TECHNICAL PROGRAM

The 128th TMS Annual Meeting & Exhibition

San Diego Convention Center ✻ San Diego, California U.S.A. ✻ February 28 - March 4, 1999

1999 TMS ANNUAL MEETING & EXHIBITION
FEBRUARY 28 - MARCH 4, 1999

San Diego Convention Center

	Monday March 1		Tuesday March 2		Wednesday March 3		Thursday March 4
	AM	PM	AM	PM	AM	PM	AM
Room 1A				General Recycling of Materials I	General Recycling of Materials II	General Recycling of Materials III	
Room 1B	Leaching Theory Process Development & Industrial Practice I	Leaching Theory Process Development & Industrial Practice II	Leaching Theory Process Development & Industrial Practice III	Leaching Theory Process Development & Industrial Practice IV	Abatement of Greenhouse Gas Emissions in the Met & Matls Proc Industry		
Room 2	Fluid Flow Phenomena In Metals Processing I	Fluid Flow Phenomena In Metals Processing II	Fluid Flow Phenomena In Metals Processing III	Fluid Flow Phenomena In Metals Processing IV	Fluid Flow Phenomena In Metals Processing V	Fluid Flow Phenomena In Metals Processing VII	Fluid Flow Phenomena In Metals Processing IX
Room 3	Automotive Alloys I	Automotive Alloys II	Automotive Alloys III	Automotive Alloys IV	Aluminum Alloys for Packaging I	Aluminum Alloys for Packaging II	
Room 4	Milton Blander Intl Symp on "Thermodynamic Predictions & Applications" I	Milton Blander Intl Symp on "Thermodynamic Predictions & Applications" II	Milton Blander Intl Symp on "Thermodynamic Predictions & Applications" III	Milton Blander Intl Symp on "Thermodynamic Predictions & Applications" IV			
Room 5A			Materials Processing Fundamentals I	Materials Processing Fundamentals II	Materials Processing Fundamentals III	Materials Processing Fundamentals IV	Materials Processing Fundamentals V
Room 5B	Review of Extraction Proc, Props & Appls of Reactive Metals I	Review of Extraction Proc, Props & Appls of Reactive Metals II	Reactive Metals - General I	Reactive Metals - General II	Fluid Flow Phenomena In Metals Processing VI	Fluid Flow Phenomena In Metals Processing VIII	
Room 6C	Cast Shop Technology I	Cast Shop Technology II	Cast Shop Technology III	Cast Shop Technology IV	Cast Shop Technology V	Cast Shop Technology VII	Cast Shop Technology IX
Room 6D	Carbon Technology I	Carbon Technology II	Carbon Technology III	Carbon Technology IV	Cast Shop Technology VI	Cast Shop Technology VIII	Cast Shop Technology X
Room 6E		Alumina & Bauxite I	Alumina & Bauxite II	Alumina & Bauxite III	Alumina & Bauxite IV --- Inst. Met. Lect. & R.F. Mehl Medalist	Alumina & Bauxite V	
Room 6F	Aluminum Reduction Technology I	Aluminum Reduction Technology II	Aluminum Reduction Technology III	Aluminum Reduction Technology IV	Aluminum Reduction Technology V	Aluminum Reduction Technology VI	Aluminum Reduction Technology VII
Room 7A		Analytical Technology in the Mineral Industries I	Analytical Technology in the Mineral Industries II	Analytical Technology in the Mineral Industries III	Analytical Technology in the Mineral Industries IV	Fundamentals of Lead & Zinc Extraction & Recycling I	Fundamentals of Lead & Zinc Extraction & Recycling II

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	AM	PM	AM	PM	AM	PM	AM
Room 7B	Surface Engineering: Science & Technology I	Surface Engineering: Science & Technology II	Surface Engineering: Science & Technology III	Surface Engineering: Science & Technology IV	Surface Engineering: Science & Technology V	Surface Engineering: Science & Technology VI	Surface Engineering: Science & Technology VII
Room 8	Intl Symp on Gamma Titanium Aluminides I	Intl Symp on Gamma Titanium Aluminides II ------ ISGTA Posters I (Marriott)	Intll Symp on Gamma Titanium Aluminides III	Intl Symp on Gamma Titanium Aluminides IV ------ ISGTA Posters II (Marriott)	Intl Symp on Gamma Titanium Aluminides V	Intl Symp on Gamma Titanium Aluminides VI	Intl Symp on Gamma Titanium Aluminides VII
Room 9	Light Weight Alloys for Aerospace Applications I	Light Weight Alloys for Aerospace Applications II	Light Weight Alloys for Aerospace Applications III	Light Weight Alloys for Aerospace Applications IV	Long Term Stability of High Temperature Materials I	Long Term Stability of High Temperature Materials II	Long Term Stability of High Temperature Materials III
Room 10	Synthesis of Lightweight Metals I	Synthesis of Lightweight Metals II	Synthesis of Lightweight Metals III	Synthesis of Lightweight Metals IV	Synthesis of Lightweight Metals V	Synthesis of Lightweight Metals VI	Synthesis of Lightweight Metals VII
Room 11A	The Martin E. Glicksman Symposium on Solidification & Crystal Growth I	The Martin E. Glicksman Symposium on Solidification & Crystal Growth II	The Martin E. Glicksman Symposium on Solidification & Crystal Growth III	The Martin E. Glicksman Symposium on Solidification & Crystal Growth IV	The Martin E. Glicksman Symposium on Solidification & Crystal Growth V		
Room 11B	Sheet Metal Forming Technology I	Sheet Metal Forming Technology II	Mini Structures & Components Under Cyclic Load; Fatigue & Internal Frict I	Mini Structures & Components Under Cyclic Load; Fatigue & Internal Frict II	Mini Structures & Components Under Cyclic Load; Fatigue & Internal Frict III	Mini Structures & Components Under Cyclic Load; Fatigue & Internal Frict IV	
Room 12	Gen Abstracts 1: Mechanical Properties I	Gen Abstracts 3: Mechanical Properties II	Gen Abstracts 4: Fatigue, Corrosion & Wear	Gen Abstracts 6: General Metallurgy	Gen Abstracts 7: Issues in Materials Science: Thin Films & Temperature Sensing	Gen Abstracts 8: Melting, Solidification & Microstructure Characterization	
Room 13	Gen Abstracts 2: Ceramic & Refractory Matls & Ceramic/Metal Interfaces	Outcomes Assessments for ABET Criteria 2000	Gen Abstracts 5: Physical Metallurgy: Thermodynamics, Interfaces & Diffusion				
Room 14A	Professional Development	Hume Rothery Symp to Honor M. Hillert; Alloy Effects on Migrating Interfaces I	Hume Rothery Symp to Honor M. Hillert; Alloy Effects on Migrating Interfaces II	Hume Rothery Symp to Honor M. Hillert; Alloy Effects on Migrating Interfaces III	Manufacturing Issues in Rapid Thermal Processing I	Manufacturing Issues in Rapid Thermal Processing II	
Room 14B	Micromechanics& Micromechanisms of Def &Fract I: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract II: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract III: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract IV: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract V: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract VI: Honoring Prof. Ali Argon	Micromechanics& Micromechanisms of Def &Fract VII: Honoring Prof. Ali Argon
Room 15A	Creep Behavior of Advanced Materials for the 21st Century I	Creep Behavior of Advanced Materials for the 21st Century II	Creep Behavior of Advanced Materials for the 21st Century III	Creep Behavior of Advanced Materials for the 21st Century IV	Creep Behavior of Advanced Materials for the 21st Century V	Creep Behavior of Advanced Materials for the 21st Century VI	
Room 15B		11th Intl Symp on Experimental Methods for Microgravity Matls Science I	11th Intl Symp on Experimental Methods for Microgravity Matls Science II	11th Intl Symp on Experimental Methods for Microgravity Matls Science III	11th Intl Symp on Experimental Methods for Microgravity Matls Science IV	11th Intl Symp on Experimental Methods for Microgravity Matls Science V	11th Intl Symp on Experimental Methods for Microgravity Matls Science VI

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	AM	PM	AM	PM	AM	PM	AM
Room 16A	Nondestructive Evaluation (NDE) & Matl. Prop. I - - - - - Tutorial Lunch/Lect.	Nondestructive Evaluation (NDE) and Material Properties II	Nondestructive Evaluation (NDE) & Matl. Prop. III - - - - - Tutorial Lunch/Lect.	Nanostructured Hybrid Materials I	Nanostructured Hybrid Materials II	Nanostructured Hybrid Materials III	
Room 16B	Structural Silicides I	Structural Silicides II	Structural Silicides III		Electrical & Thermal Properties of Materials I	Electrical & Thermal Properties of Materials II	
Room 17A	InterconnectPACK Interconnections for Electronics Packaging I	InterconnectPACK Interconnections for Electronics Packaging II	InterconnectPACK Interconnections for Electronics Packaging III	InterconnectPACK Interconnections for Electronics Packaging IV	InterconnectPACK Interconnections for Electronics Packaging V	InterconnectPACK Interconnections for Electronics Packaging VI	
Room 17B	International Symposium on Advances in Twinning I	International Symposium on Advances in Twinning II	International Symposium on Advances in Twinning III	International Symposium on Advances in Twinning IV	International Symposium on Advances in Twinning V	International Symposium on Advances in Twinning VI	
Room 18	High-Temperature Superconductors: Synthesis, Fabrication & Application I	High-Temperature Superconductors: Synthesis, Fabrication & Application II	High-Temperature Superconductors: Synthesis, Fabrication & Application III	High-Temperature Superconductors: Synthesis, Fabrication & Application IV			
Room 19	High Temperature Coatings I	High Temperature Coatings II	High Temperature Coatings III	High Temperature Coatings IV	High Temperature Coatings V		

Special Event	Time	Room	Location
Monday:			
Tutorial Luncheon Lecture: "Damascus Steels"	12:00 Noon - 1:30 PM	16A	San Diego Convention Center
Hume-Rothery Award Symposium	2:00 PM	14A	San Diego Convention Center
Intl. Symp. on Gamma Titanium Aluminides III - Posters	6:00 - 10:00 PM	Ballroom A & B	Marriott Hotel, North Tower
Tuesday:			
EPD Luncheon	12:00 Noon	Marina Ballroom	Marriott Hotel
Tutorial Luncheon Lecture: "Dynamic Behavior of Materials"	12:00 Noon - 1:30 PM	16A	San Diego Convention Center
EPD Distinguished Lecturer	1:45 PM	Marina Ballroom	Marriott Hotel
Intl. Symp. on Gamma Titanium Aluminides III - Posters	6:00 - 10:00 PM	Ballroom A & B	Marriott Hotel, North Tower
Wednesday:			
Institute of Metals Lecture and Robert F. Mehl Medalist	12:00 Noon	6E	San Diego Convention Center
LMD Luncheon	12:00 Noon	Marina Ballroom	Marriott Hotel

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

Sponsored by: TMS

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
March 1, 1999 Location: Convention Center

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.

