTERPLAY OF THERMODYNAMICS AND TRANSFORMATION KINETICS ON MICROSTRUCTURAL DEVELOPMENT IN TiAl-BASED ALLOYS: *Guosheng Shao*[1]; Panos Tsakiropoulos[1]; [1]University of Surrey, School of Mech. and Mats. Eng., Guildford, Surrey Gu2 5XH UK

Understanding the phase transformations in Ti-Al-X (X: b stabilising element/s) is important for the development of titanium aluminide based alloys. The present work shows firstly how a thermodynamic database can be used in conventional processing of TiAl-based alloys and secondly, how the database can be combined with nucleation and growth for the interpretation and prediction of phase/microstructure selections under non-equilibria conditions. Effects of high cooling rates on phase selection will be discussed in the Ti-50Al alloy. Phase equilibria during solid state cooling will be discussed using the competition between a®g massive transformation and a®a2 ordering. It will be shown that under metastable conditions, prediction and modelling of structural evolution must include thermodynamics, nucleation and growth, as well as the effect of crystallographic features of the competing phases

EFFECT OF THE LAMELLAR ORIENTATION ON CREEP STRENGTH IN POLYSYNTHETICALLY TWINNED (PST) Ti-48mol%Al: *Gerhard Wegmann*[1]; Ryuichi Yamamoto[2]; *Kouichi Maruyama*[1]; *Haruyuki Inui*[3]; *Masaharu Yamaguchi*[3]; [1]Tohoku University, Graduate School of Eng., Dept. of Mats. Sci., Aobayama 02, Sendai, Miyagi 980-8579 Japan; [2]Mitsubishi Heavy Industries, Ltd., Takasago Research & Development Center, Materials and Strength Laboratory, 2-1-1 Shinhama, Arai-cho, Takasago, Hyougo 676-8686 Japan; [3]Kyoto University, Department od Materials Science and Engineering, Sakyo-ku, Kyoto 606-01 Japan

Polysynthetically twinned (PST) crystals provide a fundamental unit of microstructure of the lamellar colonies present in polycrystalline (α_2+γ) TiAl. To investigate the influence of the orientation of the lamellar plates on the creep strength, three orientations of a PST crystal of the nominal composition Ti-48mol%Al were deformed in compression for different stresses and temperatures. For comparison the creep characteristics of a polycrystalline Ti-47mol%Al alloy was evaluated. The PST specimens with their lamellae 90Ỳ to the stress axis show the lowest minimum creep rates at all stresses and temperatures, followed by the specimens with the lamellae under 0Ỳ to the stress axis. The soft orientation with the lamellae under 45Ỳ to the stress axis gives much higher minimum creep rates that are even higher than in the polycrystalline alloy. This behavior could be attributed to differences in the deformation mode, what has been confirmed by TEM observations and texture measurements.

OXIDATION PROTECTIVE COATINGS FOR GAMMA-TiAl BASED ALLOYS: Hans-Peter Martinz[2]; *Helmut Clemens*[1]; Wolfram Knabl[2]; [1]Universität Stuttgart, Institut für Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany; [2]Plansee AG, Technology Center, Planseestrasse, Reutte, Tyrol A-6600 Austria

Oxidation resistance might become a critical factor for g-TiAl based alloys to be used for long durations at temperatures >700ỲC, especially under cyclic thermal conditions and mechanical load. Three different oxidation protective coatings were investigated: (1) an Al/Cr coating deposited by pack cementation, (2) a NiAl coating prepared by Ni-electroplating and subsequent Al pack cementation and (3) a CoNiCrAlY coating deposited by atmospheric plasma spraying. Ti-47at%Al-2at%Cr-0.2at%Si sheet was selected as base material because this alloy exhibits a rather low oxidation resistance and shows strong spallation effects when submitted to cyclic oxidation. All selected coatings improve the oxidation resistance significantly. The interaction between the different coatings and the base material has been investigated by 4-point-bending tests at room temperature. The coated samples show a significant decrease of bending strength and bending angle in most cases already after the coating process. This behaviour is caued by the formation of brittle intermetallic phases and/or interdiffusion zones which act as initiation sites for cracks upon loading.

CHARACTERIZATION OF GAMMA TITANIUM ALUMINIDE SHEET MATERIAL FOR AEROENGINE APPLICATION: Helmut Clemens[2]; Hartmut Baur[3]; Rainer Joos[3]; Rainer Gerling[4]; Gürel Cam[4]; Arno Bartels[5]; *Heinrich Kestler*[1]; Wilfried Smarsly[6]; [1]Plansee AG, Technology Center, Planseestrasse, Reutte, Tyrol A-6600 Austria; [2]Universität Stuttgart, Institut für Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany; [3]Daimler Benz AG, Forschung und Technologie, Wilhelm-Runge-Str.11, Ulm D-89013 Germany; [4]GKSS Research Center, Institut for Material Research, Max-Planck-Strasse, Geesthacht D-21502 Germany; [5]Technical University Hamburg-Harburg, Phys. and Tech.of Mats., Eißendorferstr. 42, Hamburg-Harburg D-21071 Germany; [6]MTU Motoren-und Turbinen-Union, Advanced Mats., Dachauer Str. 665, München D-80995 Germany

In 1995, a German materials technology program has been started aiming to demonstrate the feasibility of hollow turbine blades out of gamma-TiAl sheet material. The selected alloy ("Gamma-TAB") was developed by GKSS. Sheets up to 1000 x 280 x 1.2 mm were rolled from HIPed prealloyed powder compacts. Heat-treatments studies were conducted to optimize the mechanical properties of the sheets with regard to the expected demands. This paper summarizes the results of an extensive characterization program, which was carried out to establish the technological background for turbine blades processing as well as to provide data for design purposes and life-time predictions. The following topics will be discussed: superplastic behavior of gamma-TiAl sheets, diffusion bonding, tensile and creep properties of different microstructures as well as LCF and HCF behavior including first results on the effect of foreign object damage on fatigue behavior.

MICROSTRUCTURAL EVALUATION OF CREEP IN XD-47Al ALLOY: *Isabelle Haurie*[1]; Ian Perrin[2]; Leo Christodoulou[1]; Alan Partridge[3]; [1]Imperial College of Science, Technology and Medicine, Dept. of Mats., Prince Consort Rd., London SW7 2BP UK; [2]GEC ALSTHOM, Mechanical Engineering Centre, Cambridge Road, Whetstone, Leicester LE8 6LH UK; [3]DERA, Structural Materials Centre, Farnborough GU1 4OLX UK

High temperature applications of near-gamma TiAl alloys require study of the possible microstructural changes occurring during creep deformation. To understand such effects a number of crept samples has been investigated using SEM and TEM to study parameters such as void coalescence, recrystallization, dislocation multiplication and twinning. A particular interest was the study of several samples which were discontinued at four different strain levels in order to elucidate the damage mechanisms that occur during the various stages of creep. A goal of this work is to construct a deformation-damage map for this alloy for the regimes in which it is likely to be used in power-plants.

EFFECT OF LAMELLAR ORIENTATION ON CONSTANT-STRAIN-RATE DEFORMATION IN TiAl AT HIGH TEMPERATURE: *Jorg Michael Wiezorek*[1]; Subramanian Karthikeyan[2]; *Hamish L Fraser*[2]; *Michael J Mills*[2]; [1]Ohio State University - Now @ University of Pittsburgh, Dept. of Mats. Sci. and Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Ohio State University, Materials Science and Engineering, 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

The anisotropic mechanical properties associated with lamellar TiAl based intermetallics are very well documented in the literature and so-called “soft” and “hard” deformation modes can be distinguished. Lamellar interfaces between neighboring g-TiAl lamellae and a2-Ti3Al and g-TiAl are efficient obstacles for dislocation motion during “hard” mode deformation and have been suggested to influence significantly the strength of lamellar TiAl alloys. Interestingly, the elevated temperature creep resistance of lamellar TiAl appears to be related to the “soft” mode behavior, which has generally been considered as unaffected by the presence of the lamellar interfaces, yet refinement of the lamellar spacings improves the creep strength. The present systematic study investigates the role of interface related deformation processes active during elevated temperature loading on the level of strength retention and plasticity of lamellar TiAl. Poly-synthetically twinned (pst-TiAl) coupons of Ti-48at.%Al have been compressed at various constant strain rates and to different levels of plastic strain at 750Ɣ, the envisaged maximum-use-temperature of TiAl. Appropriately oriented pst-TiAl coupons have been used to differentiate between the unique “hard”- and “soft”-loading geometries. The deformation modes active in the various g- and a2-lamellae under these different sets of loading conditions are identified by detailed TEM and SEM characterization, with an emphasis on interface related processes. The results of these experiments are discussed in relation to the high temperature deformation behavior of lamellar TiAl alloys. Implications for the development of a better understanding of the fundamental mechanisms of elevated temperature creep in these microstructures are outlined. Financial support by the National Science Foundation with Dr. Bruce MacDonald as program manager is acknowledged.

MICROSTRUCTURE EVOLUTION AND HOT-WORKABILITY OF THE TiAl ALLOYS CONTAINING MINOR Ni AND Mg: PROS AND CONS: *Ji Zhang*[1]; [1]Central Iron and Steel Reserach Institute, Dept. of Superalloys, Beijing 100081 China

Hot-workability of TiAl alloys is mainly determined by the microstructure evolutions before and during the processing. Since the static recrystallization of the cast lamellar microstructure is not yet successful, the studies on the hot-workability and microstructure evolutions in the primary thermomechanical processing of TiAl alloys are rather limited. In this talk, the addition of minor Ni will be employed to promote the cast lamellar microstructure to evolve into an equiaxed grain near gamma microstructure before the hot-working. Then, the hot-workability of the transformed microstructure and the microstructure evolutions during the hot pressing will be evaluated in the temperature range of 950-1150Ɣ. The follow-up influences of the occurred microstructure evolutions on the following microstructure modification and secondary hot-workability of the TiAl alloy containing Ni will be prospected as well.

THE EFFECT OF HEAT TREATMENT ON THE MICROSTRUCTURE AND CREEP PROPERTIES OF XD TiAl INTERMETALLICS: *Linruo Zhao*[1]; [1]National Research Council of Canada, Institute for Aerospace Research, Montreal Rd., M-13, Ottawa, Ontario K1A 0R6 Canada

Systematic heat treatment studies have been performed on investment cast XD Ti-45Al-2Nb-2Mn+0.8 vol.%TiB2 and Ti-47Al-2Nb-2Mn+0.8 vol.%TiB2 intermetallics with a view to improving their creep resistance. In contrast to the commonly practiced post-cast heat treatment applied to the materials, a modified heat treatment was developed to produce a fully lamellar microstructure with straight gamma and alpha-2 plates, interlocked grain boundaries as well as fine lamellar grains. Preliminary creep tests at 760Ɣ/138 MPa have shown that the modified fully lamellar structure in the XD TiAl intermetallics yields considerably higher creep resistance relative to the ‘standard’ lamellar microstructure. The effect of cooling rate on the lamellar spacing and subsequently on creep resistance of the XD materials has also been investigated.

CHARACTERIZATION OF AL-DEPLETION LAYER IN A TiAl BASED ALLOY: *Mahesh C. Chaturvedi*[1]; Uttara Prasad[1]; Qiang Xu[1]; A. K. Jena[1]; [1]University of Manitoba, Dept. of Mech. and Indust. Eng., 364 Eng. Bldg., Winnipeg, Manitoba R3T 2N2 Canada

This paper will present chemical and microstructural characterization of Al-depletion layers that formed in a Ti-45Al-2Nb-2Mn alloy after the alloy was exposed to high temperature for various periods of time. It was found by a detailed SEM and TEM observation that a significant loss of aluminium occurred at the subsurface resulting in formation of an Al-depletion layer. The microstructure in the Al-depletion layer was found to be remarkably different from the bulk with alpha 2, B2 and “omega-type” phases nearby the surface and the alpha 2 phase nearby the “intact” bulk. It was also observed that phases present in the Al-depletion layer remained the same irrespective of heat treating time after the alloy was heat treated for a certain period of time while thickness of the Al-depletion layer increased with the heat treating time. Based on the research conducted so far, mechanisms of Al-depletion will be proposed.

ATOM PROBE FIELD ION MICROSCOPY OF POLYSYNTHETICALLY TWINNED TITANIUM ALUMINIDE: *David J. Larson*[1]; Michael K. Miller[1]; H. Inui[2]; M. Yamaguchi[2]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Bldg. 5500, MS 6376, Oak Ridge, TN 37831-6376 USA; [2]Kyoto University, Dept. Materials Science and Engineering, Sakyo-ku, Kyoto 606-8501 Japan

Polysynthetically twinned (PST) TiAl crystals have been developed in order to study systematically the a2+g TiAl lamellar microstructure because interface stability is a key to providing usable high temperature materials. These PST materials contain no high angle grain boundaries and have a single set of aligned lamellae of a2 and g phases. Previous transmission electron microscopy studies of PST TiAl have shown that Cr and Mo segregation occurs at certain g/g twin boundaries. These studies also found a depletion of aluminum at certain g/g interfaces, showing “a2-like” compositions. The advantages of applying the technique of atom probe field ion microscopy to PST TiAl samples will be described. The results of atom probe characterizations of binary PST TiAl and ternary PST crystals doped with V and Zr will be presented. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. This research was conducted utilizing the Shared Research Equipment (SHARE) User Program facilities at Oak Ridge National Laboratory.

THE INFLUENCE OF Nb AND Zr ADDITIONS ON THE HIGH TEMPERATURE OXIDATION OF GAMMA-TiAl ALLOYS IN Ar/O2.: *Marija Yurechko*[1]; Vladimir Shemet[1]; [1]Kiev Polytechnic Institute, High Temperature Mats. and Powder Metall., Pr. Peremogy 37,1103, Kiev 252056 Ukraine

The effects of small single and combined additions of Zr and Nb on the high temperature oxidation of gamma-TiAl alloys at 900°C in Ar/O2 were examined by thermogravimetry, X-ray diffraction and scanning electron microscopy. The gamma-TiAl(Nb) and gamma-TiAl(Zr) alloys followed approximately parabolic oxidation. The Nb-containing alloys formed TiO2/Al2O3 scales doped with Nb while the Zr-containing alloys formed a thin Al2O3/Z-phase layer whereby the Z-phase (Ti5Al3O2) was doped with Zr. The Nb-containing alloys showed internal oxidation precipitates mainly consisting of alpha-Al2O3. In con-

trast, the Zr-containing alloys did not show any internal oxidation. Even after longer oxidation times, the sub-surface Al-depletion layer in these alloys consisted of a single Z-phase layer beneath the external Al2O3 base scale. The best long term oxidation resistance for temperatures up to 900°C was obtained for the gamma-TiAl alloys containing small additions of both Zr and Nb.

PHASE EQUILIBRIA IN MULTI-COMPONENT GAMMA TITANIUM ALUMINIDES: *Nigel J. Saunders*[1]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey GU2 5YG UK

A reasonable experimental literature has now been built up on Ti-Al-X ternary phase diagrams. However, although these basic systems give insight to phase equilibria in certain commonly used TiAl-based alloys it is difficult to interpret phase relationships in multi-component alloys using just this information. Thermodynamic calculations via the CALPHAD route (ref.1) offer a means by which phase equilibria in multi-component alloys can be predicted and the present paper presents typical results which can now be achieved using this methodology. As well as basic relationships between the TiAl, Ti3Al, beta and alpha phases, the ordering of the beta phase to B2 and the effect of minor impurities such as C, N and O will be analysed and discussed. References: 1. N.Saunders and A.P.Miodownik, "CALPHAD - A comprehensive guide" (Elsevier Science, New York, 1998)

THE EFFECT OF Ti/Al RATIO ON THE SITE PREFERENCE OF ALLOYING ELEMENTS IN GAMMA-TiAl: Yulin Hao[1]; Yuyou Cui[1]; *Rui Yang*[1]; Dong Li[1]; [1]Institute of Metal Research, Chinese Academy of Sciences, Titanium Alloy Laboratory, 72 Wenhua Rd., Shenyang, Liaoning 110015 China

The site occupancies of V, Cr, Mn, Fe, Ni, Zr, Nb, Ta, Mo, Ga and Sn (1~5at.%) in TiAl alloys with different nominal Ti/Al ratios were measured by the atom location channeling enhanced microanalysis (ALCHEMI) method. The results show that Zr, Nb and Ta invariably occupy Ti sublattice sites, while Fe, Ni, Ga and Sn occupy Al sublattice sites, the alloy composition having no significant influence on their site preference. By contrast, the site preference of V, Cr, and Mn changes significantly with alloy composition (the Ti/Al ratio in particular), the probability of these elements occupying Ti sites decreasing in the above order. In general, with increasing atomic number, elements in the same period show increasing tendency to substitute for Al, so is the tendency to substitute for Ti for elements in the same group down the periodic table. A discussion is made in terms of a Bragg-Williams type model and bond order data obtained by electronic structure calculations, allowing qualitative interpretation of the experimental findings.