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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.

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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.

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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%.

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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.

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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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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.

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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.

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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

2:00 PM

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

Monday PM — Room: 15A
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.

3:50 PM BREAK

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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.

5:00 PM

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

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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

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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.

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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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3:50 PM INVITED PAPER

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.

4:30 PM INVITED PAPER

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.

4:50 PM INVITED PAPER

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.

2:20 PM

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.

2:40 PM

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.

5:00 PM

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.

5:20 PM

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.

3:30 PM BREAK

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.

3:00 PM INVITED PAPER

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.

3:30 PM

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.

3:55 PM BREAK

4:05 PM INVITED PAPER

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.

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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.

3:45 PM BREAK

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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.

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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.

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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.

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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.

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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.

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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.

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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.

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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

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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.

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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.

2:30 PM INVITED PAPER

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.

4:50 PM
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.

5:10 PM
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.

5:00 PM

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.

4:00 PM

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

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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

3:45 PM BREAK

4:00 PM
NIOBIUM: *S. Yuan*[1]; [1]Cabot Corporation, Performance Materials, P.O. Box 1609, County Line Rd., Boyertown, PA 19512 USA

4:30 PM
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.

2:20 PM
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.

2:40 PM
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.

3:00 PM
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

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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.

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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

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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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4:00 PM INVITED PAPER
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.

4:50 PM
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.

5:05 PM

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.

5:20 PM

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.

5:35 PM

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.

2:20 PM INVITED PAPER

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.

2:40 PM

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.

3:00 PM BREAK

3:15 PM

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.

3:35 PM

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.

3:55 PM

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.

4:15 PM

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

2:15 PM INVITED PAPER

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.

2:45 PM INVITED PAPER

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.

3:15 PM

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.

3:35 PM BREAK

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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.

4:25 PM

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.

4:45 PM INVITED PAPER

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.

5:25 PM

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.

TUESDAY AM

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

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

Tuesday AM Room: 15B
March 2, 1999 Location: Convention Center

Session Chair: Carlo Patuelli, Instituto Nazionale ci Fisica per la Materia, Alma Mater Studiorum, Bolonga 40127 Italy

8:30 AM

WETTING BEHAVIOR OF BSO MELT ON PLATINUM: *Yu Zheng*[1]; A. F. Witt[1]; [1]Massachusetts Institute of Technology, 77 Massachusetts Ave., Cambridge, MA M.I.T. 13-4154 USA

Single crystal of Bi12SiO20 (BSO) is conventionally pulled from its melt by the Czochralski method [1-2]. To better understand the growth process and the mechanism of formation of defects, a Vertical Bridgman growth of BSO crystals under micro-gravity conditions has been proposed [3]. The crucible has to be made of Platinum, the only known material that is chemically stable when in contact with BSO melt. The interaction between BSO and Pt is extremely important when concern the thermal stresses caused by their different thermal expansion properties. In this work, the wetting behavior of BSO melt on platinum under an ambient with various partial pressure of oxygen is studied by the sessile drop method. The wetting angles as well as the surface tension of BSO melt are calculated by analyzing the shape of the sessile drops [4-5]. It is found that the BSO melt partially wets Pt at low pressure of oxygen ($\sim 10^{-4}$ atm) with an equilibrium contact angle around 45ϒ. The contact angle decreases as the pressure of oxygen goes up and approaches to zero when it is above 10-2 atm. The surface tension of BSO is about 200 mJ/m^2, which doesn't change with the pressure of oxygen. Finally, a design of the crucible for VB growth of BSO under micro-gravity conditions is given based on this wetting behavior. References: [1] O.F. Hill and J.C. Brice. The composition of crystal of bismuth silicon oxide. Journal of Materials Sciences, 9:1252, 1974. [2] C. Lin. Crystal growth and characterization of BSO. Doctoral Dissertation, Massachusetts Institute of Technology (1994) [3] A.F. Witt. Proposal on a Vertical Bridgman growth of BSO under Micro-gravity conditions [4] S. Harland and R. Hartley. Axisymmetric Fluid-Liquid Interfaces (Elsevier, Amsterdam, 1976) [5] S. Lahooti, O.I. Del Rio, A.W. Neumann, P.Cheng. Axisymmetric Drop Shape Analysis (ADSA). Applied Surface Thermodynamics (Marcel Dekker, Inc., New York, 1996), pp. 441

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DIRECTIONAL SOLIDIFICATION OF IMMISCIBLE ALLOYS UNDER MICROGRAVITY CONDITIONS: *J. B. Andrews*[1]; L. J. Hayes[1]; Y. Arikawa[1]; S. R. Coriell[2]; [1]University of Alabama at Birmingham, Mats. and Mech. Eng., Birmingham, AL 35294 USA; [2]NationalInstitute of Standards and Technology, Metall. Div., Gaithersburg, MD 20899 USA

Findings obtained from the Coupled Growth in Hypermonotectics experiment, which flew aboard the Life and Microgravity Spacelab mission, will be presented. During the mission, three immiscible Al-In samples were directionally solidified using the Advanced Gradient Heating Facility. These samples consisted of one 17.3wt% In monotectic composition sample and two hypermonotectic samples containing 18.5 and 19.7wt% In. All samples were initially heated to a temperature of 1100ϒ for 6 hours for homogenization and then directionally solidified at a rate of 1mm/s. X-ray analysis revealed that voids were present in some of these samples that could lead to local variations in thermal gradient and solidification rate. The microstructural variations in these samples will be reported and compared with the anticipated results from this experiment.

9:10 AM

STRIATION INDUCED BY UNSTEADY BRIDGMAN DIRECTIONAL SOLIDIFICATION: *R. Guerin*[1]; P. Haldenwang[2]; [1]Universite d'Aix-Marseille II, Laboratoire Matop, URA/CNRS 1530, Faculte des Sciences et Techniques de Saint-Jerome, Case 151, Marseille Cedex 20 F-13397 France; [2]Universite de Provence, IRPHE/UMR-CNRS 6594 IMT/Lajetee, Technopole de Chateau Gombert 38, Avenue Joliot-Curie, Marsceille Cedex 20 F-13451 France

Time-dependent behaviours induced by the solutal convection appearing above a solidification front are numerically investigated. The system under study corresponds to the upward Bridgman configuration for the directional solidification of the Pb-30%Ti alloy. The purpose of this work is to characterise the striations induced in the crystal when the flow becomes unsteady. We determine the onset threshold of unsteadiness as a function of confinement, and investigate the time behaviour of a given pattern versus the Rayleigh number for two different aspect ratios. More precisely, we estimate the magnitude of the time fluctuations in solute composition that the crystal incorporates. The period of the striations is also discussed. We indicate the flow conditions under which both quantities are appropriate for an experimental detection: a subcritical Hopf bifurcation is found more favourable for experimental observation of striation than a supercritical one. Unsteadiness in 3-D solutal convection is also considered.

9:30 AM

THE INFLUENCE OF G-JITTER ON PARTICLE INCORPORATION BY AN ADVANCING SOLID/LIQUID INTERFACE: *Carolyn L. Russell*[1]; Reginald W. Smith[1]; [1]Queen's University, Dept. of Mats. and Metall. Eng., Kingston K7L 3N6 Canada

It has been observed in transparent analogues of freezing metals, that particles existing in the melt may be pushed ahead of a macro-planar interface or occluded (captured) depending on whether the rate at which the solid/liquid interface is advancing is smaller or greater than some critical value (Vc). Various attempts have been made to provide a physical understanding of this. However, when experiment values for Vc are obtained they often differ widely from those predicted by the various models. In an attempt to simplify the experimental observations, a series of aluminum-based liquid metal matrix composites have been processed on the MIR Space Station. The samples were frozen unidirectionally using the gradient-freeze technique in the QUELD II furnace facility. This was attached to the Canadian Microgravity Isolation Mount (MIM). The MIM was used in three modes "Latched", "Isolating" and "Forcing". This permitted the influence of the fluid transport resulting from g-jitter, both "natural" and "forced", on the particle incorporation process to be determined. In addition, these experiments attempted to assess the influence of particle shape, thermal conductivity and volume fraction on Vc. The experiments and the results obtained will be presented.

9:50 AM BREAK

10:10 AM

ROTATING MOLTEN METALLIC DROPS AND THEIR APPLICATION TO SURFACE TENSION MEASUREMENT: *Won-Kyu Rhim*[1];

Takehiko Ishikawa[2]; [1]California Institute of Technology, Jet Propulsion Laboratory, 4800 Oak Grove Dr., Pasadena, CA 91109 USA; [2]Space Utilization Research Center, NASDA, (On leave from the Nationals Space Development Agency of Japan), 2-1-1 Sengen, Tsukuba, Ibaraki 305 Japan

Shapes and stability of rotating molten metal drops were experimentally investigated, and the feasibility of an alternative method for surface tension measurement was examined. Molten aluminum and tin drops approximately 3 mm in diameter were suspended in a high vacuum in a high temperature electrostatic levitator, and they were systematically rotated by applying a torque which was generated by a rotating magnetic field along the vertical axis. As the drop angular momentum was gradually (or step by step) increased from the static state, the drop shape evolved first along the axi-symmetric branch until the bifurcation point was reached at which transformation from axi-symmetric to triaxial shape took place With the assumption of 'effective surface tension' which includes the effect of reduced surface tension due to the surface charge, the results agreed quantitatively well with the Brown and Scriven's prediction. The normalized angular velocity at the bifurcation point agreed with the predicted value of 0.559 within 2%. Finally, an alternative surface tension measurement technique for high viscosity liquids, where the sample oscillation technique cannot be applied, will be presented.

10:30 AM

FINITE ELEMENT ANALYSES OF DROPLET DEFORMATION AND OSCILLATIONS IN MICROGRAVITY: Suping Song[1]; *Ben Q. Li*[1]; [1]Washington State University, School of Mech. and Mats. Eng., Pullman, WA 99163 USA

Finite element models are developed to represent the phenomena of surface deformation, oscillation, fluid flow and heat transfer associated with droplets positioned by either electrostatic or magnetic fields in microgravity. Numerical results are presented and compared with analytical or asymptotic solutions and/or experimental measurements that are available. The implications of results to the space measurements of thermophysical properties such as viscosity and surface tension are also discussed.

10:50 AM

DIFFUSION-LIMITED AND INTERFACE KINETIC-CONTROLLED CRYSTAL GROWTH IN SUCCINONITRILE AND PIVALIC ACID (PVA): *Afina Lupulescu*[1]; Martin E. Glicksman[1]; Mathew B. Koss[1]; Jeffrey C. Lacombe[1]; Laura T. Tennenhouse[1]; Julie E. Frei[1]; Douglas Corrigan[1]; [1]Rensselaer Polytechnic Institute, Dept. of Mats. Sci. and Eng., Troy, NY 12180 USA

Dendritic growth is generally considered to be kinetically controlled by a diffusion-limited process. The growth of dendrites in pure melts is controlled by the transport of latent heat from the moving boundary as it advances in supercooled melt. However, determining the interfacial speed at which attachment kinetics dominate the growth process and diffusion becomes relatively important, and remains uncertain except in broad theoretical terms. We focus here on experimental evidences of diffusional limited transport, and interfacial kinetic effects. The data will be presented for succinonitrile (BCC), and pivalic acid (FCC). The Isothermal Dendritic Growth Experiment (IDGE) is a basic science experiment designed to provide terrestrial and microgravity data which measure the kinetics, morphology, and dynamics of dendritic solidification under diffusion control. This experiment is a NASA-developed flight experiment and has already flown three times (USMP-2 and USMP-3 using SCN, and USMP-4 using PVA). USMP-2 and USMP-3 flight data, when compared to terrestrial dendritic growth data demonstrate that: 1) in the supercooling range from 0.47K to 2.1K microgravity data are free of growth chamber boundary and convective effects; b) these data may also be used to examine diffusion-limited, dendritic growth theories. Previous ground based experiments showed that the SCN dendritic growth is controlled by kinetic effects only at high supercoolings (higher than 5K). Trivedi & Mason (1991) claimed to show large kinetic effects in a PVA sample growing with a planar interface under steady-state conditions. The liquid-solid interface temperature, in their experiment, deviated substantially from the PVA melting temperature. The authors considered Ò highly nonlinear interface kinetic effects present during the solidification of PVAÓ. The IDGE results contrast with the results of Trivedi & Mason. USMP-4 microgravity data indicate that PVA dendritic growth is limited by diffusional transport. The effects of interface attachment kinetics are insignificant, even for the highest velocity observed (875 microns/sec).

11:10 AM

DIFFUSION EFFECTS ON LIQUID PHASE SINTERED Co-Cu SAMPLES IN MICROGRAVITY: *Y. He*[1]; J. Naser[1]; J. Chiang[1]; J. E. Smith[1]; [1]University of Alabama in Huntsville, Chemical and Mats. Eng., Huntsville, AL 35899 USA

This paper discusses the diffusion controlled grain growth of Co-Cu samples processed under microgravity. Twelve powder compact samples with solid volume fraction ranging from 50% to 70% were processed at 1473K during liquid phase sintering (LPS) experiments aboard sounding rockets and Space Shuttle missions. Processing time ranged from 2.5 to 66 minutes. The diffusional layer associated with processing time was observed and the experimental results on the grain size distribution and grain-coarsening rate are presented. The concentration distribution results based on SEM analysis are also presented. The diffusional effects on dihedral angle and particle sphericity are also discussed. The microstructural characterization revealed the agglomeration and coalescence occurred during liquid phase sintering of Co-Cu samples. The modified grain growth model that incorporates coalescence and particle sphericity (LSEM) appears to describe the observed grain growth behaviors far better than the LSW model.

11:30 AM

QUANTITATIVE COMPUTER TOMOGRAPHY FOR DETERMINING COMPOSITION OF MICROGRAVITY AND GROUND BASED SOLID SOLUTIONS: *D. C. Gillies*[1]; H. P. Engel[2]; [1]NASA/Marshall Space Flight Center, Huntsville, AL 35812; [2]*Wyle Laboratories, Kennedy Space Center, Cape Canaveral, FL 32899 USA

Advances in x-ray Computer Tomography (CT) have been led by the medical profession, and by evaluation of industrial products, particularly castings. Porosity can readily be determined as a function of the density of a material, and CT is thus an industrially important NDE tool. Providing high purity, 100% dense standards of pure elements and compounds can be fabricated, the composition of solid solution alloys can be determined by measuring the CT number, which is a function of the absorption of the sample. Average densities across slices 1 mm thick can generally be determined to better than 1 percent. With present technology this spatial sensitivity is less than ideal, but important benefits can nevertheless be obtained by using CT, particularly single crystals, prior to making any destructive assault upon the sample. The sample can in fact be examined prior to removal from the mold within which it has been grown and, in the cases of microgravity flight samples, before removal from the cartridge assembly. This greatly assists the researcher in the characterization of the products, particularly as a guide to cutting and sampling. Examples of work with germanium-silicon alloys and mercury cadmium telluride taken with a radioactive cobalt source will be demonstrated.

ALUMINA AND BAUXITE: Spent Bauxite Use and Processing

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

Tuesday AM Room: 6E
March 2, 1999 Location: Convention Center

Session Chair: Kenneth W. Ryan, ALCOA, Point Comfort Works, Point Comfort, TX USA

8:30 AM
DRY STACKING OF BAUXITE RESIDUE: *Fred S. Williams*[1]; Dana T. Smith[1]; [1]ALCOA, Point Comfort, TX USA

Dry stacking of bauxite residue has been shown to be an economical method for disposal compared to the wet lake disposal methods previously used in the alumina industry. A number of factors affect the slope which a bauxite residue will form in a stack deposit. Relationships useful for design and control of the slope of bauxite residue stacks will be discussed in this paper.

9:00 AM
TRACE ELEMENT PARTITIONING AND BIOAVAILABILITY IN RED MUD SYNTHETIC WETLAND SEDIMENT: *Gerald L. Goldstein*[1]; Robert S. Reimers[1]; David B. Kirkpatrick[2]; [1]Tulane University, Environmental Health Sciences, 1501 Canal St., New Orleans, LA 70112 USA; [2]Kaiser Aluminum and Chemical Corporation, Gramercy, LA USA

Over twenty-five square miles of Louisiana wetlands are lost per year due to coastal erosion and sediment starvation. A sediment source is needed for replenishment of the marshes, and dredged sediment is often polluted and unsuitable for placement in the wetlands. Consequently, it has been proposed that spent bauxite be used as an alternative to natural sediment. However, prior to use, it is important to evaluate the potential environmental impact of red mud in the wetlands. Specifically, the fate of red mud trace metals and the potential toxicity of red mud amended sediments to aquatic organisms need definition. Tulane University, in conjunction with Kaiser Aluminum & Chemical Corp., investigated the binding, partitioning, and bioavailability of key trace metals in synthetic blends comprised of red mud and natural sediment. Heavy metal behavior in red mud/sediment blends was studied using extraction, adsorption and bioaccumulation experiments. The results form these experiments are discussed herein.

9:30 AM
RED MUD PRODUCT DEVELOPMENT: *Seymour O. Brown*[1]; David B. Kirkpatrick[1]; [1]Kaiser Aluminum & Chemical Corporation, P.O. Box 3370, Gramercy, LA 70052 USA

Kaiser Aluminum & Chemical Corporation has impounded spent bauxite behind levees for over 20 years. In 1994, Kaiser embarked on a project to de-water the mud lakes at its Gramercy Louisiana Plant and to develop beneficial uses for the reclaimed lands and processed red mud. The work was presented at the 1996 TMS Annual Meeting. Kaiser is continuing its research into red mud uses. Kaiser has developed methods to use this material to make levees. Kaiser and Colorado School of Mines have researched the extraction of other metals from the spent bauxite. Kaiser and Tulane University have researched the making of synthetic soils as well as the use of red mud as a sewerage disinfectant. The status of these projects will be discussed in this presentation.

10:00 AM BREAK

10:30 AM
WASTE FREE ALUMINA PRODUCTION FROM NON-BAUXITIC RAW MATERIALS: *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 aggregates and other materials is such a process. This paper covers the potential of utilzing alkaline alumosilicate and alunite raw material and the economics of these processes. Some flowsheets of alunite and alumosilicate processing and the techno-economic parameters of complete treatment are shown.

11:00 AM
UTILIZING ALUMINA PLANT WATER DISCHARGE STREAMS: *Gan GuoYao*[1]; Wang Longzhang[1]; [1]Pingguo Alumina Plant, Pingguo, Guangxi China

A new technology of making use of the Contaminated water produced in the Pingguo Alumina plant was developed. After treating the water in a processing center, the contaminated water is used to cool precipitators and to clean the boiler smoke generated by the combustion of coal. By developing this technology, the productivity of the plant was increased by 5%, and the rates of removing sulfur dioxide and dust from the boiler smoke by 35%. This new technology gives good results, is economical and protects the environment.

ALUMINUM REDUCTION TECHNOLOGY: Environmental

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

Tuesday AM Room: 6F
March 2, 1999 Location: Convention Center

Session Chair: Reidar Huglen, Hydro Aluminium Karmoy Metallverk, Havik N-4265 Norway

8:30 AM
THE ELECTRODE KINETICS OF PERFLUOROCARBON (PFC) GENERATION: *D. R. Sadoway*[1]; *H. Zhu*[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Room 8-109, Cambridge, MA 02139-4307 USA

The generation of CF_4 and C_2F_6 is being studied in a laboratory-scale aluminum reduction cell. During electrolysis in well behaved cells and in cells on anode effect, anode gases have been analyzed by gas chromatography on-line. Electroanalytical techniques such as ac voltammetry and electrochemical impedance spectroscopy are being used to determine the values of the kinetic parameters associated with the electrochemical reactions occurring on the anode as a function of various operating conditions, e.g., anode composition, bath chemistry, and temperature. The research is sponsored jointly by the Aluminum Association and the U.S. Environmental Protection Agency.