THE EFFECT OF PROLONGED HIGH TEMPERATURE AIR EXPOSURE ON MONOTONIC AND CYCLIC PROPERTIES OF A GAMMA TITANIUM ALUMINIDE ALLOY: *Stetson K. Planck*[1]; Andrew H. Rosenberger[2]; [1]University of Dayton Research Institute, Structural Integrity, 300 College Park, Dayton, OH 45469-0128 USA; [2]Air Force Research Laboratory, Materials Behavior, 2230 Tenth Street, Ste. 1, WPAFB, OH 45459-0128 USA

Past efforts have examined the effects of high temperature exposure on the tensile ductility of gamma titanium aluminides. Currently, however, there is not a clear understanding of the effect of high temperature exposure on the fatigue performance of this class of alloys. Tensile and fatigue tests at room temperature, 540ϒC, and 760ϒC were conducted following 50 and 500 hour air exposures at 760ϒC to determine the degree of degradation of the monotonic and cyclic properties due to prolonged high temperature exposure of a gamma titanium aluminide. The embrittled layer that formed during exposure was characterized using hardness and scanning electron microscopy. The fracture of the embrittled layer leads to a reduction in the room temperature ductility and strength. The removal of the affected surface layer by low-stress grinding restores the unexposed tensile properties. The high temperature exposure results in a modest degradation of the fatigue resistance of this gamma alloy at room temperature and 760ϒC. However, at 540ϒC, there is a more severe reduction in fatigue performance due to the high temperature exposure. The brittle surface layer aids crack initiation which, when combined with the poor fatigue crack growth resistance, results in the largest overall reduction in fatigue performance. Again, removal of the affected surface layer restores the unexposed fatigue properties.

ATOMISTIC SIMULATIONS OF CROSS-SLIP PROCESSES IN GAMMA TiAl: *Satish I. Rao*[1]; P. Hazzledine[1]; C. Woodward[1]; D. Dimiduk[2]; [1]UES Inc., 4401, Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Wright-Patterson AFB, Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson AFB, OH 45433 USA

Gamma TiAl exhibits a yield stress anomaly in the high temperature regime which is attributed to the easy cross-slip ability of ordinary a/2<110] screw dislocations (Viguier et.al., Phil.Mag.A 1995 and Sriram et.al., Phil.Mag.A 1997). In this manuscript, empirical embedded atom method (EAM) potentials are used to simulate atomistically the structure and energetics of different cross-slip processes of ordinary a/2<110] screw dislocations in gamma TiAl. Results on the energetics of cross-slip from these simulations are consistent with previous first principles calculations and atomistic estimates of planar fault energies in Gamma TiAl as well as in agreement with microstructural observations of ordinary dislocations in Gamma TiAl in the yield anomaly regime.

THE MECHANISM OF GRAIN REFINEMENT IN TiAl-BASED ALLOYS BY BORON ADDITIONS: *T. -T. Cheng*[1]; [1]The University of Birmingham, IRC in Mats., Elms Rd., Edgbaston, Birmingham, West Midlands B15 2TT UK

It has become common practice to grain-refine TiAl-based alloys by adding boron. The general consensus is that the grain refining effect of boron is a switch on/switch off phenomenon in that a minimum amount of boron (=0.5%) is required to refine these alloys. Below this level there is little effect on the grain size whereas adding much higher levels does not further reduce the grain size. Irrespective of the grain refining effect, boride particles are always observed and they can have several different crystal structures and morphologies. There are three different mechanisms which have been proposed for the grain refinement in gamma alloys. In the first the added borides are considered as inoculants, i.e. they do not melt completely. In the second it is suggested that borides precipitate first during solidification and subsequently act as nucleants for the alloy grains. In the third it is suggested that boron weakens the base of dendrite arms and causes them to break off and act as nucleants. This paper will describe the results of a microstructural study on a series of gamma TiAl-based alloys to which different levels of boron have been added. It will be shown that none of the mechanisms proposed previously can account fully for the observed characteristics of the grain refinement. An alternative mechanism based on constitutional supercooling will be described which is consistent with both the data obtained from these alloys and that published previously.

DIRECTIONAL SOLIDIFICATION AND CREEP DEFORMATION OF TiAl-Si ALLOYS: *Takamitsu Yamanaka*[1]; David Ray Johnson[1]; Yosihiro Masuda[1]; Haruyuki Inui[1]; Masaharu Yamaguchi[1]; [1]Kyoto University, Dept. of Mat. Sci. & Eng., Yoshida Honmachi, Sakyo-ku, Kyoto 606-8501 Japan

Creep tests were conducted on directionally solidified TiAl-Si alloys to discern the effect of the lamellar orientation and the addition of silicon on the high temperature strength. Growth from a seed was used to align the lamellar microstructure for ingots containing up to 3 at.% Si. The as-processed material consisted of a PST matrix containing large eutectic silicide particles within the interdendritic spaces and small silicide precipitates along the alpha2/gamma lamellar boundaries. In addition, ingots with large columnar grains of various lamellar orientations were also produced by the floating zone technique and texture measurements by X-ray diffraction were used to identify the lamellar orientation. Tensile creep tests were conducted on both sets of ingots and the results are discussed in terms of the lamellar orientation and the silicide precipitates.

GAS-ATOMIZED GAMMA-TITANIUM ALUMINIDE BASED ALLOYS - PROCESSING, MICROSTRUCTURE AND MECHANICAL PROPERTIES: *Ulrike Habel*[1]; C. Frederick Yolton[1]; John H Moll[1]; [1]Crucible Materials Corporation, Crucible Research Center, 6003 Campbells Run Rd., Pittsburgh, PA 15205 USA

Ti gas atomization yields fine, almost segregation-free powders. It is increasingly used to produce Ti-intermetallics including a variety of TiAl based alloys. Scale-up and process improvements have significantly increased the yield of powder making. The powders are consolidated by hot isostatic pressing (HIP) into fully dense compacts of various shapes and sizes. This paper encompasses the whole process of powder making, powder characterization and consolidation as well as microstructure and properties of the final product. Powders of Ti-48Al-2Cr-2Nb with and without additions of 0.1at% B have been produced. The powders are generally spherical and show few satellites. The HIP-consolidated 48-2-2 and 48-2-2-B exhibit fine and homogeneous near-microstructures. They can be heat treated to achieve isotropic duplex or lamellar microstuructures. Tensile properties of as-HIP and HIP plus heat treated materials are evaluated.

AUTOGENOUS GAS TUNGSTEN ARC WELDING OF GAMMA TITANIUM ALUMINIDE AND THE EFFECTS OF POSTWELD HEAT TREATMENT: Mario Arenas[1]; *Sarah Agee*[1]; Viola L. Acoff[1]; [1]The University of Alabama, Metall. & Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

Alloys of gamma TiAl were butt-welded using traveling autogenous gas tungsten arc (GTA) welding without preheating. Microstructural examination revealed a nearly lamellar microstructure in the base metal which was transformed to a dendritic structure with evidence of interdendritic gamma phase in the fusion zone. This dendritic microstructure exhibited enrichment of titanium and depletion of Al in the dendrite cores relative to the dendrite interstices. The microstructural transformation upon welding seems to be related to compositional changes produced during non-equilibrium solidification through the peritectic reactions. Mechanical properties of the weld zone were discussed in terms of hardness measurements. For all heat inputs, microhardness profiles indicated an increase in hardness from the base metal to the fusion zone suggesting a reduction in mechanical properties such as ductility and toughness relative to the base metal. Postweld heat treatment was used to optimize mechanical properties of the fusion zone.

TENSILE AND CREEP PROPERTIES OF DIFFUSION BONDED TITANIUM ALLOY IMI 834 TO GAMMA TITANIUM ALUMINIDE IHI ALLOY 01A: *Viktor Recina*[1]; Magnus Holmquist[1]; Bengt Pettersson[1]; [1]Volvo Aero Corporation, Mats. R&D, Maloga, Trollhattan, Vastergotaland 461 81 Sweden

Diffusion bonding of the Ti-alloy Ti-5.8Al-4.0Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si-0.06C (wt.%) to the intermetallic g-based alloy Ti-33Al-2Fe-1.8V-0.1B (wt.%) using hot isostatic pressing at 900ϒC, 200 MPa held for 1 hour was studied. Sound joints without any pores or cracks with a width of approximately 5-7 mm could be produced. Tensile testing showed that the strengths of the joints are similar to the strength of the g-TiAl base material at temperatures between room temperature and 600ϒC. The fracture occurs either at the joint or in the g-TiAl material. The fracture initiation process is a competition between initiation in the g-TiAl base material and initiation at the g-TiAl/diffusion-bond interface. Creep testing showed that most of the creep elongation occurs in the Ti-alloy, but failure is initiated in the joint bond line. Creep causes degradation and pore formation in this line. Interlinkage of these pores creates a crack which is growing slowly until the fracture toughness of the g-TiAl is exceeded and the crack starts to propagate in the g-TiAl material and terminates creep life.

INFLUENCE OF Nb-ION IMPLANTATION UPON HIGH TEMPERATURE OXIDATION BEHAVIOR OF GAMMA TiAl UNDER THERMAL CYCLING CONDITIONS: Yonggang Zhang[1]; [1]Beijing University of Aeronautics and Astronautics, Dept. of Mats. Sci. and Eng., 37 37, Xueyuan Rd., Haidian, Beijing 100083 China

The effect of niobium implantation (3?017ions/cm2) on the oxidation behavior of /Ã-TiAl in air has been studied under thermal cycling conditions for periods of hundreds of hours in the temperature range 850ϒC-950ϒC. The experimental results clearly showed that the thermal cycling oxidation resistance of /Ã-TiAl alloy could be remarkably improved by Nb ion implantation. Nb-implantation followed by post-implantation annealing could further improve the cyclic oxidation resistance of the alloy, and particularly the alloy showed much better resistance to spallation presumably because the implantation improves the mechanical integrity of the alumina scale and decrease the growth rate, and the critical scale thickness for the occurrence of the spallation was reached at a much longer time. The evolution of microstructure and residual stresses in the scale has been examined and measured by SEM and XRD. In this paper, the experiment results will be offered and the mechanism of the cyclic oxidation will be discussed.

LEACHING THEORY PROCESS DEVELOPMENT & INDUSTRIAL PRACTICE: Gold Leaching

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee, Copper, Nickel, Cobalt Committee
Program Organizers: Akram Alfantazi, Falconbridge, Ltd., Falconbridge Technology Centre, Falconbridge, Ontario P0M 1S0 Canada; Arash Kasaaian, Elkem Metals Company, Marietta, OH 45750 USA; Alexandre J. Monteiro, Indosuez Capital Emerging Markets, Sao Paulo, SP 01311-902 Brazil

Monday PM Room: 1B
March 1, 1999 Location: Convention Center

Session Chairs: Dr. D. Dixon, UBC; Dr. M. Misra, University of Nevada, Reno, NV USA

2:00 PM

PROCESS DEVELOPMENT: RECOVERY OF COBALT, GOLD AND BISMUTH FROM A POLYMETALIC CONCENTRATE. PART 1: LEACHING: *A. Mezei*[1]; C. J. Ferron[1]; R. B. Goad[2]; [1]Lakefield Research, Ltd., Lakefield Canada; [2]Fortune Minerals Ltd., Lonton, Ontario Canada

Concentrate samples produced form the NTCO deposit in Northwest Territories were subjected to a comprehensive metallurgical testwork consisting in: pressure oxidation ferric chioride leach and carbon in leach, to recover the cobalt, bismuth and gold, respectively. A specific testwork program was designed to allow for the determination of the optimum parameters under multiple restrictive criteria. The overall objective was to establish a leaching sequence that could produce acceptable recoveries while generating environmentally friendly residues as well as solutions suitable for further processing. The recoveries for the leaching stage, under the optimum conditions were 94% for cobalt, 99% for bismuth and 96% for gold, respectively.

2:20 PM

DEVELOPMENTS IN PERCOLATION LEACHING WITH AMMONIUM THIOSULFATE FOR GOLD EXTRACTION OF A MILD REFRACTORY ORE: *W. T. Yen*[1]; H. Guo[1]; G. Deschenes[2]; [1]Queen's University, Dept. of Mining Eng., Ontario Canada; [2]CANMET, Mining and Mineral Sciences Laboratory, 555 Booth St., Ottawa, Ontario Canada

A mild refractory gold ore containing chalcopyrite was used to investigate the development of thiosulfate as an alternative heap leaching technology. Preliminary bottle roll tests indicated that similar gold extraction was obtained with cyanide and thiosulfate. In the column leach test effects thiosulfate, copper and ammonia concentrations, and their ratio on both gold extraction and reagent consumption, were assessed. The range of reagent concentration were: O.1-O.5 M (MH_4 2S2O3, 0.01-0.1 McuSo3 *5H2) and 1.0-6 OM NH4OH. The solid-liquid ratio was in the range of O.83:1 to 5: 1. Best results indicated that 70% of gold was extracted in 50 days leaching with a solution containing O.3 M (NH4)2S2O3 0.05 M CuSO4* 5H2/esb/O and 6 M NH4/esb/ OH. The reagent consumption at solid/solution of 0.83:1 was 37 kg/t (NH4)2S2O3 and O.62 kg/t CuSO4*5H2O. The result was also compared with cyanide column leach.

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THE PROCESS OF LIXIVATION OF ORES OF GOLD WITH ACIDIC SOLUTIONS OF THIOURREA: *Cesar Garban*[1]; Mokka N. Rao[2]; [1]Fundacite Guayana, Puerto Ordaz Venezuela; [2]Universidad Nacional Experimental de Guayana, P.O. Box 302, Puerto Ordaz 8015 Venezuela

The samples of the washings are characterized of the ore for physical, chemical and metallurgical properties for representatively, The optimum values are determined for the recuperation of gold of the washings of the gold ore from the zone EI CaIIao, Venezuela by acidic solutions of thiourea of the following parameters: Ph, contact time, composition of the liquid mixture, ratio of solid to liquid, grade of liberation, in an apparatus designed for contacting solid powder of the mineral with liquid solution of thiourea maintained in suspension by bubbles of compressed air. The experimental results showed that the velocity of lixivation in primier time of (20) minutes is rapid and practically 213 part of the reaction is completed. For experimental design of parameters the optimum conditions of recuperation of gold determined in this experimental investigation are: PHC5, (NH2) 2IFc2 (S04)3% SOLH,~ Liquid particle size time,1 3:1, 3,325 mesh ,O.4Smm>° hr.<1-hrs.

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HOW THE THERMODYNAMICS EXPLAIN THE THIOSULFATE GOLD LEACHING PROCESS: *Didier Michel*[1]; Jean Frenay[1]; [1]University of Liege, Dept. of Metall. and Mineral Proc., Rue A. Stevart 2, Liege 4000 Belgium

Both laboratory and industrial practice available from the literature show that the thiosulfate gold leaching process is rather versatile and that some curious behaviors are difficult to understand. A complete study of the thermodynamics of that system allows to determine the kinds of complexed regents which catalyze the reactions, and their stability domain. The influence of the different operating parameters on these stability domains is determined and can explain clearly the influence of each components of that sophisticated system on the rate of gold leaching. From that study, indications on the way to manage such system can be found.

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A KINETIC STUDY ON PRESSURE OXIDATION OF PYRITE AT HIGH TEMPERATURE: *Hu Long*[1]; David G. Dixon[1]; [1]University of British Columbia, Dept. of Metals and Mats. Eng., 309-6350 Stores Rd., Vancover B.C. V 6T 1Z4 Canada

Pyrite, the host mineral in refractory gold ores and concentrates, has been treated by pressure oxidation in sulfuric acid solutions at temperatures above 180ϒC to liberate gold prior to cyanidation since the 1980's. However, no data is available on the kinetics of pyrite during acid pressure oxidation over the temperature range of 180 to 230ϒC, which is the range employed by most commercial plants. The dissolution of pyrite (FeS_2) is sulfuric acid solution under oxygen pressure was investigated at temperatures ranging from 170ϒC to 230ϒC. The effects of temperature, particle size, agitation speed, oxygen partial pressure, and pulp density were evaluated. The effects of foreign ions addition and the concentration of sulfuric acid were also examined. The apparent activation energy was estimated to be 42.6 kj/mo1 in the temperature range 170ϒC to 230ϒC. The reaction order with respect to particle size and oxygen partial pressure were found to be -1.5 0.5 at 210|C, respectively. Experimental evidence suggests that the rate-controlling stop of the reaction is likely to be the diffusion of ferrous ions away from the mineral surface.