9:05 AM
SPECTRUM ANALYSIS OF THE BUBBLING ACOUSTIC SIGNALS THROUGH CARBON ANODES: *Jilai Xue*[1]; Harald A. Øye[2]; [1]SINTEF Applied Chemistry, Trondheim N-7034 Norway; [2]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway

The information on anode bubbling is important for cell operation and new cell design. However, it is extremely difficult to have direct observation on the bubbling process during cell operation due to the high temperature and corrosive conditions. This paper will present an alternative approach to investigate the in situ bubbling behavior by detecting the sound signals from the anodes. The correlation between the sound signals and the anode bubbling was observed in both water modeling and industrial tests. A portable computer system with on-line data acquisition and spectrum analysis functions has been developed for use in industrial environment. The signal patterns on normal anodes in a cell were obtained as reference. The variation in signal pattern with anode spike was investigated.

9:35 AM
POT GAS FUME AS A SOURCE OF HF EMISSION FROM ALUMINIUM SMELTERS - LABORATORY AND FIELD INVESTIGATIONS: *A. B. Heiberg*[1]; G. Wedde[1]; [1]ABB Miljø AS, P.O. Box 6260 Etterstad, Oslo N-0603 Norway

HF measurements carried out in treated pot gas from aluminium smelters equipped with dry scrubbing occasionally show higher emission levels than expected from current HF penetration-saturation relationships. The excess HF emission might be caused by a chemical reaction between particulate fluoride compounds (fume particles) originating in the electrolytic bath and moisture present in the pot gas. To test this hypothesis samples of dust collected at an aluminium smelter were ex-

posed to hot, humid air in the laboratory. The HF concentration was measured in the exit gas at frequent intervals over a time period of several hours. Both "pure" fume, and mixtures of fume and alumina identical to the dust that is constantly added to the filter bags used in the scrubbers, were investigated. Samples of exposed filter bags were also tested. With gas temperature and humidity kept at the relevant levels a noticeable emission of HF is observed both from dust and filter, confirming that particulate fluorides may be a significant source of HF in the clean gas. Mathematical models quantifying the relation between HF concentration and temperature, humidity and F-content in the dust were developed and applied to data collected at a smelter in the Middle East. The plant simulations indicate that reactive fume may account for about one third of total dry scrubber HF emissions. Both the fume-related and the total emission is significantly affected by temperature. Means to minimize the contribution from the fume are suggested.

10:00 AM BREAK

10:30 AM
VENTILATION OF POTROOMS IN ALUMINIUM PRODUCTION: *N. J. Holt*[1]; N. M. Anderson[2]; M. Karlsen[1]; T. Foosnaes[1]; [1]Hydro Aluminium AS, Technology Centre Ardal, P.O. Box 303, Ovre Ardal N-5870 Norway; [2]Hydro Research Centre, P.O. Box 2560, Prosgrunn 3901 Norway

Potrooms for the production of aluminium are preferably naturally ventilated. Air flow rates may vary from 8-24 Nm^3/s/pot, depending on cell technology, building layout and location. As focus on environmental issues increases, it becomes also more important to control the ventilation so that the best possible working atmosphere may be achieved. This paper concentrates on the main parameters which must be kept under control, and which may be used to control the ventilation, e.g. the design of the cellar, the use of wind shields and the area and position of the floor gratings. We also present measurements and numerical simulations to illustrate how existing potroom buildings may be converted from mechanical to natural ventilation. It is further important to realize that climatic conditions should be considered and that these may vary significantly among places where aluminium smelters are located. Locations with tropical climates with average temperatures well above 30ϒC and often high air humidity, thus represent a different challenge with respect to ventilation when compared to a smelter located in a temperate climate.

11:00 AM
TREATMENT AND REUSE OF SPENT POT LINING, AN INDUSTRIAL APPLICATION IN A CEMENT KILN: *P. B. Personnet*[1]; [1]Aluminium Pechiney, LRF BP114, 73303 Saint Jean De Maurienne, Cedex France

The treatment of Spent Pot Lining (SPL) has been the occasion of numerous studies in the world. Several processes have reached the commercial stage in USA, Australia and Europe. Most of these processes generate waste that need to be disposed of. Another approach is to make total use of the SPL constituents, with no waste disposal and with no air emission. Among several processes allowing reuse of SPL, one is highlighted here: the addition of second cut (refractory) SPL to quarry materials in the raw feed of a Cement Kiln. Under close supervision of the French Environment Protection Authority, ALUMINIUM PECHINEY has developed a partnership with CIMENTS D'ORIGNY including an R&D programme to demonstrate the capability of a Cement Kiln to accept SPL as a raw material despite the presence of sodium and fluoride. Since 1997, SPL is now recycled industrially in a Cement Plant of Northern France.

ANALYTICAL TECHNOLOGY IN THE MINERAL INDUSTRIES: Instrumental Analytical Methods in Mineral Processing

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

Tuesday AM Room: 7A
March 2, 1999 Location: Convention Center

Session Chairs: Emil B Milosavljevic, Lakewood, CO 80227 USA; Charles H. Bucknam, Newmont Technical Facility, Newmont Metallurgical Services, Englewood, CO 80112 USA

8:30 AM INTRODUCTION TO SESSION

8:35 AM INVITED PAPER
EVALUATION OF ICP-MS AND GF-AAS METHODS FOR ANTIMONY DETERMINATION IN GROUNDWATERS: *Rahul S. Bhaduri*[1]; Lucinda Tear[2]; [1]Newmont Gold Company, Twin Creeks Mine, P.O. Drawer 69, Golconda, NV 89414 USA; [2]Parametrix, Inc. USA

The EPA promulgation of the Safe Drinking Water Act (SDWA) requires that the Maximum Contaminant Level (MCL) for antimony in ground waters not exceed 6 ppb. The selected analytical method(s) should reliably quantify antimony near the MCL. This paper presents an interpretation of analytical results from extensive bench and pilot scale investigations geared towards removing antimony to below the MCL in treated ground water. Filtered head and effluent sample splits, from daily runs, were sent to five State certified laboratories for a total period of 25 days; two facilities employed Graphite Furnace-Atomic Absorption Spectrometry (GF-AAS), two others used Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and a fifth adopted both methods. Scatter plots, for individual laboratories, were used to visually assess the type of relationships that existed between residual antimony and independent process variables. As the relationships were linear, the Pearson correlation coefficient was used to estimate the strength and significance of the dependency. The correlation between process variables and residual antimony concentrations were more significant, and varied less between laboratories, when the ICP-MS method was employed. The findings indicate that ICP-MS is a more precise analytical instrument for determining low levels of antimony in treated groundwater.

9:00 AM INVITED PAPER
APPLICATION OF ICP-MS IN SOLVING PRACTICAL ANALYTICAL PROBLEMS IN MINE MATERIALS: *Dana Mills*[1]; Hugh deSouza[1]; Paul Burgener[1]; [1]X-Ray Assay Laboratories, 1885 Leslie St., Ontario M3B 3J4 Canada

ICP-MS has moved from a laboratory curiosity to a functional analytical tool. This paper will discuss how ICP-MS has been used to solve difficult analytical procedures in analyzing mine materials. Comparisons between analytical techniques applied to the analysis of gold and the platinum group metals will illustrate the advantages and problems of ICPMS when applied to trace levels of some metals in the presence of ore grade metals. Interferences and matrix effects are compared to ICP, AA and Neutron Activation. The necessity of selecting chemical extractions which are compatible with ICPMS are also discussed. The ICPMS techniques explained in the examples on Au and PGE's have validity in analytical problems encountered in testing of trace metals in the presence of major elements found in mine materials. Exploration

and environmental testing are other areas which require the same analytical approach to quantify trace levels in the presence of major interfering elements normally associated with mining environments.

9:25 AM INVITED PAPER

ANALYTICAL TECHNIQUES USED FOR THE RECYCLING OF LEAD FROM FIRE ASSAY WASTES: *Carl C. Nesbitt*[1]; Sui Xue[2]; [1]Michigan Technological University, Dept. of Metall. and Mats. Eng., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Eichrom Industries, Inc., 8205 S. Cass Ave., Suite 107, Darien, IL 60561 USA

A project has been completed which investigated the efficacy of recycling lead or lead monoxide from various fire assay wastes, including crucibles, cupels and slag. The scope of the project was to determine if hydrometallurgical treatment of the materials could be optimized so that direct separation and recovery of lead salts was economically feasible. A process was developed from the study which could use either acetic acid or nitric acid as a leachant, followed by several precipitation processes which could directly recover litharge (PbO) or other lead salts, such as lead carbonate or lead sulfate. Analytical equipment such as the atomic absorption spectrophotometer and x-ray diffractometer supplied much of the information required. However, some novel analytical approaches using these instruments had to be developed to help in the determination of leaching kinetics and process quality control. These techniques will be presented.

9:50 AM INVITED PAPER

CHARACTERIZATION AND STABILITY OF ARSENITE AND ARSENATE IN THE IRON-ARSENIC AND LANTHANUM-ARSENIC SYSTEMS: *M. Misra*[1]; A. Rawat[1]; J. Nanor[1]; [1]University of Nevada, Reno, Chem. and Metall. Eng., Mackay School of Mines, MS 170, Reno, NV 89557 USA

The precipitation vs adsorption of arsenite and arsenate species onto iron and lanthanum compounds has been discussed. Several analytical and characterization techniques such as XRD, FTIR, Zeta Potential and AFM have been used to establish the nature of bonding. An advanced CHEMDRAW computer program has been used to estimate the bond strength and nature of adsorption.

10:15 AM BREAK

10:45 AM INVITED PAPER

COMBINING FTIR SPECTROSCOPY AND MULTIVARIATE CALIBRATION TO LOCATE MINERABLE ORE DEPOSITS: *Sharon L. Eyer*[1]; [1]Alcoa of Australia, Ltd., Research and Development Dept., P.O. Box 161, Kwinana 6167 Western Australia

FTIR spectroscopy can be a used to estimate the quality of an ore body based on exploration samples. Furthermore it can highlight regions of ore that are relatively unusual. Unusual ore can be investigated for potential impacts on refinery processes well in advance of mining. The technology has been successfully implemented at an alumina refinery in Kwinana, Australia, for assessment of ore attributes such as available alumina, extractable organic carbon and total iron. Classical methods of analyses for these properties involve laboratory-scale digestion of alumina ore (bauxite) or XRF analyses. Multivariate calibration is used to generate robust models that are able to estimate classical data from FTIR spectral data. The latter are collected from powdered bauxite at a throughput of less than a minute per sample. The technique is illustrated conceptually and an example application for bauxite is presented. Challenges and difficulties are discussed. The potential to expand the technique to process control is introduced.

11:10 AM INVITED PAPER

IN-SITU FT-IR/IRS AND MLRS EXAMINATION OF OLEATE ADSORPTION AT FLUORITE AND CALCITE SURFACES: *Courtney A. Young*[1]; J. D. Miller[2]; [1]Montana Tech of the University of Montana, Dept. of Metall. Eng., Butte, MT 59701 USA; [2]University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA

Adsorption isotherms previously determined by in-situ Fourier transform infrared/internal reflection spectroscopy (FT-IR/IRS) for oleate adsorbed at calcite and fluorite surfaces near pH 9 and at various temperatures are compared. Results indicate that, at both mineral surfaces, chemisorption occurs at equilibrium concentrations below approximately $1x10^{-5}$ M oleate while calcium dioleate surface precipitation occurs at higher equilibrium oleate concentrations. The extent of chemisorption at the calcite surface is 3 to 5 times less than that at the fluorite surface while the extent of calcium dioleate surface precipitation on calcite is nearly a magnitude larger than that on fluorite. However, for both minerals, surface precipitation decreases with increasing chemisorption. Contact angle measurements and flotation recoveries show that the hydrophobicity of fluorite to be greater than calcite at each of the temperatures and oleate concentrations studied. Significant differences are noted only in the chemisorption region. Unlike calcite, the hydrophobicity of fluorite was sensitive to the presence of oxygen in the system. Multichannel Laser Raman Spectroscopy (MLRS) showed this behavior was caused by the double-bond reactivity of adjacent chemisorbed oleate molecules and subsequent formation of an epoxide/polyether complex at the fluorite surface. Such a reaction product could not be detected at the calcite surface. These differences in adsorption density, hydrophobicity, and polymerization behavior are discussed in terms of the intrinsic surface properties of the minerals.

11:35 AM

CAN WE CLOSE THE "CYANIDE CYCLE"?: *Emil B. Milosavljevic*[1]; Ljiljana Solujic[2]; 8143 W. Eastman Place, #14-201, Lakewood, CO 80227 USA; [2]University of Nevada, Reno, Dept. of Chem., MS216, Reno, NV 89557 USA

Cyanide is exceptionally reactive, and in typical gold/silver processing liquors, cyanide may be transformed into various species and forms depending on the mineralogy of the ore and chemical make-up of the process water. The exact speciation and relative concentrations depend on the Eh and pH values, as well as the free CN^- concentration in the system. Very good cyanide balances can be achieved by the judicious selection of the analytical techniques used. The choices an analyst has to make (which analytical methods to use and which ones to avoid) in order to gain better understanding of cyanide speciation and balances in a particular system under investigation will be discussed. Examples of cyanide balances obtained for the laboratory scale tests (agitated leach and column tests) will be presented and discussed.

AUTOMOTIVE ALLOYS III: Session III — Developmental Studies

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

Tuesday AM Room: 3
March 2, 1999 Location: Convention Center

Session Chairs: Subodh K. Das, ARCO Aluminum, Inc., Louisville, KY 40232 USA; J. D. Bryant, Reynolds Metal Company, Chester, VA 23836-3122 USA

8:30 AM

PREDICTION OF YIELD STRENGTH OF THE PAINTED ALUMINUM AA6111 PANEL: *A. K. Gupta*[1]; [1]Alcan International, Kingston Research and Development Center, P.O. Box 8400, Kingston, Ontario K7L 5L9 Canada

The knowledge of the strength of the final part at the design stage is an important parameter in determining the optimum sheet gauge for maximum weight savings without sacrificing the desired dent resistance. The AA6111 sheet materials is relatively soft in the forming temper and becomes stronger from combination of work and age hardening during forming and paint cure operations, respectively. Presently, the strength of the painted part is determined from over-simplified experiments simulating the forming and paint cure processes. In this paper, the hardening model of AA6111 alloy is used to predict the strength following multiple step ageing, both with and without prestrains. The

approach developed in the paper is used to predict the strengths that are obtained from the simulation experiments and comparisons are made with the strength and dent data measured from different locations of an actual painted part.

8:50 AM

OPTIMIZED 6XXX ALUMINUM ALLOY SHEET FOR AUTOBODY OUTER PANELS: *Ravi A. Shahani*[1]; Dominique Daniel[1]; [1]Pechiney, Centre de Recherches de Voreppe, Centr'Alp, BP 27, 38340 Voreppe, Isere France

AA6016 is the aluminum alloy typically specified in Europe for autobody outer panels. Compared with the copper-rich alloys 6111, 6009 and 6010 often used in the US, 6016 shows better formability in the T4 condition but lower strength after the final bake-hardening treatment. As a result of the current trend towards lower paint-bake curing temperatures, a higher strength material than standard 6016 will be preferable for some applications in order to maximize the weight reduction offered by aluminum body panels while maintaining equivalent dent resistance to the steel alternative. This paper describes Pechiney's development programme for an alloy giving higher strength after bake-hardening than 6016, but with improved formability in stamping and hem flanging operations compared with current high strength aluminum automotive sheet alloys.

9:10 AM

MICROCHEMISTRY AND MICROSTRUCTURAL ASPECTS LEADING TO STRESS CORROSION CRACKING IN AA5083: *John S. Vetrano*[1]; M. J. Danielson[1]; D. R. Baer[1]; R. H. Jones[1]; [1]Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352 USA

The addition of Mg to Al increases its formability and strength, but at levels greater than about 3.5 wt.% it can increase the susceptibility to stress corrosion cracking (SCC) in salt water when exposed to moderate temperatures for long times. This precludes the use of high-Mg aluminum alloys for automotive use in load-bearing components. Careful measurements of grain boundary structure and composition in a commercial Al-Mg alloy (5083) have been carried out as a function of annealing time at 175ŸC using high-spatial resolution analytical electron microscopy in conjunction with Auger electron spectroscopy (AES). These results were compared to U-bend specimens heat-treated under the same conditions and exposed to an alternate immersion test in a 3.5% NaCl solution. It was found that the precipitation of the b-phase (Al3Mg2) was a necessary, but not sufficient, pre-requisite for SCC susceptibility. It was noted that specimens that were susceptible had a pronounced depletion of Mg at most grain boundaries between the b-phase particles. Pre-notched compact tension samples were then tested in a 3.5% NaCl solution under increasing loads until a crack was propagated. These measurements confirmed the susceptibility of the different conditions as found in the U-bend tests. The impact of these results on the understanding of the SCC mechanisms in Al-Mg alloys will be discussed and possible mitigation methods will be proposed.

9:30 AM

HOT DUCTILITY OF 6XXX ALUMINUM ALLOY FOR AUTOMOTIVE BODY SHEET: *Tsutomu Itou*[1]; Masayuki Ishikawa[1]; Masahisa Otsuka[1]; Makoto Saga[2]; Masao Kikuchi[2]; [1]Shibaura Institute of Technology, Dept. of Mats. Sci. and Eng., 3-9-14, Shibaura, Minato-Ku, Tokyo 1088548 Japan; [2]Nippon Steel Corporation, Shintomi-cho, 20-1, Futtsu, Chiba 2930011 Japan

Recently 6XXX aluminum alloys have begun to be used for both automotive body sheet panel and space frame in order for weight saving. However, their application is limited to sports cars or high-glade cars. This is partially caused by the lack of plastic workability in these alloys, though both specific stiffness and strength are sufficiently high. In particular, maintaining the hot workability seems to be essential for the substitution of steel sheet with aluminum one. In this paper, the high temperature characteristics of two kinds of 6XXX alloy rolled sheets in T4 condition have been investigated with special reference to the applicability of superplastic forming. Constant cross head speed tensile tests are conducted at various temperature and strain rate ranging, respectively, from 623 to 803K and from 3x10-5 to 1x10-1s-1. Both at 773K and at 803K the total elongation vs. strain rate diagram has a minimum of 160% near the strain rate about 1x10-3s-1. It is to be noted that the typical fracture mode changes from intergranular type in lower strain rate range to transgranular one in higher strain rate range. Total elongation increases rapidly with decrease in strain rate resulting in superplastic-like ductility more than 300%. The deformation mechanism is discussed on the basis of microstructural observation. The mechanical anisotropy will also be described briefly.

9:45 AM

AUTOMOTIVE ALLOYS: *D. J. Lloyd*[1]; [1]Alcan International, Ltd., Kingston Research and Development Center, P.O. Box 8400, 945 Princess St., Kingston, Ontario K7L 5L9 Canada

The ability to form the 6000 series Al alloys into complex shapes is important for the application of these alloys as automotive skin sheet. The alloys therefore require a level of ductility and bendability to achieve the degree of forming required. In this paper the work hardening, tensile ductility and bendability of several 6000 series alloys are examined. It is shown that the tensile ductility is predominantly controlled by the work hardening behavior, which subsequently controls geometrical instability and tensile failure. However, the bendability of the alloys is fracture controlled, and is essentially independent of tensile elongation. The bendability of the different alloys can be understood on the basis of fracture controlled models and the different fracture strains of the alloys. An approximate equation, developed previously primarily for steels, provides an adequate description of the minimum radius achieved in bending.