LIGHT WEIGHT ALLOYS FOR AEROSPACE APPLICATIONS V: High Strength Aluminum Alloys

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Eui W. Lee, Naval Air Warfare Center, Code 4342, MS5, Patuxent River, MD 20670 USA; William Frazier, Naval Air Warfare Center, Aircraft Div. Patuxent River, MD 20670-1908 USA; K. Jata, Wright-Patterson Air Force Base, WL-MLS, Dayton, OH 45433-7718 USA; Nack J. Kim, Center for Adv. Aerospace Mats., Pohang 790-330 Korea

Monday PM Room: 9
March 1, 1999 Location: Convention Center

Session Chair: K. K. Sankaran, Boeing Aircraft, Dept. of Mats., St Louis, MO USA

2:00 PM

EFFECT OF NON-EQUILIBRIUM SOLUTION TREATMENT IN SQUEEZE CAST Al-Cu-Si-Mg-Ag ALLOYS.: Do Hyang Kim[1]; *Yoo Chan Kim*[1]; Yo Sub Han[2]; Ho In Lee[2]; [1]Yonsei University, Dept. of Metall. Eng., 134 Shinchon-dong Seodaemun-ku, Seoul 120-749 Korea; [2]Korea Institute of Science and Technology, Division of Metals, P.O.Box 131, Chengryang-ri, Seoul 130-010 Korea

Dissolution behavior of secondary solidification phases in squeeze cast Al-4.0wt%Cu-1.5wt%Mg, Al-4.0wt%1.5wt%Mg-0.7wt%Ag and Al-4.0wt%Cu-1.5wt%Si-1.0wt%Mg-0.7wt%Ag has studied using a combination of optical microscope, image analyzer, scanning electron microscope(SEM), energy dispersive spectrometer(EDS), X-ray diffractometer(XRD), transmission electron microscope(TEM) and differential scanning calorimeter(DSC). To confirm effect of Ag addition clearly, kinetic analysis measured using DSC. Special emphasis was placed on the investigation of the effects of the non-equilibrium heat treatment - heat treated above the lower eutectic temperature - on the dissolution of the second solidification phases and Ag addition. As-cast microstructure consisted of α-Al and various types of secondary solidification phases such as of Al2Cu, Al2CuMg and Mg2Si. Detailed thermal analysis showed that various type of dissolution occurred depending on the alloy composition. Equilibrium and non-equilibrium solution treatment were carried out at the temperatures of 495ϒ, 502ϒ and 515ϒ for 3 to 5 hours. The amount of the dissolved secondary phases increased with increasing solution treatment temperature, for example, area fractions of Al2Cu, Mg2Si and Al2CuMg were approximately 0%, 1.6% and 4.2% after solution treatment at 495ϒ for 5 hours, and were approximately 0%, 0.36% and 2% after solution treatment at 515ϒ for 5 hours in Al-4.0wt%Cu-1.5wt%Si-1.0wt%Mg-0.7wt%Ag alloys. The best combination of tensile properties was obtained when the as-cast alloy was solution treated at 515 degree for 3 hours followed by aging at 180ϒ for 10 hours. When Ag added, precipitation behavior became faster and activation energy became lower. Detailed DSC and TEM study showed that higher strength in Al-Cu-Si-Mg-Ag alloys was due to: i) dissolution of secondary solidification phases during non-equilibrium solution treatment; ii) the strengthening behavior during aging was due to enhanced precipitation of the platelet type θ' phase; and iii) enhanced precipitation by the addition of alloying element such as Ag.

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EFFECT OF CORROSION ON THE MECHANICAL BEHAVIOR OF AGING AIRCRAFT ALUMINUM ALLOYS : *Krishnan K. Sankaran*[1]; *Kumar V. Jata*[2]; [1]Boeing, Boeing Aircraft, St.Louis, MO USA; [2]Air Force Research Laboratory, 2230 Tenth St., WPAFB, OH 45433-7718 USA

Many of the US Air Force aircraft fleet (such as KC-135) will operate well into the next century and the effect of corrosion on the aircraft structural integrity is a major issue confronting the materials and structures community. Although corrosion is a costly maintenance issue it's effect on safety of flight is also a concern particularly as the USAF fleet ages beyond the original design life. A comprehensive program to understand "corrosion growth rates" and the impact of corrosion on mechanical behavior is in place. The work reported here addresses the "pitting and crevice corrosion growth rates" and effect on fatigue life of aluminum alloys in structural configurations typically used in aircraft construction. Corrosion was induced through exposure of bare 7075-T6 alloy and lap joint specimens to environments for up to several hours. "Corrosion growth rate" was measured optically as a function of time and the growth rate kinetics was obtained. Corroded smooth fatigue coupons as well as lap-joint -type fatigue coupons were tested in laboratory air. A model developed by the Boeing company that computes the life based on the number of cycles needed to grow an initial flaw to a critical crack size for catastrophic failure was used to predict fatigue life. Results suggest a good agreement between measured and predicted fatigue lives of the corroded alloy.

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THE ROLE OF PITTING CORROSION ON FATIGUE CRACK INITATION IN 2024-T3: *L. B. Simon*[1]; T E Matikus[1]; M Khobaib[1]; C S Jeffcoate[2]; [1]University of Dayton, Center for Materials Diagnostics, School of Engineering, Kettering Hall, Dayton, OH 45469 USA; [2]Brookhaven National Laboratory, Uptown, NY USA

In recent years the US Air Force has focused attention on extending the life of its aging fleet. Consequently, the Air Force is concerned about a growing number of aging aircraft that require extensive maintenance. One of the main causes of aircraft failure is due to corrosion damage and fatigue of its aluminum alloy parts. Several types of corrosion damage may affect the aluminum structure of an aircraft. In this paper results of on controlled pitting corrosion are discussed. Pitting corrosion causes the most acute damage in aircraft structures , because pits act as one of the nucleation sites for fatigue crack formation. To study the role of pits in fatigue crack initiation, pits were created on high strength aluminum 2024-T3 samples by an accelerated electrochemical method. These pits have a variety of diameters and depths. The pit morphology was characterized using white light interference microscopy. The pitted surface was examined and parameters such as average roughness and pit depth were determined. I n addition 3-dimensional images of the pitted surface were recorded. These samples were then fatigued and the stress intensity factor was measured. The objective of this study was to characterize corrosion pitting using nondestructive evaluation (NDE) methods and to relate NDE parameters describing the level of corrosion damage to loss of structural integrity of the material due to fatigue.

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NEW HIGH STRENGTH LIGHTWEIGHT MATERIAL FOR TURBOPUMPS: *Patrick B. Berbon*[1]; [1]Rockwell Science Center, MS-A25, 2021049, Caminos Dos Rios, Thousand Oaks, CA 91360 USA

This presentation describes a new procedure to obtain a high strength lightweight Al alloy to be used in turbopumps. Powders are mechanically alloyed in a high energy attritor and in a liquid nitrogen atmosphere in order to obtain the proper composition and a nanocrystalline structure. The resulting powder is subsequently compacted and forged. The obtained material has an extremely fine microstructure and excellent values of specifics strength at room temperature and at liquid nitrogen temperature.

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MICROSTRUCTURAL EVOLUTION IN STRIP CAST Al-Mg-Si-X ALLOYS: *Yong S. Park*[1]; Nack J Kim[1]; [1]Pohang University of Science & Technology, Center for Advanced Aerospace Materials, Pohang 790-784 Korea

DC casting has been the most important casting technique for producing rolling slabs and extrusion billets of Al alloys. However strip casting can be an alternative for the production of rolled Al alloys. Strip casting offers a beneficial effect on the as-cast microstructure due to a relatively high solidification rate and also has an advantage of one step processing of flat rolled products. However, strip casting has primarily been used in the production of Al alloys with limited alloy contents. The present research is aimed at developing the high performance Al-Mg-Si alloys by strip casting. The main processing variables are roll gap, roll velocity, and melt temperature. Microstructure and mechanical properties of strip cast Al-Mg-Si alloys will be discussed with particular emphasis on the solidification behavior during strip casting. The effect of dispersoid-forming Mn addition will also be discussed.

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EFFECTS OF STRAIN ACCUMULATION ON THE SUPERPLASTIC DEFORMATION BEHAVIOR OF 7075 Al ALLOY: *Yong Nam Kwon*[1]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Center for Advanced Aerospace Materials, Pohang 790-784 Korea

The superplastic deformation behavior of a fine grained 7075 alloy has been investigated within the framework of an internal variable theory for inelastic deformation. The theory takes the dislocation glide process within and across the grain boundaries (GMD) as the major accommodation mechanism for the grain boundary sliding (GBS). The flow curves were obtained by performing a series of load relaxation tests at the various prestarin values to examine the effects of accumulated strain on the superplastic deformation behavior. The most significant result obtained in this study is that the grain boundary characteristics changes gradually with the strain accumulation from an initially Newtonian viscous flow signified with the power index value of Mg=1.0 to a Non-newtonina flow with the value of Mg=0.5 commonly observed in the various microduplex alloys such as Ti64. The variation of GBS characterstics with the prestarin is then examined by observing the microstructural evolution with the strain through the use of a TEM.

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WARPAGE BEHAVIOR OF 7075 Al ALLOY : *T. M. Ruprto*[1]; S L Vasquez[1]; A Y Yue[1]; D J Manriquez[1]; J C Quilla[1]; S Hannan[1]; J Foyos[1]; Omar Es-Said[1]; E W Lee[2]; [1]Loyola Marymount University, NSF Research Experience for Undegraduate Program, Los Angeles, CA 90045 USA; [2]NAWC, Code 4342, MS Bldg. 2188, Pauxtent River, MD 20670-1908 USA

Extruded I sections were machined into four different section shapes, L, short depth L, T and short depth T. Points on the samples were taken prior to solution treatment using a three coordinate measuring system. The furnace was preheated at 780°F and the samples were placed inside. The temperature was raised to 880°F at a rate of 50°F per hour. The extrusions were solution treated for 2 hours at 880°F and then quenched in either a 30% polyalkylene glycol solution or water at 15°F. Points on the distorted samples were again recorded and the difference between the measurements indicated the extent of warpage. A finite element analysis predicting thermal gradients of quenched samples is also included. In a second experiment, six tension samples were further machined from each of the sections. Four were parallel and two were perpendicular to the grain orientation. Two of the parallel samples were stretched 0.5% and aged hardened at 340°F for 6 hours and air cooled to T73 tempering for Marine Applications. The tensile properties were evaluated and compared.

MICROMECHANICS AND MICROMECHANISMS OF DEFORMATION AND FRACTURE: A SYMPOSIUM IN HONOR OF PROFESSOR ALI S. ARGON: Session II

Sponsored by: Structural Materials Division, Mechanical Metallurgy Committee, High Temperature Alloys Committee

Program Organizers: K. Jimmy Hsia, University of Illinois, Dept. of Theor. & Appl. Mech., Urbana, IL 61801 USA; Mary Boyce, Massachusetts Institute of Technology, Dept. of Mech. Eng., Cambridge, MA 02139 USA; Tresa M. Pollock, Carnegie Mellon University, Dept. of Metall. Eng. & Mat. Sci., Pittsburgh, PA 15213 USA

Monday PM Room: 14B
March 1, 1999 Location: Convention Center

Session Chairs: Mary C. Boyce, MIT, Dept. of Mech. Eng., Cambridge, MA 02139 USA; Robert E. Cohen, MIT, Dept. of Chem. Eng., Cambridge, MA 02139 USA

2:00 PM INVITED PAPER

MECHANISMS OF DEFORMATION AND TOUGHNESS IN PARTICLE-MODIFIED SEMICRYSTALLINE THERMOPLASTICS: *Robert E. Cohen*[1]; [1]Massachusetts Institute of Technology, Dept. of Chem. Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA

Many semicrystalline polymers exhibit brittle behavior in certain circumstances such as notch brittleness under impact loading. In many instances this brittleness has been alleviated by the incorporation of rubbery particulate components. There exist many ad-hoc explanations for the effectiveness of this practice. Here dramatic improvements of notch brittle polyamide and polyethylene are presented; the mechanism of toughening relies on the percolation through the structure of a crystalline texture of low plastic resistance. This condition is achieved when the half thickness of the inter-particle matrix ligament becomes smaller than the characteristic thickness of the layer of crystallization with preferred orientation. Generalization to rigid-particle modified HDPE will also be discussed.

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STUDIES OF LOCAL PROCESSES INVOLVED IN PARTICLE ADHESION TO POLYMERS: *Hugh R. Brown*[1]; [1]University of Wollongong, Steel Institute, Northfields Ave, Wollongong, NSW 2522 Australia

A new instrument based on atomic force microscope technology have been developed to study adhesion processes of fine inorganic particles to polymer surfaces. This instrument permits both the direct observation of the contact patch using a scanning electron microscope and the measurement of the slow build up of pull-off force. The results can be analysed using contact mechanics to find how particle adhesion is controlled by polymer surface reorganisation and diffusion.

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MORPHOLOGY AND ORIENTATION OF BULK SPHERULITIC POLYPROPYLENE DUE TO PLANE-STRAIN COMPRESSION: *Zbigniew Bartczak*[1]; Miroslaw Pluta[1]; Tomasz Kazmierczak[1]; Andrzej Galeski[1]; [1]Polish Academy of Sciences, Centre of Molecular and Macromolecular Studies, Sienkiewicza 112, Lodz 90-363 Poland

Studies of texture development in isotactic polypropylene (iPP) subjected to the plane-strain compression are reported. Samples of iPP were compressed in a channel-die at 110 degC to various true strains up to 1.89 (compression ratio, CR=6.6). The structure of deformed specimens was investigated by means of microscopy, calorimetry, small- and wide-angle X-ray diffraction techniques and dynamic mechanical analysis. A scheme of morphology changes on all structural levels was proposed. It was found that initial spherulitic morphology was destroyed and was transformed into stacks of crystalline lamellae with their normals rotating towards loading direction with increasing strain up to 1.1 (CR 3), while chain axis tending towards the flow direction. The main active deformation mechanisms found were the crystallographic slips along the chain direction: (010)[001], (110)[001] and (100)[001] slip systems, supported by the deformation of the amorphous component by interlamellar shear. No evidence of twinning modes was found. At higher strain the intense chain slip caused the fragmentation of the lamellae into smaller crystalline blocks due to slip instabilities. That transformation occurred above true strain of 1.39 (CR=4). Further slips in these fragmented crystallites led to formation of a sharp orientation of the chains along the flow direction. The final texture of the compressed iPP found at the true strain of 1.89 (CR=6.6) was the multicomponent texture with two main components of (010)[001] and (110)[001]. Mechanical properties of deformed samples follow the evolution of their structure through successive increase of storage modulus and a decrease of mechanical loss, ascribed to the glass-rubber transition, with increasing strain. The behavior of mechanical loss indicates substantial stiffening of the amorphous component with increasing strain. Using another set of samples the influence of temperature and deformation rate on the structure and properties of iPP deformed by plane-strain compression was investigated. The temperature of plane-strain compression was changed within the range of 130ϒC-160ϒC while the initial deformation rate in the range of 0.02 min^-1 - 1 min^-1. It was found that the stress at any deformation stage decreases markedly with increasing temperature of the deformation process. On the other hand, the variation of the deformation rate within two orders of magnitude influences the deformation to much less extent - the stress increases with increasing deformation rate but the samples deform easily without break up to CR 12 even at the highest deformation rate employed.