10:00 AM BREAK

10:30 AM

THE EFFECT OF PRE-AGING ON ARTIFICIAL AGING RESPONSE IN Al-Mg-Si-Cu ALLOY 6111: *Weifang Miao*[1]; David E. Laughlin[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Although heat treatable Al-Mg-Si-Cu alloys are increasingly used as automotive body materials, the potential of these alloys is not fully utilized due to low temperature and short duration of the automotive paint bake cycle, during which the alloys are artificially age hardened after stamping. Moreover, practically unavoidable natural aging process is usually detrimental to the artificial aging response of these alloys. In this study, the effect of various pre-aging treatments on the artificial aging response of an aluminum alloy 6111 was investigated using hardness measurements, differential scanning calorimetry and transmission electron microscopy. It has been found that appropriate pre-aging treatments slow down the natural aging process and significantly increase the artificial aging response. The changes in artificial aging response are closely related to the variations in size and distribution of precipitates. Financial support from Ford Motor Company and NEDO is gratefully acknowledged.

10:45 AM

EFFECT OF ALLOY COMPOSITION AND PROCESSING ON THE FORMABILITY OF ALLOYS BASED ON Al-3 WT.% Mg: *S. A. Court*[1]; [1]Alcan International, Ltd., Banbury Laboratory, Southam Rd., Banbury, Oxon OX16 7SP England

Aluminum alloys in sheet form based on Al-3 wt.%Mg(AA5754and AA5454) have found application in automotive structures as they offer attractive combination of strength, formability, corrosion resistance and weldability. However, to allow down-gauging there is a continued need for 5xxx series alloys with increased strength as compared with the existing alloys, which must be achieved without adversely affecting other sheet properties. In this paper, the results of studies aimed at understanding the effects of both alloy composition and sheet processing on the strength and formability, in particular, of alloys based on Al-3 wt%Mg, are described.

11:00 AM

INVESTIGATION OF PROCESSING-MICROSTRUCTURE-PROPERTY RELATIONSHIPS IN Al-Mg-Mn ALLOYS: *Mark C. Carroll*[1]; Jörg M. K. Wiezorek[2]; Michael J. Mills[1]; *Glenn S. Daehn*[1]; Brady R. Dunbar[3]; K. Paul Smith[3]; [1]Ohio State University, Dept. of Mats. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Uni-

versity of Pittsburgh, Dept. of Mats. Sci. and Eng., University Way, Pittsburgh, PA 15261 USA; [3]Century Aluminum Corporation, Ravenswood Operations, Ravenswood, WV 26164 USA

The ever-increasing demand for high-strength, low-cost materials in various applications has necessitated the development of premium quality Al-Mg-Mn (5000 Series) alloys. Progress has been made in processing which can produce alloys with an optimum combination of dispersion and precipitation hardening, solid-solution strengthening, and fine grain sizes. Some of the optimum characteristics of these alloys, however, are subject to the deleterious effects of grain growth and Mg diffusion, leading to the formation of the Mg-based beta-phase along grain boundaries. Together with a decrease in strength of the alloy due to the loss of the Mg solid-solution strengthening, there is also concern over the alloys susceptibility to stress corrosion due to the galvanic reaction between the Mg-enriched areas and the Al matrix. The present work investigates strength and corrosion characteristics of Al-Mg-Mn alloys, with a focus on derivatives of AA5083. Observations have been made which correlate microstructure with mechanical strength and corrosion resistance in relation to established AA5083 baseline properties. Microstructural characterization by optical microscopy and analytical techniques of transmission electron microscopy (TEM) has been combined with physical testing, which includes hardness and exfoliation tests. The local microchemistries at grain boundaries and in the vicinity of inhomogeneities, as well as the identity of various types of dispersoid and precipitate phases, have been determined by energy dispersive X-ray spectroscopy and diffraction techniques using a state-of-the-art field-emission CM300FEG TEM. The microstructural observations are correlated to the macroscopic property measurements. For example, the tendency of such alloying elements as Zn to preferentially diffuse to grain boundaries and limit the diffusion of Mg is demonstrated. On the basis of observations and experimental results, the performance of Al-Mg-Mn alloys with regard to microstructural characteristics is discussed. Financial and technical support by Century Aluminum Corporation, Ravenswood Operations, is acknowledged.

11:15 AM

A SMALL-CRACK FRACTURE MECHANICS-BASED MODEL FOR PREDICTING THE S-N RESPONSE OF CAST ALUMINUM ALLOYS: *Michael J. Caton*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Mats. Sci. and Eng., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; [2]Ford Motor Company, Mats. Sci. Dept. - Research Staff, MD 3182 Scientific Research Lab, 20000 Rotunda, Dearborn, MI 48121 USA

With increased use of cast aluminum alloys in demanding automotive applications, there exists a need for a reliable method to accurately predict an alloy's fatigue properties. It has been speculated that the fatigue properties of aluminum castings are dominated by the propagation of cracks which initiate from shrinkage pores ranging in size from ~ 50 to 500 µm. With this in mind, the growth of small fatigue cracks (~15µm to 2 mm) was monitored in a 319-type aluminum, a common Al-Si-Cu alloy used in casting engine blocks and cylinder heads. A plot of the growth rates as a function of the stress intensity factor range, ΔK, indicates the existence of a small crack effect as well as a significant influence of the solidification rate and applied stress level on the resulting da/dN vs. ΔK curves. A crack growth relation proposed by Nisitani and others is modified and used to correlate the small crack data. Predictions of the S-N response based on this small crack model and based on a Paris relation for long crack data are compared to experimental S-N data.

11:30 AM

AGE HARDENING BEHAVIOR IN A COMMERCIAL 319-TYPE ALUMINUM ALLOY: *Ray Jahn*[1]; William T. Donlon[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Research Laboratories, P.O. Box 2053, MD 3135, Dearborn, MI 48121 USA

Cast Al-Si-Cu alloys are utilized by the automotive industry for engine blocks and cylinder heads. Detailed understanding of the ageing characteristics of these types of alloys is important to optimize the processing of these components to yield the desired physical and mechanical properties. Age hardening curves for temperatures between 100 and 305°C have been determined after a solution treatment at 495ϒC for 8 hours for a commercial grade 319-type Al alloy (7.08wt% Si, 3.26%Cu, 0.16%Mg, 0.23%Mn, 0.4%max Fe, 0.25%max Zn, 0.25%max Ti). The influence of casting solidification rate on the subsequent ageing response was characterized by measuring the hardening curves in samples solidified at 2.38ϒC/sec (SDAS=30ÿm) and 0.033°C/sec (SDAS=110ÿm). The phases formed within the primary aluminum dendrites during each stage of age hardening were characterized using a JEOL 2000FX TEM and an OXFORD ISIS microanalysis system. The initial stage of hardening at 100 and 150°C is believed to be due to clustering of Cu atoms, followed by the formation of GP zones. At longer times and higher temperatures small (5nm) lath-shaped Q (Al5Mg8Si6Cu2) precipitates and disk-shaped ÿ‘ precipitates occur at peak hardness and coarsen during overaging. The observed orientation relationship for ÿ‘-Al is [100]ÿ‘ // [100]Al and [001]ÿ‘ // [001]Al, and that for Q-Al [0001]Q // [001]Al and (1120)Q // (310)Al, consistent with the literature results. Coprecipitation of Q and ÿ‘ phases were found in as-cast material with an approximate orientation relationship [0001]Q // [100]ÿ‘ // [100]Al. The size, spacing and density of ÿ‘ precipitates have been determined as a function of ageing temperature and time.

11:45 AM

A PROCESS MODEL FOR THE AGE HARDENING OF A 319-TYPE ALUMINUM ALLOYS: *P. M. Reeber*[1]; [1]University of Michigan, Dept. of Mats. Sci. & 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 Al 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.

CARBON TECHNOLOGY: Anode Plant Retrofitting & Operation

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

Tuesday AM — Room: 6D
March 2, 1999 — Location: Convention Center

Session Chair: Doug Parrish, Tomago Aluminium, Raymond Terrace, NSW, 2324, Australia

8:30 AM SESSION CHAIRMAN INTRODUCTION

8:35 AM

ADVANCES IN FORMING: Werner K. Fischer[1]; *Markus W. Meier*[1]; [1]R&D Carbon, Ltd., P.O. Box 362, Sierre CH-3960 Switzerland

Anodes of any size can be vibrated or pressed. Various reasons have led to the introduction of vibrators in the late seventies, mainly: - Increasing anode size - Higher investment cost for large presses - Pressing capacity limited by the mixing throughput - Unsatisfactory paste cooling. These reasons have become irrelevant due to the technical development in the recent years. Presses actually have some distinct advantages compared to vibrators: lower investment cost for anode

cooling, lower maintenance, higher availability, lower noise level and lower PAH emissions. The resulting anode quality is similar for both pressed and vibrated anodes. It is therefore believed that the anode press will experience a renaissance in the near future.

9:00 AM

COOLING OF GREEN ANODES AFTER FORMING: Werner K. Fischer[1]; Markus W. Meier[1]; *Mauriz Lustenberger*[1]; [1]R&D Carbon, Ltd., P.O. Box 362, Sierre CH-3960 Switzerland

The cooling process is limited by the temperature conductivity of the green anode. Experimental work and simulations indicate, that only a small outer crust is cooled down during the main cooling process, while the inner part of the anode body remains hot. This may result in frozen stresses, which can be reduced if the anodes are cooled with a higher temperature level of the cooling medium. No significant loss of cooling performance is observed. A lower forming temperature drastically reduces the heat flow from the inner part to the outside of the anode. The anodes can then be cooled by forced air convection in a wind tunnel during the transport to the anode storage. Therefore, anode cooling by water spraying or immersion in a water bath can be avoided. The requirement of lowering the forming temperature emphasizes the installation of an anode press.

9:25 AM

CONTROL OF PAH AND CTPV'S FOR ANODE MANUFACTURING PROCESS BY REGENERATIVE THERMAL OXIDATION: *Gerard Gosselin*[1]; Guy Drouin[1]; [1]Biothermica International, 3333 Boul. Cavendish, Suite 440, Montreal, Quebec H4B 2M5 Canada

Regenerative thermal oxidation process was applied to an anode paste plant. This plant has a non standard mixing configuration. Ko-kneader and intensive mixers operate in series. This configuration allows to increase petroleum coke and binder pitch temperatures prior to the first mixing stage with the Ko-kneader mixer. Then, the hot paste is sent to an intensive mixer that is operated as a mixer-cooler. By using water to cool down the paste, PAH (polycyclic aromatic hydrocarbons) and CTPV's (coal tar pitch volatiles) emissions increased to a level higher than usual, requiring a more efficient control. RTO (regenerative thermal oxidation) was selected as the more interesting scrubbing process. PAH and CTPV's emissions measurements after installation of the new equipment show a reduction of 99.9%.

9:50 AM BREAK

10:10 AM

EXHAUSTION AND SCRUBBING OF PITCH FUMES, AN ENVIRONMENTAL PROBLEM AT ALBRAS: *Paulo Douglas Vasconcelos*[1]; Andre Mesquita[2]; Joao Quaresma[2]; Daniel Cruz[2]; [1]Albras Aluminiol Brasileiro, ROD.PA483 KM21, Barcarena, PA 68447-000 Brasil; [2]Universidade Federal do Para, Centro Tecnologico, Av. Bernardo Sayao, s/n, Belem Brasil

Albras operates two anode plants with a capacity of approximately 280,000 mtpy using 40,500 mtpy of pencil pitch, which is used as a dry aggregate (coke) binder. In the green paste production, a fraction of pitch volatiles results in form of vapor (70ppm) that is released from the mixers, belt conveyor and anode vibrocompactor. These volatiles contain polyciclic aromatic hydrocarbons (PAH) that in certain concentrations are harmful to the health. Therefore the exhaustion and the treatment of these volatiles are strictly necessary to improve the workplace environment. The paper shows how the original inefficient wet scrubber was successfully substituted by a dry scrubber, designed in cooperation with the local Federal University. A mathematical model was developed to study the adsoption of fumes by ultrafine coke particles from the ball mill bag filter.

10:35 AM

CONVERSION OF A CLOSED FURNACE TO THE OPEN TYPE TECHNOLOGY AT ALUMINIUM BAHRAIN: *Jean-Claude Thomas*[1]; Jaffar G. Ameeri[2]; Philippe Breme[1]; Jean-Christophe Rotger[1]; [1]Aluminium Pechiney, Aluval, B.P.7, Voreppe F-38340 France; [2]Aluminium Bahrain, B.S.C. (C), P.O. Box 570, Manama Bahrain

As part of an expansion program of its smelter in Bahrain, Alba signed in 1995 a contract with Pechiney for the reconstruction of one of its closed furnaces in an open type one, inside the existing concrete casing. The furnace was stopped in May 1996 and restarted six months later after complete transformation. Further to production which has been increased by 34% at a low capital cost, this conversion has much improved the consistency and homogeneity of the baking parameters. The transformation required some modifications of the casing, in particular the construction of a central air cooled wall to separate the two bays. The new fluewall characterized by an unusual rectangular shape was optimized in detail thanks to an improved modelization of the internal gas flow.

11:00 AM

METHODS TO PREDICT FLUE LIFE AND FLUE FAILURE RATES IN ANODE BAKING FURNACES: *Gerald F. Chovanec*[1]; [1]Century Aluminum of West Virginia, Primary Products, P.O. Box 98, Ravenswood, WV 26164 USA

Methods to predict flue life/failure rates in anode baking furnaces have been developed. One method incorporates extrapolation of cumulative percent failure graphs using linear regression; the second method is based on a correlation between flue condition ("flue severity") and flue age. Failure patterns were analyzed for flue groups installed over a period of about eight years. Cumulative percent flue failure curves for groups ranging in size from 11 to 322 flues displayed "near normal" failure distributions. By "backtracking" failure data, it was deduced that flue life/failure rate would have been satisfactorily predictable after about 25 - 30% failure; below this level, predictions would have been erratic. To provide an alternative method of flue failure predictions for groups in which an insufficient number, or no failures have occurred, a relationship between "flue severity" and flue age was developed and provides a satisfactory first approximation of flue failure rates.

11:25 AM

FLUE GAS MANAGEMENT: *W. Leisenberg*[1]; [1]Innovatherm, GmbH + Company, Butzbach D-35510 Germany

Because of environmental demands In the last time flue gas analysis and control became more important. Contradicting goals as are: a minium of flue gas volume, sufficient heat transfer to the preheating zone, maximum fuel efficiency and perfect volatile combustion ask for better knowledge of the process and for advanced strategy of the flue gas control and the fuel injection, especially for furnaces with narrow flues. Furthermore the implications of CO and NOx content in the flue and the way of fuel injection as are short or long pulses and the addition of primary combustion air have to be taken in account and ask for a control strategy, which regards all those parameters and should be called a flue gas management rather than a control. In the last year investigations have been done on this objective and the results will be presented on the session.

CAST SHOP TECHNOLOGY: DC Casting/ Modeling I

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

Tuesday AM Room: 6C
March 2, 1999 Location: Convention Center

Session Chair: Dr. Yogesh Sahai, Ohio State University, Columbus, OH 43210-1179 USA

8:30 AM

INTERPRETATION OF THE BLEED OUT PHENOMENON OF EXTRUSION INGOT CASTING APPLYING THE MATHEMATICAL

MODEL ALSIM: *Bjorn Rune Henriksen*[1]; Einar K. Jensen[1]; Dag Mortensen[2]; [1]Elkem ASA Research, P.O. Box 8040 Vaagsbygd, 4602 Kristiansand, Vest-Agder Norway; [2]Institute for Energy Technology, P.O. Box 40, 2007 Kjeller, Lillestrøm Norway

The DC-casting process has been continuously improved with respect to ingot quality and recovery since its invention. The main challenge at the Elkem Lista cast house was to reduce the scrap rate due to billet bleed out in the start-up phase. The aim of this investigation was to understand the mechanism of bleed out and to find actions to avoid this phenomenon. Based on experimental casting trials and corresponding mathematical modeling a mechanism for billet bleed out is proposed. The results from ALSIM and experimental observations (micographs, temperature measurements) show that one of the most important factors affecting the bleed out is the filling of the mold table. The effect of starting block geometry/material, casting speed/temperature, starting block position and hot top design is studied as well.

8:55 AM

DETERMINATION OF THE THERMAL BOUNDARY CONDITIONS DURING ALUMINUM DC CASTING FROM EXPERIMENTAL DATA USING INVERSE MODELING: I.J. Opstelten[1]; J.M. Rabenberg[1]; [1]Koninklijke Hoogovens N.V.; P.O. Box 10000, 1970 CA IJmuiden, The Netherlands

The work described in this contribution is part of the EMPACT project. In this project several European partners (both from industry and university) have joined to develop tools for improving the casting of aluminum ingots. It involves the use and development of mathematical models which describe the micro- and macro segregation, the fluid flow and the thermo-mechanical behavior of the ingots. An accurate description of the thermal boundary conditions is of paramount importance for the correct prediction of the casting processes. Moreover, these boundary conditions serve also as an important control parameter in practice. Until now the heat transfer data obtained from immersion quenching experiments or semi-empirical relations are frequently used in the numerical models. The conditions during actual casts can differ substantially though, from the conditions for which this heat transfer data was obtained. Recently inverse modeling techniques have been used to obtain the thermal boundary conditions from thermocouple measurements during an actual cast. Although this seems the best way to obtain thermal boundary conditions for actual casting conditions, it is rather impractical when the influence of a number of parameters is to be established. In the present study the influence of several practical parameters on the thermal boundary conditions are experimentally determined. To this end the temperature-time history is measured at several locations inside an aluminum test block during steady-state waterfilm quenching. These measurements are input to an inverse model, which finds the thermal boundary conditions. The influence of casting speed, aluminum alloy type, cooling waterflow rate and water temperature, surface structure and waterfilm generator type is thus investigated. Several results of these measurements and the inverse modeling process will be shown.

9:20 AM

MECHANISMS OF SURFACE FORMATION DURING DIRECT CHILL (DC) CASTING: *Steinar J. Benum*[1]; Arild Håkonsen[1]; [1]Hydro Aluminium A.S., R&D Materials Technology, P.O. Box 219, Sunndalsoera, Sunndal N-6600 Norway

The mechanisms of surface zone formation during DC casting were summarised by K. Buxmann in 1974 (Metall). After his overview, changes have been utilized both in casting technology as well as knowledge of the solidification process. The present paper is and review of the mechanisms explained by Buxmann put in light of new casting technology. The paper emphasises the major change in casting technology that occurred when the air/gas-slip technology was developed by Showa early in the 1980's. The most characteristic of this process is the large meniscus constructed by detgas pocket between the metal and the mould. There are two segregation phenomena connected to the meniscus; a) meniscus segregation and b) change in meniscus shape. The first phenomenon is a metallostatic driven segregation that occurs when the pressure in the metal becomes larger than the additive pressures from the surrounding atmosphere, i.e., air pressure and meniscus strength. This segregation mechanism is similar to that often associated with Bergmann zones (Bergmann, J. of Metals (1973)). The second segregation phenomenon is given by the fluctuations of the meniscus itself. The size of the meniscus makes it susceptible to pertubations caused by changes in metal level and gas pressure. The resulting movements affect both the segregation pattern as well as the surface topography.

9:45 AM

COUPLED STRESS, THERMAL AND FLUID FLOW MODELLING OF THE START-UP PHASE OF ALUMINIUM SHEET INGOT CASTING: *Hallvard G. Fjær*[1]; Dag Mortensen[1]; Arild Håkonsen[2]; Einar A. Sørheim[1]; [1]Institute for Energy Technology, Mats. and Corrosion Technology Dept., P.O. Box 40, Kjeller N-2007 Norway; [2]Hydro Aluminium, R&D Materials Technology, P.O. Box 219, Sunndalsøra N-6601 Norway

The start-up of the DC-casting process of aluminium sheet ingots is a complex process where the development of temperatures, melt flow and deformations and stresses in the solid are essentially coupled phenomena. This work focuses on the influence of thermally induced deformations on the heat transfer at the ingot surfaces, which in particular involves development of an air gap between the ingot and the starting block as well as water intrusion into this gap. It also involves development of air gaps at the mould surface. These mechanisms are discussed and investigated by transient 3D simulations involving coupled stress, thermal and fluid flow modelling. This coupling has been implemented by communication between submodels which may run on different CPUs in a heterogeneous computer network. The coupled model have been applied on the casting of commercial size sheet ingots. Calculated results are compared with temperature measurements.