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ROLLING OF POLYMERIC MATERIALS WITH SIDE CONSTRAINTS: *Andrzej - Galeski*[1]; Jerzy Morawiec[1]; Zbigniew Bartczak[1]; [1]Polish Academy of Sciences, Centre of Molecular Macromol.Studies, Sienkiewicza 112, Lodz 90-363 Poland

Molecular orientation is one of the advantageous results of plastic deformation of polymeric materials. In most cases it leads to an increase in material's toughness and strength. It is often observed that the plastic deformation of crystalline polymers causes significant amount of cavitation. Cavitation was always identified as the cause of material whitening during loading. Although deformation in a chanel die is kinematically very similar to drawing the pressure component which arises due to compression prevents cavitation. Considerably similar effects can be obtained by rolling of wide strips. Due to a high pressure component in rolling the cavitation is usually not observed. However, for wider strips the force required to roll the material increases unacceptably high if high deformation ratio is targeted, while fissures, cracks and cavitation at edges of a rolled material are formed. An innovative method of obtaining of highly oriented polymeric materials is by unidirectional rolling in a channel geometry by introducing side constraints in rolling. The process relies on rolling of a material inside a chanel located on the circumference of a roll with another roll having the thickness matching the width of the chanel. The side constraints are the side walls of the chanel. The other roll is serving as a plunger. The advantage of rolling with side constraints is the possibility of compressing thick and long shapes in a continuous manner. The resulting shape or rod can have considerably high cross-section area. In the paper the construction of the rolling machine is described. The examples of rolling of isotactic polypropylene and high density polyethylene shapes are presented. The rolling of iPP and HDPE was performed at various temperature by several steps of low compression up to the final compression ratio of 5.4-5.6 with the rolling rate of 4.23 m/min. Long rods of 12mm*10mm in cross-section were produced. The load applied to rolls was up to 250kN at peak. Higher compression ratio lead to a fracture of the samples during rolling with that rate. Tensile strength of the produced rods approaches 200 MPa for both polymers. At a lower rolling rate of 0.17 m/min higher compression ratios were achieved leading to thinner rods but exhibiting tensile strength above 200 MPa. The rolled samples can still accommodate up to 30% of elongation in tensile experiments. Samples rolled at higher temperature show longer elastic response, up to

15% of elongation and then the fracture process sets in. HDPE and iPP rolled rods are highly transparent although they exhibit high crystallinity level. The rods are highly textured as revealed by x-ray pole figures. It is demonstrated that the texture was produced in HDPE by (100)[001], (010)[001] and (100)[010] crystallographic slips while in iPP (010)[001], (110)[001] and (100)[001] slip systems were active. The texture of rolled samples is quite similar to the texture obtained in a chanel die compressed HDPE [1] and iPP samples [2]. The plane strain compression of iPP performed in a channel die with various rates and at various temperatures indicates that it is possible to obtain rods by rolling with side constraints with the tensile strength well above 300 MPa at compression ratio of 10-15 by choosing a proper combination of rolling rate, temperature, initial thickness of a bar and molecular weight of the polymer.

3:40 PM BREAK

3:50 PM INVITED PAPER
MECHANICS OF STRAIN-INDUCED CRYSTALLIZATION IN POLY(ETHYLENE TEREPHTHALATE): *Mary C. Boyce*[1]; Patricia G. Llana[1]; [1]Massachusetts Institute of Technology, Mech. Eng., Room 1-304, 77 Massachusetts Ave., Cambridge, MA 02139 USA

Poly(ethylene terephthalate) (PET) is a thermoplastic polymer found in numerous commercial products including x-ray films, overhead transparencies, and beverage containers. PET products are primarily manufactured by warm deformation processing where the polymer is stretched at temperatures above its glass transition temperature. PET can take on both purely amorphous and semi-crystalline structure. In many processing operations, PET initially begins in an amorphous state and undergoes strain-induced crystallization during processing. This paper presents experiments which explore the mechanical behavior of PET at temperatures above the glass transition where the stress-strain behavior is found to strongly depend on rate, temperature and state of deformation. The evolution in structure with strain and its dependence on rate, temperature and state of deformation is measured using differential scanning calorimetry and wide angle x-ray scattering. A physically-based constitutive model is developed which successfully captures the competition between the effects of molecular relaxation and molecular orientation on the stress-strain behavior at these processing temperatures and rates.

4:20 PM INVITED PAPER
PLASTIC DEFORMATION OF AMORPHOUS POLYMERS; SIMULATIONS AND EXPERIMENTS ON THE ATOMISTIC SCALE: *Ulrich W. Suter*[1]; [1]Institute of Polymers, Dept. of Mats., ETH, CNB E 92, Zurich CH-8092 Switzerland

The simulation of mechanical properties of dense polymer systems has been limited to treatments based on excessively simplifying assumptions. While the elastic properties have largely yielded to quantitative approaches, atomistic in scale for homogeneous media and mesoscopic for heterogeneous materials, the investigation of plastic deformation has not been as successful. Todate, little is known beyond a zero-temperature quasi-static method or Molecular Dynamics techniques that employ stresses higher than the experimental yield stress or strain rates that are orders of magnitude higher than the experimentally and technologically interesting range. A simultaneous effort in modeling and experiment is necessary to proceed. Investigations initiated by Professor Ali S. Argon over a decade ago have been continued in Zurich and in these, solid-state NMR spectroscopy and atomistic-level simulations have been concertedly employed in order to clarify the mechanisms of plastic deformation in amorphous polymers. We will discuss the results from these studies as well as their ramifications for the large-scale deformation of polymeric materials.

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ATOMIC-LEVEL INSIGHTS ON THE NATURE OF AMORPHOUS MATERIALS: *Sidney Yip*[1]; [1]Massachusetts Institute of Technology, Dept. of Nuclear Eng., Room 24-208, Cambridge, MA 02139 USA

Atomistic simulation techniques of molecular dynamics and Monte Carlo are well suited to probing the structural and physical properties of various states of matter. Thus the approach has provided valuable insights into the atomic configurations and dynamical behavior of equilibrium states such as a crystal and a liquid. These techniques are also useful for delineating the properties of disordered media, where our ability to quantitatively correlate local atomic structure with transport and mechanical properties in glassy materials is still limited. In this commentary we will examine several characteristics of the amorphous (metastable) state using as specific examples well-defined models prepared by atomistic simulation, a liquid quenched at high cooling rate, a crystalline solid amorphized by the rapid introduction of self interstitials, and a glassy polymer. We will find in each case illuminating features which contribute to the overall picture that the amorphous material should be viewed as a structurally inhomogeneous entity, its vibrational spectrum is enhanced in the low-frequency region relative to that of a crystal, and the atomistic mechanism for local motion and relaxation is a cooperative percolation-like process. We will also explore connections with the physical basis underlying a successful dynamical theory of relaxation kinetics in supercooled liquids, the self-consistent mode-coupling theory of density correlations, which is currently being tested by a variety of neutron and light scattering experiments.

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THE MICROMECHANICS OF BRITTLE VS. DUCTILE FRACTURE IN AMORPHOUS SOLIDS: *Michael L. Falk*[1]; [1]Harvard University, Div. of Eng. and Applied Sci., Pierce Hall, Cambridge, MA 02138 USA

Molecular-dynamics simulations of fracture in model amorphous solids are shown to exhibit brittle or ductile behavior depending on small changes in interatomic potential. Yet, simulations of these two model solids under pure shear reveal no significant difference in their ultimate yield stress. To understand this change in failure mode we consider the relationship between crack dynamics, rate-dependent plasticity, and molecular-level structures in the glassy solid. In particular we draw connections between Freund and Hutchinson's theory of brittle fracture and the theory of viscoplasticity proposed by Falk and Langer. A simplified model of the microscopics of plastic deformation pioneered by Argon and Spaepen is considered as a first-step toward constructing first-principles models of dynamic plasticity and the brittle ductile transition in noncrystalline materials.

MILTON BLANDER INTERNATIONAL SYMPOSIUM ON "THERMODYNAMIC PREDICTIONS AND APPLICATIONS": Solution Modeling and Solution Databases

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee, ASM International: Materials Science Critical Technology Sector, Thermodynamics & Phase Equilibria Committee
Program Organizers: Ramana Reddy, University of Alabama, Dept. of Met. & Mats. Engr., Tuscaloosa, AL 35487 USA; Dr. A. D. Pelton, Montreal, Quebec H3C3A7 Canada

Monday PM Room: 4
March 1, 1999 Location: Convention Center

Session Chairs: Ramana G. Reddy, University of Alabama, Dept. of Metall. and Mats. Eng. Tuscaloosa, AL 35487-0202 USA; J. Ernesto Indacochea, University of Illinois at Chicago, Civil and Mats. Eng. (MC246), Chicago, IL 60607-7023USA

2:00 PM KEYNOTE
FUNDAMENTAL THEORIES AND CONCEPTS FOR PREDICTING THERMODYNAMIC PROPERTIES OF HIGH TEMPERATURE IONIC AND METALLIC LIQUID SOLUTIONS AND VAPOR MOLECULES: *Milton Blander*[1]; [1]QUEST Research, 1004 E.167th Pl., South Holland, IL 60473-3114 USA

Concepts and theories developed by the author and a large number of coworkers permit one to predict the thermodynamic properties of many high temperature multicomponent liquid solutions (metal alloys and ionic solutions) from the properties of lower order systems. In addition, a dimensional theory leads to reliable predictions of the non-electronic entropies of molecules without knowledge of the structure or vibrational frequencies. The concepts and theories include a cycle first proposed by Flood, Forland and Grjotheim which was used to predict solubility products of ionic solutes in multicomponent molten salt and metallic solutions, a generalized quasichemical theory for predicting temperature coefficients of ionic association constants (and the Wagner interaction coefficients) in dilute solutions in metals and molten salts, and the Coordination Cluster Theory which led to predictions of the temperature and concentration dependence of activities of dilute solutes (e.g. oxygen and sulfur in binary alloys or a molten salt in binary salt solutions). An intuitive approach pioneered by Flood, Forland and Grjotheim combined with quasichemical theory by the author led to a means for the prediction of the thermodynamic properties of multicomponent reciprocal salt systems. The Conformal Ionic Solution Theory proved the validity of these equations for molten salts which had been deduced intuitively. A modified quasichemical theory for molten silicates (and other ordered liquid solutions) permits one to predict the properties of multicomponent silicate solutions from those of the subsidiary binaries. An application of Flory polymer theory led to the prediction of the solubtlities (and capacities such as e.g., sulfide capacities) of sulfides, halides, phosphates, sulfates etc. in molten silicates. Discussion of these concepts and theories will illustrate their usefulness and practicality with some key examples.

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COMPUTATIONAL THERMODYNAMICS FOR LIQUIDS: *Bo Sundman*[1]; [1]Royal Institute of Technology, Stockholm S10044 Sweden

Simulation of materials properties is an essential part in much computer software. The models used are in many cases very crude and valid only within a small range of compositions and temperature. The use of computational thermodynamics with databases and equilibrium software which can provide chemical potentials and driving forces for a wide range of materials is growing but computational times are then much longer. But the predictions that can be made from a better thermodynamic modeling are much more accurate and the treatment of the geometrical and kinetic problems can instead be simplified. Models for the liquid are important in many process problems and solidification simulations and require predictions of solubilities, latent heat and heat capacities. A number of models are currently in use and some comparisons and examples of applications will be given.

3:00 PM

QUASICHEMICAL MODELING OF SHORT-RANGE ORDERING: *Arthur D. Pelton*[1]; Gunnar Eriksson[2]; [1]Ecole Polytechnique, CRCT, P.O. Box 6079, Station Downtown, Montreal, Quebec H3C 3A7 Canada; [2]GTT-Technologies, Kaiserstrasse 100, Herzogenrath 52134 Germany

A modified quasichemical model in the pair-approximation was proposed by Blander and Pelton in 1984. This model has proved successful in modeling short-range-ordering in molten mattes, slags and salts, and in estimating properties of multicomponent solutions from the properties of binary and ternary sub-systems. The model is used in many of the databases of the FACT computer system. The model will be outlined for one- and two-sublattice phases, and recent improvements will be discussed. It will be shown how the model can be formally reduced to an "associate" model, with an entropy correction term, with the pairs as the formal "components." This greatly facilitates the development of unified model software. Polynomial point-approximation models (such as the two-sublattice Compound Energy Formalism) become limiting cases of the quasichemical model. Inter- and intra-sublattice short-range ordering can be treated simultaneously by the quasichemical model in the quadruplet-approximation, which reduces to the pair-approximation as a special case.

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THERMODYNAMICS OF SOLID AND LIQUID METALLIC ALLOYS: Mark Asta[2]; Stephen Foiles[2]; Dane Morgan[1]; Jeff Althoff[1]; *Didier de Fontaine*[1]; [1]University of California at Berkeley, Dept. of Mats. Sci. and Mineral Eng., Berkeley, CA 94720 USA; [2]Sandia National Laboratory, Computational Mats. Sci.. Dept., Livermore, CA USA

The structural and thermodynamic properties of metallic alloys, particularly in the liquid phase, are studied using the embedded-atom method combined with Monte Carlo simulation. Displacive and replacive short-range-order (SRO) and free energies of mixing are calculated as a function of temperature in order to study the effects of undercooling upon liquid alloy properties. Special attention is devoted to chemical SRO in ordering and phase-separating alloys. The roles of atomic size and chemistry effects in determining SRO are investigated. A detailed investigation of the thermodynamics of the Ni3Al system is made, including the solid, liquid, and undercooled liquid phases, with particular attention given to the effects of SRO on solid-liquid free energy differences. (This research is supported by the U.S. Dept. Energy, Office of Basic Energy Sciences, Materials Science Division.)

3:40 PM BREAK

4:00 PM

A NOTE ON ESTIMATION OF PARAMETERS IN THERMODYNAMIC MODELS: *Leiv Olav Kolbeinsen*[1]; [1]SINTEF, Mats. Tech., Alfred Getz v. 2B, Trondheim N-7034 Norway

The general problem of fitting measured data to a model may be stated as wanting to determine the model parameters that best describe the connection between a set of X-variables, often called the independent variables, and a set of (dependent) Y-variables. In the model, $Y = f(X)$, we use "b-coefficients" for the model parameters and X will generally be a matrix with number of columns equal to the number of terms in the model, and the number of lines will be the number of objects (experiments, sets of X/Y-data). The X-variables are terms based on composition information, and the Y-variables are free or excess energies, activities, entropies, enthalpies, etc., depending on the actual model. The alternative often chosen will be a method belonging to the Multiple Linear Regression (MLR) "family". In this case these methods will be impeded by the fact that many of the X-variables of interest will be more or less linearly dependent on each other. This is so because the MLR methods rely on the transpose and inverse of X, or rather the construction $(X^TX)^{-1}$, collinearity in X may have a detrimental effect on the stability of the "b-coefficients" and render them useless for prediction. In this paper it is shown that by employment of Principal Component Regression (PCR) or Partial Least Squares Regression (PLS) collinearity between X-variables represents a stabilizing advantage rather than a problem.

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DEVELOPMENT OF A THERMODYNAMIC DATABASE FOR HARD MATERIALS FOR DESIGN AND PROCESSING SIMULATIONS: *Lucia Dumitrescu*[1]; Malin Ekroth[2]; Bo Jansson[2]; [1]Royal Institute of Technology (KTH), Dept. of Mats. Sci. and Eng., SE-100 44 Stockholm Sweden; [2]SECO Tools AB, 737 43 Fagersta Sweden

Carbonitrides have a wide range of applications in demanding conditions like high temperature, hardness and high strength. The technique to manufacture high quality products based on such materials exists and has been developed over the years. The experience of the well known thermodynamic databases for steels has shown the possibilities to improve properties by calculations. By the CALPHAD approach, using the PARROT module of the Thermo-Calc software, a new database for metal carbonitrides with Co-W-Ti-Ta-Nb-C-N has started. The steps of creating this database from experimental and theoretical bases are presented. As an illustration of the usefulness of the database, for the composition and temperature ranges covered by the database, some materials processing predictions will be given.

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THE PARTITIONING OF TRACE ELEMENTS BETWEEN GAS, SLAG, MATTE AND SALT MELTS DURING THE GASIFICATION OF COAL: *D. Thompson*[1]; Bernard B. Argent[1]; [1]University of Sheffield, Dept. of Mats. Sci., Mappin St., Sheffield S1 3JD UK

The mobilisation of trace elements present in solid fuels used in gasification systems can lead to accelerated deterioration of the plant and to environmental damage. The damage can be caused by transfer of very low levels of trace elements which are difficult to monitor, or

temporary off-design operation leading to transient enhanced mobilisation. A model of the underlying processes which predicts the degree of mobilisation of each element from fuel of specified characteristics is thus desirable. We have used the Equilibrium module of the FACT suite of computer programs to make predictions for trace element mobilisation from a coal which has been the subject of extensive study of trace element speciation and behaviour. The conditions simulated are those of pressurised air blown gasification with and without limestone added to the bed together with cooling of the product gas. The effects of varied sulphur and chlorine levels have been examined. Predictions made using the various oxide melt models, matte, salt and solid solution models available in FACT are combined to allow meaningful comment on Pb, Cu, As, Zn, Ni and Cr distributions. Reasonable agreement is obtained with observation and the cooling study indicates dominant deposition of the elements as sulphides and matte components. If solution of Pb, Zn and Cu chlorides is permitted in alkali chloride melts then they are predicted to be largely removed from the gas stream into these melts.

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RESIDUAL ENTROPY AND THE THIRD LAW: Howard Reiss[1]; and D. Kivelson; [1]Dep't. Chemistry, University of California at Los Angeles, Los Angeles, CA 90025-1569 USA

A system quenched into a "so called" disordered state at zero temperature is said to have a residual entropy that reflects this disorder, i.e. the multiplicity of microstates that corresponds to the single macrostate of the disordered system. However, since the system is trapped in just a SINGLE microstate, even though it is not an ordered one, the system cannot explore the multitude of alternative states that is supposed to give rise to the nonzero residual entropy. Thus the simple requirement of causality demands that the system entropy should still be zero, i.e. S=kln(omega) where omega = 1. We show that, in fact, the entropy is zero, i.e. there is no residual entropy. The appearance of residual entropy in third law cycle experiments is due to the fact that the entropy is measured along a path some of whose segments are irreversible. In order to define the thermodynamic state of the a trapped system, additional constraints, equivalent to additional independent thermodynamic variables must be applied. These constraints convert irreversible segments of a cycle path into reversible ones, and the work of applying these constraints reduces the residual entropy to zero. For example, conventional heat capacity measurements must be reversible, but in cycle experiments some such measurements are not reversible, even though the system may be drifting infinitely slowly toward equilibrium. Entropy, then evaluated via heat capacity measurements is spurious.

NONDESTRUCTIVE EVALUATION (NDE) AND MATERIAL PROPERTIES IV: Nondestructive Evaluation (NDE) and Material Properties Session II

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Nuclear Materials Committee

Program Organizers: Peter K. Liaw, University of Tennessee, Dept. of Mats. Sci. & Eng., Knoxville, TN 37996-2200 USA; Richard J. Arsenault, University of Maryland, Nuclear Eng. Bldg., College Park, MD 20742-2115 USA; Robert E. Green, The John Hopkins University, Baltimore, MD 21218-2689 USA; K. Linga Murty, North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA; R. Thompson, Iowa State University, Ames Laboratory, Ames, IA 50011 USA

Monday PM Room: 16A
March 1, 1999 Location: Convention Center

Session Chairs: Robert E. Green, The Johns Hopkins University, Dept. of Mats. Sci. and Eng., Baltimore, MD 21218 USA; Richard J. Arsenault, University of Maryland, Mats. and Nuclear Eng., College Park, MD 20742-2115 USA

2:00 PM INVITED PAPER

MATERIALS CHARACTERIZATION BY RAPID X-RAY DIFFRACTION IMAGING: *Robert E. Green*[1]; [1]The Johns Hopkins University, Center for Nondestructive Evaluation, 3400 N. Charles St., 206 Maryland Hall, Baltimore, MD 21218 USA

This paper will give a brief history of real-time x-ray x-ray diffraction imaging instrumentation. Next, an overview will be presented of research efforts illustrating how rapid x-ray diffraction imaging has served to study the plastic deformation of metals, grain boundary migration during recrystallization, structure of explosively loaded metals, transformation of rapidly solidified amorphous metals to the more stable crystalline state, and structural phase transformations in ferroelectric crystals. In combination with synchrotron radiation rapid x-ray topographic imaging reveals vibrational modes in quartz crystals, and defects in quartz, gallium arsenide, and nickel alloy turbine blade single crystals. Finally, comments will be made with respect to recent new developments which will contribute to improvement in operating systems and increase in spatial resolution of real-time-x-ray diffraction images.

2:30 PM INVITED PAPER

NONDESTRUCTIVE EVALUATION OF FATIGUE OF ALCLAD 2024T3 RIVETED LAP JOINTS: *Zayna M. Connor*[1]; Morris E. Fine[1]; Jan D. Achenbach[1]; [1]Northwestern University, 2225 N. Campus Dr., Evanston, IL 60208 USA

The nondestructive study of short fatigue cracks in riveted lap joints has long been a dilemma due to the cracks originating in the subsurface of the joint and not being visible on the outside surface. The scanning acoustic microscope may be used to quantitatively investigate subsurface fatigue cracks such as at and near countersunk rivets in riveted lap joint specimens even when they are very small. When combined with optical and electron microscopic examination of the surface and with fractography of fractured specimens, the formation and growth of subsurface cracks near rivets may be characterized in detail. Such an investigation was made on specimens fabricated from two pieces of Alclad 2024-T3 sheet material riveted with 2017-T4 aluminum alloy flathead chamfered rivets. These specimens are similar to the riveted lap joint found in the fuselages of many aircraft. The fatigue testing was interrupted periodically for examination and some specimens with just detectable and larger subsurface cracks were fractured in tension to reveal

the fatigue fracture surface for analysis. A detailed study of crack formation and microcrack growth kinetics near such rivets is presented including location of the initiation sites as affected by applied load.

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QUALITY CLASSIFICATION FOR Al-Cu DIFFUSION BONDS: *Diane J. Chinn*[1]; Chol K. Syn[1]; Graham H. Thomas[1]; [1]Lawrence Livermore National Laboratory, P.O. Box 808, L-333, Livermore, CA 94551 USA

Lawrence Livermore National Laboratory (LLNL) is implementing classification techniques to correlate information contained in ultrasonic bondline signals with the quality of the bond. Quality of the bond is determined by strength testing and microstructural analysis. Sets of aluminum-copper diffusion bond specimens are tested in ultrasonic pulse-echo mode. Feature extraction and feature selection software identify the pertinent characteristics of the ultrasonic signals for bond quality determination. The classification software is based on statistical pattern recognition. The best classification process is determined for a set of ultrasonic signals from specimens with known bond quality. The classifier performance is verified on a set of ultrasonic signals from unknown bonds. *Work performed under 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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SCANNING SQUID MICROSCOPY AS AN NDE TOOL FOR STRUCTURAL STEELS: *J. W. Morris*[1]; J. W. Chan[1]; J. Clarke[2]; T. J. Shaw[2]; [1]University of California, Dept. of Mats. Sci. and Eng., Berkeley, CA USA; [2]University of California, Dept. of Phy., Berkeley, CA USA

This paper describes initial research on the potential of the scanning SQUID microscope as a probative tool for the non-destructive characterization of ferromagnetic materials such as structural steels. The research employs a high-Tc scanning SQUID microscope, recently developed at LBNL, that combines high magnetic sensitivity with good spatial resolution. Research to date has primarily addressed microstructural changes and plastic deformation in carbon steels. By measuring small changes in remanent magnetization, the SQUID can detect local regions of plastic deformation and map deformation gradients. It can also distinguish the different microstructures developed during heat treatment of carbon steel, and map microstructural gradients. The sources of the magnetic changes that are detected by the SQUID microscope will be discussed.

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MICROSTRUCTURAL STRESS/STRAIN INHOMOGENEITY IN MMCS: *Bjørn Clausen*[1]; Mark A. M. Bourke[1]; [1]Los Alamos National Laboratory, Manuel Lujan Jr. Neutron Scattering Center, P.O. Box 1663, Mail Stop H805, Los Alamos, NM 87545 USA

Neutron diffraction measurements have been used to verify finite element calculations of mean phase stresses and strains in composite materials with varying degrees of success. In reality most materials display elastic and plastic anisotropy on the scale of the microstructure. This results in variation in the lattice strains for different hkl reflections, which may not be the same in monolithic material as in the same material used in a composite. The continuum mechanic finite element models can not provide information at this level of detail, but using a self-consistent polycrystal deformation model it is possible to predict the elastic lattice strains as measured by neutron diffraction. Measured and predicted results for different MMC systems is presented.

OUTCOMES ASSESSMENTS FOR ABET CRITERIA 2000

Sponsored by: Accreditation Committee, Education Committee
Program Organizers: Joseph F. Thomas, Wright State University, School of Graduate Studies, Dayton, OH 45435 USA; David R. Gaskell, Purdue University, School of Mats. Eng., West Lafayette, IN 47907 USA

Monday PM Room: 13
March 1, 1999 Location: Convention Center

Outcomes assessment has become prominent in engineering education for the purpose of program improvement, accountability, and accreditation. Most recently the criteria for accreditation of engineering programs by ABET has transitioned from criteria based upon program inputs to Criteria 2000 based in large part on outcomes assessment. This requires a major adjustment in faculty viewpoints and the nature of preparations for an accreditation review. Program faculty will need to set objectives, determine desired education outcomes, select and apply assessment tools, and use assessment results for program improvement. Alignment of institution and department issues and long range plans will be critical to success. Preparation for accreditation will become a continuous rather than a periodic process. Speakers in this session are among the leaders in this visible transition in promoting quality in engineering education. The objectives of the session are to introduce you to outcomes assessment and report on the implementation of ABET Criteria 2000. The desired outcome is your involvement in a successful accreditation review for your institution. Industry participants are encouraged to learn about the process and actively support it.

2:00 PM SPEAKERS PANEL

George D. Peterson Executive Director Accreditation Board for Engineering and Technology Baltimore, MD 21209

Keynote: Outcomes-Based Engineering Program Accreditation

Gerald L. Liedl School of Materials Engineering Purdue University West Lafayette, IN 47907-1289

Developing Degree Program Objectives and Outcomes

L. Fredrick Norris Howmet Research Center, Howmet Corporation Whitehall, MI 4946

Accreditation and Quality: An Industry View

Anthony D. Rollett Dept. of Mats. Sci. & Eng. Carnegie-Mellon University Pittsburgh, PA 15213

Assessment of Education Outcomes at Carnegie Mellon University

Ashok Saxena School of Materials Science and Engineering Georgia Institute of Technology Atlanta, GA 30332-0245

Assessment, Accreditation, and Accountability: Beyond ABET

Carl J. McHargue Center for Materials Processing University of Tennessee Knoxville, TN 37996-2350

Preparation for Meeting ABET Criteria 2000

4:30 PANEL DISCUSSION

REVIEW OF EXTRACTION PROCESSING, PROPERTIES & APPLICATIONS OF REACTIVE METALS: Session II

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizers: Brajendra Mishra, Colorado School of Mines, Dept. of Metall. & Mats. Eng., Golden, CO 80401-1887 USA; Georges J. Kipourous Dal Tech, Dalhousie University, NS B3J2X4 Canada

Monday PM Room: 5B
March 1, 1999 Location: Convention Center

Session Chairs: D. R. Sadoway, Massachusetts Inst. of Technology, Dept. of Mats. Sci. and Eng., Cambridge, MA 02139-4307 USA; Dr. J. N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA

2:00 PM
MAGNESIUM: *G. J. Kipouros*[1]; [1]Dal Tech, Dalhousie University, NS B3J2X4 Canada

2:30 PM
CALCIUM: *B. Mishra*[1]; S. Benjamin[1]; [1]Colorado School of Mines, Dept. of Metall. & Mats. Eng., Golden, CO 80401 USA

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ZIRCONIUM AND HAFNIUM: *J. Haggarth*[1]; [1]Oremet Wah Chang, P.O. Box 460, Albany, OR 97321 USA

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NIOBIUM: *S. Yuan*[1]; [1]Cabot Corporation, Performance Materials, P.O. Box 1609, County Line Rd., Boyertown, PA 19512 USA

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TUNGSTEN: *K. Osseo-Asare*[1]; [1]Pennsylvania State University, Dept. of Mats. Sci. and Eng., 202-A Steidle Bldg., Univeristy Park, PA 16802 USA

SHEET METAL FORMING TECHNOLOGY: Session II

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee
Program Organizer: Mahmoud Y. Demeri, Ford Research Labs, Manuf. Sys. Dept., Dearborn, MI 48121 USA

Monday PM Room: 11B
March 1, 1999 Location: Convention Center

Session Chair: Amit K. Ghosh, University of Michigan, Mats. Sci. & Eng. Dept., Ann Arbor, MI 48109 USA

2:00 PM
AN INVESTIGATION OF THE STRAIN EVOLUTION IN THE BENDING-UNDER-TENSION FRICTION TEST: *Matthew Joseph Alinger*[1]; Sriram Sadagopan[1]; David K. Matlock[1]; [1]Colorado School of Mines, Metall. and Mats. Eng., 1400 Illinois, Golden, CO 80401 USA

Laboratory friction tests, designed to assess the frictional behavior and formability of automotive sheet steels, rely on direct measurement of pulling and contact forces for friction assessment. One such test is the bending-under-tension (BUT) friction test in which a strip is pulled through one bend as it slides over a cylindrical die surface of a specific radius. To gain a more complete understanding of laboratory friction data, it is necessary to evaluate the bending and unbending response of the sheet as it contacts the forming die. This response can be characterized by the strain history experienced by a sheet during a BUT test, and is assessed both experimentally and numerically. Direct strain gage measurements of longitudinal strain are compared to predictions from a 2-D, plane strain, finite element model of the BUT test. Data were obtained on a zinc coated automotive sheet steel. The implications of the bending and unbending strain path on the calculated frictional coefficients are presented and discussed.

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STRAIN RATE EFFECTS ON SHEET METAL DURING CYCLIC BENDING UNDER TENSION: *L. Rafael Sanchez*[1]; [1]University of Colorado at Denver, Mechanical Engineering Dept., Campus Box 112, 1200 Larimer Street, Denver, CO 80217-3364 USA

A finite difference method was used to estimate the effects of strain rate on plane strain sheet metal forming. The model was used to describe the strain rate effects on the flow of the sheet under cyclic bending and unbending under tension. The strain rate effects were experimentally evaluated by pulling the sheet through a set of three roller "frictionless" device at various speeds. Experimental measurements were performed on high strength steel, mild steel and aluminum alloy sheet metal. An estimate of the strain rate sensitivity was obtained and substantiated with experimental data. The significance of strain rate on the mechanics of deformation of the materials tested was discussed. Although the strain rate sensitivity was found to be small, its effects on the total deformation could not be neglected due to the strain rate gradient caused by bending and unbending.

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THE COLD BENDABILITY OF SHEET METALS: *Mai Huang*[1]; *James C. Gerdeen*[2]; [1]Weirton Steel Corporation, Techonolgy Center, 3006 Birch Dr., Weirton, WV 26062-5133 USA; [2]University of Colorado at Denver, Dept. of Mech. Eng., 1200 Larimar St., Denver, CO 80217-3364 USA

The bendability, e.g. the minimum bending radius that sheet metal can endure when it is bent along a straight line, is a critical process parameter in some sheet metal forming operations (such as in a hemming operation of automotive exterior panels) and an important subject for research. It is found that all results of bendability published previously were based on elementary bending theory with the simplifications for small curvature bending. In this paper, an analysis is developed for the sheet metal of anisotropic work hardening materials subjected to large curvature plane strain bending. A strain ratio (R) was introduced to represent the anisotropy in the thickness direction. It is shown that the result of Datsko and Yang [1] that has been adapted in the text book [2] is a special case of the current analysis. It is also proven that their result was obtained with an oversimplified assumption. Two examples were given to examine and illustrate the theory.

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NUMERICAL SIMULATION OF THE U-BENDING PROCESS FOR VDIF STEEL SHEETS USING DAMAGE ANALYSIS AND LS-DYNA: *Chi L. Chow*[1]; Weihua Tai[1]; Mahmoud Y. Demeri[2]; [1]University of Michigan-Dearborn, Dept. of Mech. Eng., 4901 Evergreen Rd., Dearborn, MI 48128 USA; [2]Ford Motor Company, Manufacturing Systems Dept. / Scientific Research Labs, 20000 Rotunda Drive, Dearborn, MI 48121 USA

This paper presents a computer simulation of the U-bending process of VDIF steel sheets using LS-DYNA. The analysis is based on the theory of damage mechanics that is incorporated in LS-DYNA through its user-defined transversely anisotropic plasticity model. The plane-strain stretching, bending, unbending and straightening of sheet metal are considered during the forming operation and the damage evolution and accumulation are recorded. The effect of plastic damage on strain localization and ductile failure of the sheet metal is studied by simulating

two U-channel forming processes. In the first process, the numerical procedure is terminated at the threshold of localized necking to determine the limit strains. In the second process, varying clamp forces are applied to produce different uniform major strains prior to localized necking, such that the effects of plastic damage, clamp force, friction condition, and die radius can be examined. Good agreement is achieved between the numerical and experimental results.

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NUMISHEET'93 2D DRAW BENDING SPRINGBACK SIMULATION REVISIST: *Yang Hu*[1]; [1]Chrysler, ASME, 800 Chrysler Dr., Auburn Hills, MI 48326 USA

Although formability study using various commercial FEA codes has been widely adopted by the automobile industry, springback compensation based on FEA prediction is rarely applied. Achieving a consistent accuracy of springback prediction remains a tough challenge for FEA software vendors. The problem is addressed by various authors who claim that one of the contributors to inaccurate and inconsistent prediction is the dynamic effect inherent in dynamic FEA codes such as LSTC-Dyna3D [Ref 1 2]. It is also suggested that more accurate friction models and material models should be employed in order to obtain an accurate consistent prediction. [Ref. 3] In this paper, a springback prediction study is discussed on numisheet-93 2D draw bending springback analysis. An input parameter sensitivity analysis is discussed and the emphasis is addressed on the penalty factor and non-uniform friction coefficient. It is suggested that an improved contact algorithm should be employed in order to achieve consistent accuracy on springback prediction while a proposed non-uniform friction coefficient has little effect on the results of numisheet-93 2D draw bending springback prediction.

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THE EFFECT OF STRAIN PATH ON FORMABILITY OF SHEET METAL: *Abdi Majlessi*[1]; X. H. Zhu[1] ; E. C. Aifantis[2] ; Faruk Unsakar[3] ; [1]Michigan Tech University, Department of Mechanical Engineering-Eng. Mechanics, 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Laboratory of Mechanics, Aristotle University of Thessaloniki, Thessaloniki 540 06 GREECE; [3]Visiting Professor, Selcuk University, TURKEY

A method is developed to predict the forming limit diagram (FLD) of sheet materials based on their deformation history. The analysis employs Hill's 79 yield function for a planar isotropic material, and uses the gradient theory to prescribe the constitutive equation for the flow stress. In order to utilize a previously developed analysis for proportional loading, a concept of an "effective deformation" is introduced. The "effective deformation" is defined to have a strain path identical to the final deformation stage, and yet would produce a plastic strain energy the same as the actual deformation it is replacing. Using this concept, the entire deformation path can be treated as a continuous proportional strain. Although the present technique is capable of treating any complex strain history, it has been applied only to a bilinear strain path in this paper. Analytical results show that a uniaxial tension prestrain raises the FLDo upwards and shifts it to the left. An equibiaxial-stretch prestrain has the opposite effect. A plane-strain prestrain does not shift FLDo but reduces the size of the failure zone of FLD. The agreement between predicted FLD and experimental results is satisfactory.

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SIMPLE PREDICTION OF SHEET METAL FORMING LIMIT STRESSES AND STRAINS: *Kamineni Rao*[1]; Emani V. R Mohan[1]; [1]City University of Hong Kong, Dept. of Manuf. Eng. & Eng. Mgmt., Tatchee Ave., Kowloon, Hong Kong China

A method to predict Forming Limit Curves (FLCs) from tensile test data of sheet materials is proposed. In this method, a single limit yield stress is first obtained from a tension test. Using Hill's anisotropic yield criteria, a continuous limit yield locus can be calculated. It has been reported that several materials exhibit linear limit stress with respect to stress ratio. Such linear limit stress boundary may be obtained by simple regression of the elliptical limit stresses, and either of them can be termed as Forming Limit Stress Curve (FLSC) depending on which case is true. From this FLSC, FLCs corresponding to any strain path can be obtained using relevant hardening equation, normality flow rule and Hill's anisotropic yield criterion. Analytical results obtained for deep drawing quality steels and brass will be presented and compared with experimental data.

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ON THE RELATIONSHIP BETWEEN TEXTURE TYPES AND MICROSTRUCTURE IN COLD ROLLING AA3003 ALLOY SHEET: *Wen Xi Yu*[1]; [1]The Hong Kong Polytechnic University, Dept. of Manuf. Eng., Hong Kong China

In this paper, pole figures of AA3003 aluminum alloy sheets were measured and their Orientation Distribution Function (ODF) was calculated. Transmission Electron Microscopy (TEM) studies were performed and correlations between texture types and microstructure were obtained for this material. The relationship between grain orientation in the rolling plane of the sheets and the calculated ODF was analyzed and discussed.

STRUCTURAL SILICIDES: Corrosion, Creep, Wear

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee; ASM International: Materials Science Critical Technology Sector, Mechanical Behavior of Materials Committee

Program Organizers: J.H. Schneibel, Oak Ridge National Laboratory, Metals & Ceramics Div. Oak Ridge, TN 37831 USA; Michael J. Kaufman, University of Florida, Dept. of Mats. Sci. & Eng., Gainesville, FL 32611-2066 USA; Matthew J. Kramer, Iowa State University, Ames Laboratory, Ames, IA 50011USA

Monday PM Room: 16B
March 1, 1999 Location: Convention Center

Session Chairs: N. K. Natesan, Argonne National Laboratory, Argonne, IL 60439 USA; M. J. Kramer, Iowa State University, Ames, Iowa 50011-3020 USA

2:00 PM INVITED PAPER

THE EFFECT OF MICROSTRUCTURE AND COMPOSITION ON THE ENVIRONMENTAL RESISTANCE OF MOLYBDENUM-RICH Mo-Si-B ALLOYS: *Douglas M. Berczik*[1]; Mark Garguilo[1]; [1]United Technologies-Pratt & Whitney, MS 707-26, P.O. Box 109600, West Palm Beach, FL 33410-9600 USA

Recent research into high temperature materials for use in the turbine of an advanced jet engine has been concentrated on the oxidation resistant T2 phase (Mo_5SiB_2) and alloys in the molybdenum metal Mo_5SiB_2 Mo_3Si phase field. While alloys that are high in T2 volume fraction (>60 volume percent) demonstrate the best oxidation resistance, it is the alloys that contain molybdenum metal as the majority phase that hold the greatest promise for possessing the needed balance of environmental resistance and mechanical properties. This paper will present some of the preliminary work in defining the effect of the relative volume fraction and morphology of the phases present as well as the overall chemistry and silicon to boron ratio on the environmental resistance of alloys containing more than 50 volume percent molybdenum metal.

2:30 PM INVITED PAPER

HIGH TEMPERATURE PROPERTIES OF LIGHT ELEMENT DOPED Mo AND Ti SILICIDES: *M. Akinc*[1]; Andrew J. Thom[1]; Matthew J. Kramer[1]; Ozer Unal[1]; Jason J. Williams[1]; Jesse J. Huebsch[2]; R. Radhakrishnan[1]; [1]Iowa State University, Ames Laboratory, Ames, IA 50011 USA; [2]Seagate Technology, 7801 Computer Ave, Minneapolis, MN USA

Transition metal disilicides show excellent high temperature oxidative stability but suffer from poor creep strength. In contrast, silicides with the general formula A_5Si_3 (A = transition metal) generally show good high temperature creep strength but exhibit poor oxidation resistance. The oxidation resistance of these silicides can be greatly improved by doping with light atoms such as B, C, and O. Mechanisms for enhancing the oxidation resistance in Mo_5Si_3 (tetragonal) and Ti_5Si_3 (hexagonal) by light element additions were shown to differ. For Ti_5Si_3, a promising light weight material, interstitials such as C dramatically improve the oxidative stability by strengthening the bonding between the Ti and interstitial atoms and hence weakening the Ti-Si bonds. Recent results of structural studies from neutron diffraction and high temperature diffraction experiments using synchrotron radiation will be discussed. Boron doping improved oxidation resistance in the Mo-Si system by several orders of magnitude. The improvement in oxidation resistance is due to the formation of a borosilicate scale that has a reduced viscosity that promotes formation of a protective, continuous scale. The creep rate of boron doped material was comparable to undoped Mo_5Si_3. Fracture toughness and flexural strength values obtained from four-point bending tests will also be discussed. These properties appear to be highly dependent on microstructure, with fine grained powder processed samples showing superior properties. The Ames Laboratory is operated by the U.S. Department of Energy (DOE) by Iowa State University under Contract No. W-7405-ENG-82. This work was supported by the Office of Energy Research, Office of Computational and Technology Research, Advanced Energy Projects Division.

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COMPARISON OF PROPERTIES BETWEEN Mo-Si-B AND $MoSi_2$ ALLOY SYSTEMS: *A. K. Vasudevan*[1]; Mysore A. Dayananda[2]; M. G. Hebsur[3]; K. Sadananda[4]; [1]Office of Naval Research, Materials Science, 800 N. Quincy St., Code-332, Arlington, VA 22217-5660 USA; [2]Purdue University, School of Mats. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907-1289 USA; [3]NASA Lewis Research Center, 21000 Brookpark Rd. MS49-1, Cleveland, OH 44135 USA; [4]Naval Research Laboratory, 4555 Overlook Ave., SW, Code 6323, Bldg. 71, Washington, D.C. 20375-5000 USA

Comparison of microstructural, mechanical and environmental properties of Mo-Si-B and $MoSi_2$ systems will be considered in light of the microstructural stability. The comparison will highlight the key differences that can be used as a guide line for the development of these alloys for high temperature engine applications. The emphasis of the high temperature oxidation stability in these systems is also included, as the oxidation resistance can be adversely affected if there are possibilities of microstructural instabilities with long time exposures. Such comparison between these two systems can bring forth the lessons learned from the current $MoSi_2$ systems to aid in the development of the new Mo-Si-B systems.

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THE INFLUENCE OF Fe-IMPURITIES ON THE OXIDATION BEHAVIOR OF $MoSi_2$-BASED COMPOSITES AT 1600ϒC: O. Lensch[1]; F. Dettenwanger[1]; *M. Schuetze*[1]; R. Rix[2]; V. Guether[2]; R. Scholl[3]; [1]Karl-Winnacker-Institut der DECHEMA e.V., Theodor-Heuss Allee 25, 60486 Frankfurt am Main Germany; [2]GfE Metalle und Materialien GmbH, Nuernberg Germany; [3]FhG Institut fuer angewandte Materialforschung, Dresden Germany

The production of materials via the powder metallurgical route in many cases involves a ball-milling step in the powder production stage. Usually steel balls are used which means that quite some amount of Fe can be introduced into the powder by abrasion of the milling balls. In particular for materials which are intended for the use at very high temperatures, i.e. at 1500ϒC and higher, the Fe content can lead to a severe deterioration of the oxidation resistance by the formation of low melting Fe containing oxide phases with components from the bulk material. This was identified to be a particular problem for $MoSi_2$-based P/M-materials with second phase particles of different oxides and borides. Thus, a quantification of the Fe-effect is of high technical importance. In order to take a first step towards a quantitative evaluation of the influence of the Fe-contents on the oxidation behavior of such materials 8 different $MoSi_2$ based composites with 15 vol.-% ZrO_2, HfO_2, Y_2O_3, Al_2O_3, SiC, TiB_2, ZrB_2 and HfB_2 as second phase particles and containing 0.2 or 2 wt.-% Fe were investigated at 1600ϒC in air. After the oxidation tests the samples were examined by metallographic investigation, SEM, EPMA and X-Ray diffraction. The samples with 0.2 wt.-% Fe generally showed a better oxidation resistance compared to the samples with the higher Fe content. The samples with 2 wt.-% Fe developed a SiO_2-film interrupted by nodules consisting of Fe-oxide and the corresponding refractory metal oxide. The high Fe amount effects that the composites with HfO_2, HfB_2 and TiB_2 were badly damaged. The surface of the composite with Y_2O_3 became liquid as did the Al_2O_3 containing composite. The only composite among those containing 2 wt.-% Fe which showed a satisfactory behavior was that with 15 wt.% ZrB^2 indicating that this combination of phases is less prone to a deterioration of the oxidation properties by the presence of higher amounts of Fe. In the paper a ranking of the different materials concerning their sensitivity to Fe induced decrease of oxidation resistance is given and the oxidation processes are analyzed.

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TESTING OF SILICIDE AND ALUMINIDE BASED MATERIALS IN A COMBUSTION AND ENDOTHERMIC ENVIRONMENT: *Richard G. Castro*[1]; Darryl P. Butt[1]; Kendall J. Hollis[1]; John J. Petrovic[1]; Brian Bartram[1]; Harry S. Kurek[2]; [1]Los Alamos National Laboratory, Mats.Sci. and Tech. Div., P.O. Box 1663, Mail Stop G770, Los Alamos, NM 87545 USA; [2]Institute of Gas Technology, Mats. Sci. and Tech. Div., 1700 South Mount Prospect Rd., Des Plains, IL 60018-1804 USA

Nine different samples were tested in a combustion and a carburizing (endothermic gas at 0.3% carbon potential) environment at temperatures on the order of 1025°C for approximately 500 hours. The samples included SCRB21 (siliconized SiC), hot pressed $MoSi_2$, $MoSi_2$-30%SiC, $MoSi_2$-30%Si_3N_4, plasma sprayed $MoSi_2$ and Mo_5Si_3 and nickel and iron aluminides. Four-point bend test samples were attached on the inside and outside of a radiant heating tube which was operated in a commercial scale carburizing furnace. Test results showed significant degradation of plasma sprayed Mo_5Si_3 when exposed to the combustion and endothermic environment. Plasma sprayed $MoSi_2$ also showed significant degradation when exposed to the combustion environment with an improved performance when exposed to the endothermic heat-treating environment. Minimal damage was observed for the rest of the samples tested. Microstructural and mechanical property information will be presented on the nine different samples before and after exposure to the combustion and endothermic environment.

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CREEP OF NIOBIUM-SILICON BASED ALLOYS: P. W. Whiting[1]; B. P. Bewlay[2]; *C. L. Briant*[1]; A. W. Davis[1]; [1]Brown University, Division of Eng., P.O. Box D, Providence, RI 02912 USA; [2]General Electric Company, Research and Development Center, Schenectady, NY 12301 USA

This paper reports a study of creep mechanisms in composites derived from Nb-16Si based alloys. These alloys were prepared by directional solidification, and a range of alloying additions were made to this base material to determine their effects on creep behavior. Creep tests were performed in compression with the axis of compression parallel to the growth direction of the directionally solidified composites. The results show that additions of Ti, Hf, and Mo at sufficiently high levels increased the creep rate significantly. In particular, additions of 7.5% Hf had little effect on the creep rate, whereas additions of 12.5% Hf caused the creep rate to increase by approximately an order of magnitude. An increase in Ti from 21 to 33% caused a similar increase in the creep rate. The paper will also discuss the relationship between composite microstructure and creep mechanisms.

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MICROSTRUCTURE IN A SILICIDE DEFORMED AT ELEVATED TEMPERATURES: *Jinguo Wang*[1]; L. M. Hsiung[1]; T. G. Nieh[1]; Fuming Chu[2]; C. T. Liu[3]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci., P.O. Box 808, L-370, 7000 East Ave., Livermore, CA 94551 USA; [2]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Bldg. 4500S, MS-6115, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

A two-phase polycrystalline silicide alloy (nominal composition: Mo_5SiB_2) was made by arc casting. The mechanical properties of the silicide in compression at elevated temperatures (~1300ϒC) were characterized. TEM microstructures reveal that there exist many dislocations in the alloy even in the as-cast state. The nature of these dislocations will be identified. Dislocations appear to be mainly emitted from grain and interface boundaries. High-temperature deformation results in the formation of stable dislocation substructures. In the present paper, both the mechanical properties and microstructure will be presented. It was found that the hardening and recovery behaviors of the alloy are closely related to the dislocation substructural evolution.

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CORROSION AND WEAR RESISTANT Cr-SILICIDES: *Joseph William Newkirk*[1]; Jeffrey A. Hawk[2]; [1]University of Missouri-Rolla, Dept. of Metall. Eng., Rolla, MI 65409 USA; [2]U.S. Department of Energy, Albany Research Center, 1450 Queen Ave. SW, Albany, OR 97321 USA

Silicides based on refractory metals can have a very high hardness, similar to ceramic materials, but with the high thermal and electrical conductivity of metals. This allows these materials to be machined by EDM, unlike ceramic materials. The toughness of these silicides can be improved by adding ductile second phases to the matrix, while the hardness can be increased by adding hard carbides. The resulting composites have good wear properties, good high temperature properties, and good corrosion resistance in many harsh environments. The processing and properties of silicides based on Cr will be presented, with emphasis on their wear resistance and some corrosion results in sulfuric acid.

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THE DEVELOPMENT OF CAST NICKEL SILICIDE FOR SULFURIC ACID APPLICATIONS: *Joseph William Newkirk*[1]; Sanhong Zhang[1]; [1]University of Missouri-Rolla, Dept. of Metall. Eng., Rolla, MI 65409 USA

Nickel silicide (Ni_3Si) has long been known for its resistance to sulfuric acid. Nickel silicide can be given good ductility by alloying with Ti or Nb, but these elements have been found to affect the corrosion resistance of the alloy. Nb has been found to offer better corrosion properties than Ti. This paper describes the problems of balancing good mechanical properties with the corrosion resistance to sulfuric acid, and the effects of alloying elements, heat treatment, and casting technique. Typical properties will be reported. Also, in order to weld these alloys, welding rods have been fabricated and welding trials successfully carried out.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Laser Ablation and Surface Modification

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee

Program Organizers: Yip-Wah Chung, Northwestern University, Dept. of Mats. Sci & Eng., Evanston, IL 60208 USA; Ashok Kumar, University of South Alabama, Dept. of Elect. & Comp. Eng., Mobile, AL 36688-0022 USA; John E. Smugeresky, Sandia National Labs, Livermore, CA 94551-0969 USA

Monday PM Room: 7B
March 1, 1999 Location: Convention Center

Session Chairs: Jorn Larsen-Basse, National Science Foundation, Arlington, VA 22230 USA; Chang-Beom Eom, Duke University, Dept. of Mech. Eng. and Mats. Sci., Durham, NC 27708 USA

2:00 PM INVITED PAPER

SURFACE ENGINEERING OF SILICON AND CARBON BY PULSED LASER ABLATION: *Douglas H. Lowndes*[1]; Antonio J. Pedraza[2]; Jason D. Fowlkes[2]; Vladimir I. Merkulov[1]; A. A. Puretzky[1]; David B. Geohegan[1]; [1]Oak Ridge National Laboratory, Solid State Division, P.O. Box 2008, Oak Ridge, TN 37831-6056 USA; [2]University of Tennessee, Dept. of Mats. Sci. & Eng., Dougherty Engineering Bldg., Knoxville, TN 37996-2200 USA

In this paper we describe experiments in which pulsed excimer laser radiation with well-defined fluence has been used to control and vary the surface morphology, structure, and properties of two important industrial materials, silicon and carbon. Dense forests of silicon microcolumns were produced by repetitive pulsed KrF (248 nm, ~35 ns duration) laser irradiation of (001)-oriented silicon wafers. The individual Si columns can be more than 20 μm tall, are 2 to 3 μm in diameter, and appear to be single crystals with the same orientation as the substrate. For irradiation in air using KrF fluences of 1.5-2.5 J/cm2, the column morphology is fully developed after ~1000 laser pulses. Further morphological evolution is due to modification of these columns. The initial development and the evolution of columns can be altered or suppressed by using different ambient atmospheres (Ar, N2, SF6, vacuum) or by changing the laser fluence and number of laser shots. The formation of these dense, regular arrays of Si micro columns clearly involves elements of self-organization, i.e. a self-assembly process. A model for microcolumn formation will be outlined and related to the VLS (vapor-liquid-solid) mechanism for (catalyst-assisted) growth of whiskers, nanotubes, and nanowires. Striking systematic changes also have been observed in the sp3 bonding content, optical properties, and surface morphology of hydrogen-free tetrahedral amorphous carbon (amorphous diamond) films that were deposited by pulsed ArF (193 nm, ~19-25 ns duration) laser ablation of a pyrolytic graphite target in vacuum. In-situ ion probe measurements were used to monitor and control the mean kinetic energy, KEmp, of the ablated C+ ions. Electron energy loss spectroscopy and scanning ellipsometry measurements of the sp3 bonding fraction, plasmon peak energy (~film density), and optical (Tauc) energy band gap reveal that films with the most diamond-like properties are produced at a C+ KEmp of ~90 eV. Tapping-mode AFM measurements show that films deposited near this energy are extremely smooth (rms roughness ~1 Å). By reducing KEmp to ~ 30 eV, highly sp2-bonded (graphitic) films are produced with corresponding changes in surface morphology and properties. When ablation is done into an inert ambient gas, gas-phase collisions dissipate the kinetic energy and produce carbon clusters so that films consisting largely of carbon nanoparticles can be deposited. Examples of changes in the surface structure and properties of single- and multi-layered films containing these different forms of carbon will be presented. This research was sponsored by the Oak Ridge National Laboratory (ORNL), managed by Lockheed Martin Energy Research Corp. for the U.S. Dept. of Energy, under contract DE-AC05-96OR22464, and was partially supported by the Defense Advanced Research Projects Agency contract DARPA-MIPR-97-1357 with ORNL.

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A NEW APPROACH TO LASER DIRECT WRITING ACTIVE AND PASSIVE CIRCUIT MESOSCOPIC CIRCUIT ELEMENTS: *D. B. Chrisey*[1]; A. Pique[1]; R. C. Y. Auyeung[1]; R. A. McGill[1]; R. Chung[1]; S. Lakeou[1]; P. Wu[1]; M. Duiganan[2]; [1]Naval Research Laboratory, Washington, D.C. USA; [2]Potomac Photonics, Inc., Lanham, MD USA

We have combined some of the major positive advantages of laser induced forward transfer (LIFT) and matrix assisted pulsed laser evaporation (MAPLE), to produce a novel excimer laser driven direct writing technique which has demonstrated the deposition in air and at room temperature and with sub-10 μm resolution of active and passive prototype circuit elements on planer and nonplaner substrates. We have termed this technique MAPLE DW (direct write). This presentation will outline the simplistic approach to carry out MAPLE DW, give experimental conditions, and physical characterization results for the deposition of carbon thin film resistors, gold conducting lines, yttrium-iron-garnet and hexagonal ferrite patches for antenna, high ferroelectric patches, and multilayer depositions of conductors and dielectric to produce prototype capacitors and RC circuits. In addition, an overview other important aspects contained in the MAPLE DW approach, e.g.,

the ability to do in situ laser micromachining, surface pre- treatment, and annealing, will be given. Lastly, the future directions of this project and an appraisal of other approaches outside of the scope of our efforts will be presented.

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GROWTH AND MATERIAL PROPERTIES OF TITANIUM CARBIDE THIN FILMS FOR BALL-BEARING APPLICATIONS: *G. Radhakrishran*[1]; P. M. Adams[1]; [1]The Aerospace Corporation, Mechanics and Materials Technology Center, Los Angeles, CA USA

Advanced materials are being designed and tested for high-stress, high-cycle bearings employed in spacecraft mechanisms. Hybrid bearings consisting of ceramic or ceramic-coated steel balls and steel raceways are being used to provide good fatigue performance and wear resistance in such applications. One of the coating materials that has received serious consideration in hybrid systems is titanium carbide (TiC). Until now the deposition of TiC has involved a process that requires heating the steel substrates to fairly high temperatures (900ºC). Problems such as spallations and changes in composition have been observed with the TiC coatings. In light of these results, it has become critical to examine the issues involved in the deposition of TiC coatings for these applications, in particular, the deleterious effects of using a high temperature deposition process. This talk will describe the use of Pulsed Laser Deposition (PLD) to deposit high quality thin films of TiC on various bearing steels at room temperature. Such a process eliminates the problems associated with high temperature deposition, and the costs and complexities involved in the post-deposition heat treatment of steels. PLD of TiC films on steels, the material properties of these films, and the performance of TiC-coated steel bearings will be described.

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DEPOSITION AND CHARACTERIZATION OF CARBIDES AND NITRIDES HARD COATINGS USING LASER ABLATION METHOD: *Ashok Kumar*[1]; [1]Department of Electrical Engineering, University of South Alabama, Mobile, AL 36688 USA

Carbides and nitrides of metal have a large number of applications in modern technology owing to their interesting, and in some ways unique, physical and chemical properties. Thin film coatings of carbides (titanium carbide, silicon carbide and boron carbide) and nitrides (titanium nitride, silicon nitride, and aluminium nitride) were deposited on Si (100) substrates using pulsed laser deposition (PLD) method. The structural and microstructural properties of these films have been characterized using x-ray diffraction, scanning and transmission electron microscope and FTIR techniques. The mechanical properties of the films were evaluated to measure the hardness and modulus values. It has been shown that the films depsosited at higher temperature have the best crystalline quality structure and also have higher hardness and modulus values compared to the film deposited at lower temperature. Optimization of laser deposition parameters to obtain high quality thin films will be discussed in detail.

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NOVEL THIN FILM SOLID LUBRICANTS AND HARD COATINGS BY PULSED LASER BASED TECHNIQUES: *Jeffrey S. Zabinski*[1]; [1]AFRL/MLBT, Nonstructural Materials Branch, Wright-Patterson AFB, OH 45433-7750 USA

Pulsed laser deposition (PLD) based techniques can be used to grow solid lubricant and hard coatings with chemistry and microstructure optimized for controlling friction and wear in extreme environments such as elevated temperature and outer space. Laser wavelength, pulse duration, frequency, and fluence control plasma energy and composition, which in turn determine the physicochemical properties and performance of the coatings. Additional control and flexibility can be achieved by supplying reactive gases during growth and by adding energy to the plume by, for example, capacitive coupling, double laser shot, and ion beams. By simultaneously using PLD with other complementary deposition techniques (e.g., magnetron sputtering and ion beam assisted deposition), coatings with functional gradients, multilayers, nanostructures, and novel chemistries can be grown. In this presentation, different hybrid PLD configurations and how they permit design of unique coatings are discussed. Emphasis will be on the control of energy through substrate bias, ion beam assist, sample/target geometry, and degree of plasma ionization. By strategically controlling energy to the growing interface, coating chemistry and microstructure are adjusted in a predictable fashion. A review of tribological coatings which includes low friction nanostructured oxides and composite hard coatings with hard and lubricious phases will be provided to illustrate the coating designs attainable.

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HIGH ENERGY METAL ION BEAM ASSISTED DEPOSITION: *James R. Treglio*[1]; [1]ISM Technologies, 13100 Kirkham Way, Suite 211, Poway, CA 92064 USA

Experiments have been conducted on high energy (> 10 keV) metal ion beam assisted deposition. The experimental consisted of bombarding the substrate with high energy titanium ions during the deposition of titanium nitride. The deposition metal was formed via a cathodic arc with a nitrogen backfill. The high energy ions were extracted from a cathodic arc ion source operating with an extraction voltage of 30 kV. For stainless steel substrate, the ion beam was used to sputter clean the surface before deposition. It as found that the high energy metal ion bombardment during deposition increased the adhesion of the coating to the substrate and the coating hardness, while also lowering the residual stress in the coating. For aluminum oxide substrate, the ion beam was also used to ion implant titanium into the oxide before deposition. Without the high energy ion bombardment during deposition, the coating did not adhere at all the alumina substrate. However, with ion bombardment relatively good adhesion was obtained. Further research is underway to extend the process to large areas, and to explore other coatings, including chromium nitride.

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ION IMPLANTATION SURFACE TREATMENTS FOR Al AND Ni RESULTING IN IMPROVED TRIBOLOGICAL PROPERTIEs: M.T. Dugger[1]; D.M. Follstaedt[1]; J.A. Knapp[1]; S.M. Myers[1]; [1]Sandia National Laboratories, Albuquerque, NM 87185-0340 USA

Ion implantation has been used to produce surface microstructures in aluminum and nickel that impart substantial increases in strength and wear resistance to these materials. Finite element modeling of ultra-low load indentation tests were used to extract the mechanical properties of the implanted layers, and low load unlubricated sliding experiments have been used to examine the impact of increased surface strength on tribological behavior. Implantation of oxygen into aluminum results in a high concentration of nanometer-sized precipitates that impede dislocation motion and increase the flow stress to 2.9 GPa. This surface strengthening impairs adhesive junction growth and prolongs the onset of adhesive wear in this material. Implantation of titanium and carbon into polycrystalline nickel results in an amorphous surface layer with flow stress up to 5 GPa. In addition to blocking dislocation motion, strong binding reactions between the Ti and C atoms contribute to the increased strength. Significant increases in the number of contact cycles to the onset of adhesive interactions were observed during unlubricated sliding. For both systems, the change in wear mechanism resulted in reductions in the sliding friction coefficient. The mechanisms of extreme strengthening identified in Al and Ni are being explored in other metal systems.

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TIN FORMED BY LASER GAS ALLOYING (LGA) OF Ti-6Al-4V: *R. R. G. M. Peters*[1]; S. Liu[1]; [1]Colorado School of Mines, Dept. of Metall. and Mats. Eng., Center for Welding, Joining and Coating Research, Golden, CO 80401 USA

Titanium nitride is formed by melting Ti-6Al-4V alloy using a continuous wave Nd:YAG laser. Optical Microscopy (OM) and SEM analysis were done to investigate the microstructure and morphology of the coated surface. Auger electron Spectroscopy (AES), Energy Dispersive Spectroscopy (EDS), XPS and XRD analysis were done to determine the chemical composition, the structure and the stoichiometry of the formed nitride. The OM and SEM showed a dendrite microstructure with alpa-Ti

and retained beta-Ti in the intermediate region in the molten area of the sample. The XRD showed TiN, alpha-Ti and beta-Ti. XPS analysis determined that the formed nitride is near-stoichiometric TiN. From AES and EDS analysis it was found that the dendrites consisted of large amounts of nitrogen and titanium with low concentrations of vanadium and aluminum. From this it was concluded that this was the TiN. The interdendrite region showed low amounts of nitrogen and high concentrations of vanadium and aluminum, hence the formation of alpha-Ti and beta-Ti.

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Ti(NCO) LAYERS FORMED BY MO-PACVD: *Sun Kyu Kim*[1]; [1]University of Ulsan, School of Mats. and Met. Eng., Mugeo-dong, Ulsan 680-749 Korea

Recently, metallo-organic compounds have become donors of titanium in the PACVD process because a chlorine atmosphere is to be avoided. A Ti(NCO) layer was formed on tool steels by using titanium tetraisopropoxide, hydrogen and nitrogen under glow discharge conditions. Layers of good quality were obtained when the content of Ti(OC3H7)4 vapor in the gas atmosphere was 2% to 5%. The layer obtained with H2/N2 gas ratio of 1:1 had the highest hardness of HV 1645. The layers had lower hardness when using H2/N2 gas ratio of 7:3 and 3:7. The layer obtained at 500 E with H2/N2 gas ratio of 1:1 had the highest hardness and the layer obtained at 500 E with H2/N2 gas ratio of 3:7 had the highest corrosion resistance. This process was combined with the plasma nitriding process to obtain composite Ti(NCO) layers on tool steels. The layers thus obtained had high hardness, good wear and corrosion-resistant properties.

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REVIEW OF MULTILAYER FILMS FOR TRIBOLOGICAL APPLICATIONS: *Wei Zhang*[1]; Binshi Xu[1]; Shining Ma[1]; Xunji Xue[2]; Xushou Zhang[2]; [1]Surface Engineering Research Institute of CMES, Beijing 100072 China; [2]Chinese Academy of Sciences, Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Lanzhou 730000 China

The ever-increasing demands of design and production engineers have led to the research for new materials with significantly improved properties. Limitations to the further advance of manufacturing industry in the 21st century are most likely to be surface-related. With the advances in the field of surface engineering, many mechanical systems can operate under more severe application conditions. In recent years, multilayer films received more and more attention. Many favorable properties such as high yield strength, hardness enhancement, improved magnetic and optical properties and excellent tribological properties have been observed in various nano-multilayer film systems. In this paper, the research and development of multilayer films for tribological applications are reviewed. The microstructure and wear mechanism of multilayer films will be covered. Some directors for future research in tribology are also indicated.

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SURFACE MODIFICATION OF METALS USING HIGH-FREQUENCY, LOW-VOLTAGE PLASMA IMMERSION ION IMPLANTATION (HLPIII): *X. B. Tian*[1]; X. F. Wang[1]; S. F. Wang[1]; B. Y. Tang[1]; P. K. Chu[1]; [1]City University of Hong Kong, Dept. of Physics and Mats. Sci., 83 Tat Chee Ave., Kowloon, Hong Kong China

Plasma immersion ion implantation (PIII) is a cost-effective, non line-of-sight technique emulating conventional beam-line ion implantation in the surface modification of irregular samples. However, conformal implantation into a complex target is still difficult due to the large ion sheath. By reducing the ion sheath length using low-energy conditions, the implant uniformity can be dramatically improved. Coupled with high frequency pulsing which increases the sample temperature, a thicker modified layer can be produced further enhancing the wear and corrosion resistance of the treated components. We have recently developed a pulsing modulator employing an IGBT based switching device capable of delivering pulses at a peak voltage of 5kV at 10 B 50 kHz. In this paper, we will present our new hardware as well as recent results of our high-frequency, low-voltage plasma immersion ion implantation (HLPIII) experiments.

SYNTHESIS OF LIGHTWEIGHT METALS III: Applications and Processing

Sponsored by: Light Metals Division, Aluminum Committee; Structural Materials Division, Titanium Committee; ASM International: Materials Science Critical Technology Sector, Materials Synthesis & Processing Committee

Program Organizers: F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; C.M. Ward Close, DERA Farnborough, Struct. Mats. Ctr., Farnborough, Hampshire GUI14OLX UK; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Neqev Israel; P. M. McCormick, University of Western Australia, Res. Ctr for Adv. Min. & Mats. Proc., Nedlands, W.A. 6907 Australia

Monday PM Room: 10
March 1, 1999 Location: Convention Center

Session Chairs: O. Inal, New Mexico Tech., Dept. of Metall. Eng., Socorro, NM 87801 USA; Lutfi Ovecoglu, Istanbul Technical University, Dept. of Metall. Eng., Instanbul 80626 Turkey

2:00 PM INVITED PAPER

SOME RECENT DEVELOPMENTS IN LIGHT METALS APPLICATIONS - PART I: *Chenggong Li*[1]; F. H. (Sam) Froes[2]; [1]BIAM, P.O. Box 81, Beijing 100095 China; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The status of the applications of light-weight materials will be reviewed with emphasis on transportation industries including aerospace, automobiles, rolling stock (trains), marine vessels and bicycles. While performance is important the criticality of reducing cost will be discussed.

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SOME RECENT DEVELOPMENTS IN LIGHT METALS APPLICATIONS - PART II: *Chenggong Li*[1]; F. H. (Sam) Froes[2]; [1]BIAM, P.O. Box 81, Beijing 100095 China; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The status of the applications of light-weight materials will be reviewed with emphasis on transportation industries including aerospace, automobiles, rolling stock (trains), marine vessels and bicycles. While performance is important the criticality of reducing cost will be discussed.

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SYNTHESIS OF ADVANCED MATERIALS BY THERMAL PLASMA PROCESSING: *Sutham Niyomwas*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Dept. of Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

The thermal plasma processing has played an important role in many applications such as plasma spraying, thermal plasma chemical vapor deposition, waste processing, plasma synthesis of fine powders, and etc. Among them the potentials for developing new materials technologies is increasingly recognized. Advance materials such as powder of SiC, B4C, TiC, TiB2, TiN, and ZrC have been successfully synthesized. In this study, it will give a special attention on synthesis powder of TiN-Fe metal matrix material using a non-transferred direct current (DC) plasma method. Raw materials are TiO2 powder, Fe2O3 powder, methane and ammonia. The standard Gibbs energy minimization method has been used to calculate and plot the standard Gibbs energy minimization curve of this system. The synthesis-plasma reactor was designed. The vaporization time of input particle size 40 mm at power supply to the plasma torch 35 kW requires about 14 ms. While the resident time is about 42 ms for the reactor size ID 9 cm x 45 cm long. The effect of

particle size, gas flow rate, and resident time to reactor size will be discussed in the final part of the article.

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ELECTRON BEAM PROCESSING OF ADVANCED LIGHTWEIGHT METALS: *Vadim J. Jabotinski*[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow , ID 83844-3026 USA

Electron Beam Processing offers the potential for cost effective production of advanced lightweight metals. This paper will discuss the production of monolithic alloys and composite concepts using Electron Beam Processing.

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COMPACTION OF ADVANCED METALS: *M. L. Ovecoglu*[1]; O. N. Senkov[2]; Osman T. Inal[3]; F.H. (Sam) Froes[2]; [1]Istanbul Technical University, Dept. of Metall. Eng., Faculty of Chem. Metall., Maslak Istanbul 80626 Turkey; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [3]New Mexico Tech, Dept. of Metall. Eng., Jones 161, Socorro, NM 87801 USA

The present paper reports on a comparative study of two different compaction methods, i.e. hot isostatic pressing (HIP) and dynamic compaction processes used to consolidate advanced metals. Due to simultaneous application of temperature and pressure during HIP'ing, metal powders can be compacted to full density in complex, near-net shapes. On the contrary, dynamic compaction methods do not allow consolidation of complex shapes and because of less temperature-time exposure during dynamic compaction, compacts exhibit less than 100% density. Despite these drawbacks, dynamic compaction methods offer the advantage of complete retention of the powder microstructure during consolidation.

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AN ASSESSMENT OF PLASMA SPRAYING AS A METHOD FOR FABRICATION OF METAL AND INTERMETALLIC MATRIX COMPOSITES: *Adam M. Baker*[1]; P. S. Grant[1]; K.-H. Baik[1]; [1]Oxford University, Dept. of Mats., Oxford Centre for Advanced Materials and Composites, Parks Rd., Oxford OX1 3PH England

Low pressure plasma spraying (LPPS) and Atmospheric Plasma Spraying (APS) have been used to fabricate Al alloy, Ti alloy and $MoSi_2$ Metal Matrix Composites reinforced with unidirectional DERA Sigma and Textron SiC fibres. Al and Ti alloy LPPS monotapes have been densified by subsequent Vacuum Hot Pressing (VHP). SiC fibres have been chemically extracted from both as-sprayed monotapes and VHP monotapes and mechanically tested to compare damage incurred by this processing route with damage caused by the alternative solid state Foil Fibre Foil (FFF) and the Matrix Coated Fibre / Hot Isostatic Press (MCF/HIP) processes. Reduction of fibre damage from LPPS/VHP damage has been achieved by fibre selection, powder size and VHP process cycle modification. The extent and microstructural nature of the reaction between the C coating of DERA Sigma 1140+ SiC fibre after LPPS and VHP has been examined by transmission and scanning electron microscopy; this is compared with previous work on the older DERA Sigma 1240 SiC fibre and the Textron SCS-6 SiC fibre. The development of this interface reaction during heat treatment is analysed. The matrix microstructure and mechanical properties resulting from LPPS/VHP have been characterised, this data is used in conjunction with fibre damage data and bulk composite tests to assess the quality of LPPS/VHP MMCs compared with equivalent FFF and MCF/HIP material. LPPS/VHP Ti MMCs are found to be inferior to MCF/HIP material but are competitive in certain respects with FFF material, although the LPPS/VHP process shows more promise for Al-Si and $MoSi_2$ matrices.

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COMPARATIVE EXTRUDABILITY OF 7075 PARTICULATE Al2O3 COMPOSITES AND ALLOY: *E. V. Konopleva*[1]; Hugh J. McQueen[1]; [1]Concordia University, Dept. of Mech. Eng., H-549, 1455 de Maisonneuve Blvd. West, Montreal, Quebec H3G 1M8 Canada

The hot extrusion of Al matrix particulate composites produced by liquid metal matrix mixing and billet casting is optimized for 0,10 and 15% Al_2O_3 in 7075 alloy by finite element modeling. The constitutive equations were determined through torsion testing in the ranges 250-540ϒC, 0.1 to 4 s_{-1}. In the hyperbolic sine equation the stress exponent was found to be 2.5 to 3.9 and the activation energies 180 to 310 kJ/mol being higher for higher particle content. The ductility increased from 300 to 400ϒC but decreased above that. Extrusion was modeled as a two dimensional slice from an axisymmetric billet for initial temperatures (450, 475, 500ϒC) and ram speeds (2.6, 5 mm/s) for extrusion ratio 31. With sticking friction conditions, there was a dead zone and maximum near the die corner in distributions of (i) either velocity or strain rate and (ii) of either mean stress or temperature which depend on the hot strength of the materials and influence the occurrence of extrusion defects. Comparisons are made to flow patterns from physical simulations and other models. The pressure stroke curves, with traditional peak and sharp then gradual decline, are compared to results of similar modeling and trial extrusions.

THE MARTIN E. GLICKSMAN SYMPOSIUM ON SOLIDIFICATION AND CRYSTAL GROWTH: Fundamental: Solidification and Crystal Growth

Sponsored by: Materials Processing and Manufacturing Division, Solidification Committee

Program Organizers: Dr. N. B. Singh, Northrop Grumman Corp., Pittsburgh, PA 15235 USA; Dr. Steven P. Marsh, Naval Research Laboratory, Code 6325, Washington, D.C. 20375 USA; Krishna Rajan, Rensselaer Polytechnic Inst., Dept. of Mats. Sci. and Eng., Troy, NY 12180-3590 USA; Prof. Peter W. Voorhees, Northwestern Universtiy, Dept. of Mats. Sci. and Eng., Evanston, IL 60208 USA

Monday PM Room: 11A
March 1, 1999 Location: Convention Center

Session Chairs: Steve Marsh, Naval research Laboratory, Mats. Div., Washington, D.C. 20375 USA; Rohit Trivedi, Iowa State University, Dept. of Mats. Sci. and Eng., Ames, IW 50001 USA; Que-Tsang Fang, Alcoa Technical Center, Molten Metal Processing, Alcoa Center, PA 15069 USA

2:00 PM INTRODUCTION

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WHY TWINNED DENDRITES IN CAST ALUMINUM ALLOYS?: *M. Rappaz*[1]; S. Henry[2]; [1]Ecole Polytechnique, Laboratorie de Métallurgie Physique, MX-G, Fédérale de Lausanne, CH -1015 Switzerland; [2]Research Center of Pechiney, Voreppe France

Under certain solidification conditions, aluminum alloys exhibit a twinned dendrite morphology. Recent Electron Back Scattered Diffraction (EBSD) experiments combined with detailed microscopy observations have clearly shown that such morphologies are made of finely spaced columnar [011] dentrites trunks split in their middle by a (111) twin plane. [101] and (110] secondary arms, giving birth to [011] tertiaries/primaries, have been shown to quickly propagate the straight/coherent twinned planes. The impingement of [110] [110], side arms growing laterally results in wave/incoherent twin boundaries. Based upon these observations, a growth mechanism has been proposed: it involves a change in the surface tension anisotropy of aluminum alloys, a possible contribution of the attachment kinetics and convection effects. As a matter of fact under other conditions <110> and <112> untwinned dendrites have also been observed in bulk and thin aluminum specimens, besides the conventional <100> dendrites and <110> twinned dendrites.

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ENVIRONMENTAL NOISE EFFECTS IN STATISTICAL COARSENING THEORY: *Steven P. Marsh*[1]; Dan I. Zwillinger[2]; Martin E. Glicksman[3]; [1]Naval Research Laboratory, Code 6325, 4555 Overlook Ave. SW, Washington, D.C. 20375-5343 USA; [2]Aztec Corporation, 371 Moody St. - Suite 104, Waltham, MA 02154 USA; [3]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., Troy, NY 12180-3590 USA

Statistical mean-field theories of phase coarsening (Ostwald ripening) employ an effective diffusion distance that is a function of both the domain size and of the global volume fraction to formulate the characteristic growth rate of each size class. The corresponding single-valued growth rate function yields self-similar size distributions that are narrower than those generally observed experimentally and in numerical simulations. A stochastic growth function has been derived that permits an extension of the mean-field formalism to account for variations in the size-dependent growth rates ariosing from local interactions. The resulting continuity analysis yields a Fokker-Planck equation that tends to broaden the corresponding size distribution. Effects of the noise term on coarsening rate constants and on the shape of the size distribution at various volume fractions will be discussed.

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A DYNAMIC MODEL FOR THE INTERACTION BETWEEN AN INSOLUBLE PARTICLE AND AN ADVANCING SOLID/LIQUID INTERFACE: *V. Catalina*[1]; *D. M. Stefanescu*[1]; [1]The University of Alabama, P.O. Box 870202, Tuscaloosa, AL 35487 USA

Models that describe the interaction of an insoluble particle with an advancing solid - liquid interface are based on the assumption of steady-state. In this work a dynamic mathematical model was developed. The model was tested for the aluminum-ZrO2 system. The calculated values for critical velocity of pushing/engulfment transition were in same range with the experimental ones. The dynamic model shows that this interaction is essentially non-steady state and that steady-state eventually occurs only when the solidification is conducted at subcritical velocities.

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SCALING PARAMETER(S) IN DENDRITIC GROWTH: *Vladimir Pines*[1]; Arnon Chait[1]; Marianne Zllatkowski[1]; [1]NASA Lewis Research Center, Brook Park Rd., Cleveland, OH 44135 USA

A simple scaling analysis is developed to examine the fundamental relations in dendritic growth between dynamic parameters such as dendrite tip radius and growth velocity, and the dimensionless net heat flux through the dendrite surface (Péclet number). It is demonstrated that a priori assumption of a single scaling parameter as implied from modern selection theories cannot be justified both theoretically and experimentally. From the fundamental scaling theory, a two parameter expansion in Péclet number is shown to be sufficient to fit the entire range of data in supercooling of Glicksman's recent microgravity experiments. The physical origin of the new parameter is traced to the characteristic size of the perturbed nucleus, and its effect is demonstrated to be limited to the low supercooling regime.

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FINDINGS FROM USING SUCCINONITRILE TO MODEL GRAIN GROWTH IN THIN FILMS: *Mark A. Palmer*[1]; Krishna Rajan[2]; Martin E. Glicksman[3]; [1]Virginia Commonwealth University, Mech. Eng., 601 West Main St., P.O. Box 843015, Richmond, VA 23284-3015 USA; [2]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., Materials Research Center, 110 Eighth St., Troy, NY 12180-3590 USA; [3]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., CII 9111, 110 Eighth St., Troy, NY 12180-3590 USA

Succinonitrile has been used by Prof. Glicksman and others to model metallurgical transformations. As a low melting, transparent material, it lends itself to in-situ laboratory observations. Because of this, succinonitrile can be used to complement computer simulation in predicting processing phenomenon. For example, recent work has assessed the applicability of predictions made by Mullins and Von Neumann describing the 2-D grain growth, Fortes and Ferro predicting topological events in polycrystalline materials, Avron and Levine applying Mullins' and Von Neumann's to grains conforming to a curved surface, and many authors using computer simulation to predict grain size distribution. This work will be reviewed briefly. While conducting the investigations mentioned earlier the following was noted. There is a natural variation in the product of grain boundary energy and mobility which accounts for small grains showing slight deviations from ideal behavior. There is a natural broadening of the grain size distribution as time progresses. Finally, using succinonitrile one can observe the transition which occurs as a mushy zone becomes a polycrystalline aggregate. These results and their implications will be discussed.

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MULTIPLE SIMILARITY SOLUTIONS FOR SOLIDIFICATION AND MELTING: *S. R. Coriell*[1]; G. B. McFadden[1]; W. J. Boettinger[1]; R. F. Sekerka[2]; [1]National Institute of Standards and Technology, A153 Materials, Gaithersburg, MD 20899 USA; [2]Carnegie-Mellon University, Dept. of Physics, Pittsburgh, PA 15213 USA

When a solid phase of uniform temperature and composition is brought into contact with a liquid phase of different temperature and composition, there exist (under the assumption of local equilibrium at the interface) similarity solutions for which the position of the solid-liquid interface is proportional to the square root of time. The parabolic growth rate constant satisfies a transcendental equation which may have three solutions, similar to the findings of Kirkaldy et al. for isothermal ternary diffusion. We examine the stability of these similarity solutions with respect to planar perturbations and find that the solution with the intermediate value of the growth rate constant is unstable. We then relax the assumption of local equilibrium by assuming a linear kinetic law for the interface temperature and perform a small time expansion, which leads to a single solution with a finite initial velocity. Numerical finite-difference calculations of the interface motion and stability are presented.

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CHARACTERIZATION OF CAST METALS WITH PROBABILITY DISTRIBUTION FUNCTIONS: *Michael Steinzig*[1]; Francis H. Harlow[1]; [1]Los Alamos National Laboratory, T3, MS B216, Los Alamos, NM 87545 USA

Characterization of microstructure using a probability distribution function (PDF) provides a means for extracting useful information about material properties. In the extension of classical PDF methods described here, material characteristics are evolved by propagating an initial PDF through time, using growth laws derived from consideration of heat flow and species diffusion, constrained by the Gibbs-Thomson law. A model is described for determining the final global distribution of grain configuration, micro/macro segregation of species, residual stresses, and other relevant material properties. Results are shown from implementing the model into a finite-difference metal casting code.

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MEASUREMENTS OF SOLUTE SEGREGATION COEFFICIENTS IN QUARTERNARY Ni-BASED SUPERALLOYS: *Shyh-Chin Huang*[1]; Lou Peluso[1]; Dan Backman[2]; [1]GE Corporate Research and Development, Schenectady, NY USA; [2]GE Aircraft Engines, Lynn, MA USA

Solute segregation coefficients are material parameters important to the understanding of the solidification behavior of alloys. In directionally solidified nickel-based superalloys, where segregation induced defects such as freckles need to be controlled, the determination of the segregation coefficients is particularly important. This paper presents three methods used to measure the segregation coefficients in a model superalloy (Ni-Cr-Al-Ta) using electron microprobe. The methods were devised to have significantly different scan modes, but the results appear quite similar as long as the solid state precipitation of gamma prime phase can be minimized by post-cast heat treatment. Other model alloys were also studied (Ni-Cr-Al-W and Ni-Cr-Al-Re), and the Al segregation coefficient was found to change with the quaternary element. The implication of the segregation coefficient measurements will be discussed with respect to freckle formation. This work was supported by ARPA under the ICCA Micromodeling program.