10:10 AM

EFFECT OF AS-CAST MICROSTRUCTURE AND SUBSEQUENT PROCESSING ON BANDING IN ROLLED Al-SHEETS: *Trond Furu*[1]; Hans Erik Vatne[1]; [1]Hydro Aluminium A.S., R&D Mats. Tech., P.O. Box 219, Sunndalsøra, Sunndal N-6600 Norway

Typical casting defects of rolling ingots of AA1XXX series alloys have been investigated. The effect of alloy chemistry (focused on 1050 and 1200 type alloys), grain refinement practice and casting conditions have been investigated and discussed. The most common defects are fir-tree structures and feathery crystals, which both may lead to streaks after rolling and etching/anodising. The fir-tree structures are influenced by cooling rate, grain refinement procedure and Fe:Si ratio (and amount). Feathery crystals are also influenced by grain refinement practice and melt temperature. In addition, another type of rolling defect was investigated; edge cracks. This rolling defect is not directly linked to a casting defect, but certainly to the quality and microstructure of the rolling ingot.

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

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

Tuesday AM Room: 15A
March 2, 1999 Location: Convention Center

Session Chair: M. McLean, Imperial College of Science, London, England; G.S. Daehn, Ohio State University, Columbus, OH, USA

8:30 AM INTERMETALLICS

8:30 AM INVITED PAPER

MECHANISMS OF PRIMARY AND SECONDARY CREEP IN NEAR-GAMMA TiAl ALLOYS: *Thomas R. Bieler*[1]; Dong Yi Seo[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Primary and secondary creep has been measured in a number of near-gamma TiAl alloys and in a polysynthetically twinned (PST) crystal (representing a single unit of lamellar microstructure). The PST crystals exhibit activation energies from temperature change tests between 130 and 190 kJ/mol, which are about half of the activation energies commonly measured in polycrystal specimens. Normal transients after temperature or stress changes are observed in both PST and polycrystal specimens. In polycrystals, two stages of primary creep deformation are evident in measured data, an early and rapid process that is exhausted in the first 0.2-0.5% strain, and a different process that dominates the deformation up through the minimum creep rate. From interrupted creep tests, the early process causes lamellar refinement, (i.e. a reduction in lamellar spacing), which correlates closely with the PST crystal deformation, and the second process does not affect the lamellar spacing significantly, but it has deformation para meters similar to secondary creep. The effects of differing heat treatment and alloy composition on the primary creep deformation processes indicate that primary creep resistance can be improved in some alloys by as much as 3 times with heat treatments, whereas addition of refractory elements, interstitials, and elements that stimulate formation of fine precipitates can increase the creep resistance by an order of magnitude. Analysis of deformation parameters indicate that the mechanisms of creep deformation are not effectively described with theory developed for metals and alloys.

8:55 AM INVITED PAPER

MECHANISMS OF CREEP DEFORMATION IN TITANIUM ALLOYS: *Michael John Mills*[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Due to their attractive strength and low density, conventional alpha-beta titanium alloys such as Ti-6242 are utilized extensively in rotating components in gas turbine engines. Relatively new TiAl alloys with slightly sub-stoichiometric compositions, are comprised of a lamellar microstructure consisting of fine laths of the intermetallic gamma and alpha-2 phases. These "gamma" alloys are presently under development for application in higher temperature, reciprocating components. In these applications, creep deformation is often a limiting material property in these two important titanium alloy systems. In this presentation, the microstructural origins of creep deformation and the sources of creep strength will be discussed for both alpha-beta titanium alloys at ambient and intermediate temperatures, as well as for gamma-TiAl at higher temperatures. Our understanding of the modes of deformation and important dislocation/interface interactions as revealed by TEM investigation will be presented. Attempts t o model these deformation modes in light of this microstructural information will also be described. Funding for this work has been provided by the Air Force Office of Scientific Research and by the National Science Foundation.

9:20 AM

CREEP AND MICROSTRUCTURE OF NEAR Γ-TiAl INTERMETALLICS: *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

Creep and microstructure of pure γ-phase and two-phase γ / α_2 intermetallic TiAl alloys are investigated. The basic creep data obtained in tension and compression in the temperature range 923-1100K and for applied stresses between 50 and 400MPa are presented. It is shown that, while the pure g-phase Ti-52at%Al alloy is brittle in tensile creep at 1100K and for applied stresses above 150MPa, the two-phase γ / α_2 fully lamellar Ti-48Al-2Cr-2Nb-1B (in at%) alloy exhibits a remarkable ductility at considerably lower temperatures. A conventional light microscopy, SEM and TEM were used to quantify the microstructure of the alloys before and after creep. A systematic study of the grain size revealed that a grain growth occurs during creep of Ti-52at%Al alloy while a grain refinement due to recrystallization is observed after creep in Ti-48Al-2Cr-2Nb-1B alloy. The difference in the creep behaviour of the investigated alloys is attributed to the different intensity of dislo cation glide, twinning and recrystallization, the microstructural processes which operate in the range of external conditions studied.

9:40 AM

CREEP/FATIGUE DEFORMATION AND CRACK GROWTH IN A Γ-TITANIUM ALUMINIDE TURBINE BLADE MATERIAL: *Kamran Nikbin*[1]; [1]Imperial College, Mech. Eng. Dept., Exhibition Rd., London SW7 2BX UK

In order to improve the performance of gas turbines there is a trend towards the use of higher strength to weight and stiffness to weight ratio materials. In this investigation the intermetallic, γ-titanium aluminide (γ-TiAl), which is a candidate material for blade and disc applications, is examined. The intermetallic g-TiAl is a candidate material for high strength to weight applications in advanced gas turbines. However it exhibits limited creep ductility at temperatures up to about 650ϒC and there is a need to establish it's tolerance to the types of stress state generated at stress concentrations. Different batches of the material were tested. Most high temperature gas turbine components experience multiaxial stress states during operation at sites of stress concentration caused by thermal gradients, holes and sharp changes in section. In order to determine the useful lifetimes of these components at elevated temperatures data from uniaxial bar, notched bar and fracture mechanics specimens are analysed taking into account the effects of multiaxial states of stress which are present for the different geometries. Subsequently metallographic examination was carried out on failed specimens. Significant results have been achieved in the experimental and the modelling aspects of failure of materials under a tri-axial state of stress. From notch rupture tests, fatigue crack growth tests at 700ϒC and supporting microstructural evidence it has been found that the alloy has good notch strengthening properties. However it has also been shown that it has unpredictable crack initiation and growth properties. In addition the radical variation of properties from batch to batch which apparently have the same nominal composition and heat treatment suggests that further work is needed to determine the suitability of this alloy for turbine blade applications.

10:00 AM

A NEW SUGGESTION OF CREEP DEFORMATION MECHANISM OF TiAl: *Soo Woo Nam*[1]; Han Seo Cho[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Sci. and Eng., 373-1 Kusong-dong Yusong-gu, Taejon, ChungNam 305-701 Korea

It is well known that the steady-state creep deformation mechanism of pure metals and certain solid solution alloys is controlled by the

dislocation climb process with the help of self-diffusion. Therefore, the creep deformation activation energy in the steady-state is measured and known to be that of self-diffusion. However, in the primary stage, the dislocation density is increasing with primary strain and the activation energy in this stage is measure to be lower than that of steady-state. This is because of the fact that the higher effective stress (since the dislocation density is lower) in the primary stage is responsible in lowering the apparent activation energy, Qapp = Qo σeV, where Qapp is the apparent activation energy, Q_o is the self-diffusion activation energy, σ_e is the effective stress and V is the activation volume. In case of lamella TiAl, same as in metals the normal primary creep stage is observed. However, as the primary strain increases, it is found that the dislocation density is decreasing but the primary creep activation energy is increasing from the value of the activation energy of self-diffusion of Ti in TiAl (300kJ/mol) to the value of about 400kJ/mol which is measured to be the creep activation energy of TiAl obtained using the steady state creep rate. This decreasing dislocation density in primary creep stage is the opposite phenomenon to that of common metals and the higher activation energy than that of self-diffusion is also different from the cases of metals. Small amount of prestrain is found to be responsible for the reduction of the initial dislocation density and this prestrained specimen shows significantly reduced primary creep strain and the creep activation energy in the primary stage is also measured to be about 400kJ/mol. During creep deformation of lamella TiAl, as the initial dislocation density is decreasing, α_2 phase is found to be transforming to γ phase to generate new dislocations which contribute creep deformation. In other words, this phase transformation is the source of the dislocation generation for the continuous creep deformation. Therefore, the phase transformation is suggested to be one of the rate controlling processes whose activation energy is thought to be higher than that of self-diffusion, i.e., about 400kJ/mol.

10:20 AM INVITED PAPER

CREEP DEFORMATION IN DAMAGE TOLERANT NIOBIUM ALUMINIDE INTERMETALLICS: *R. W. Hayes*[1]; F. Ye[2]; W. O. Soboyejo[2]; [1]Metals Technology, Inc., 19801 Nordhoff St., Northridge, CA 91324 USA; [2]The Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

The micromechanisms of creep deformation will be elucidated for a new class of damage tolerant niobium aluminide (Nb3Al-xTi-yMo-zCr) intermetallics with partially ordered two phase (B2 + orthorhombic) crystal structures. These alloys have been shown to have attractive combinations of room temperature ductility (5-30%), fracture toughness (40-100 MPa $m^{-1/2}$) and fatigue resistance. However, there have been no published reports of the elevated-temperature creep behavior of niobium aluminides. Primary and secondary creep rate data will be presented for the temperature range (between 650 and 750°C) in which uncoated alloys have been shown to have the potential for structural applications. Activation energies and creep exponents obtained from stabilized orthorhombic + B2 microstructures will also be compared with previously reported data for B2 and orthorhombic intermetallics. The implications of the results will be discussed for potential intermediate-temperature structural applications of niobium aluminide intermetallics.

10:45 AM BREAK

10:50 AM THRESHOLD STRESS

10:50 AM INVITED PAPER

INTERPRETATION OF THRESHOLD STRESSES AND OBSTACLE STRENGTHS IN CREEP OF PARTICILE STRENGTHENED MATERIALS: *Jeffery C. Gibeling*[1]; [1]University of California, Dept. of Chem. Eng. and Mats. Sci., One Shields Ave., Davis, CA 95616 USA

Virtually all descriptions of creep deformation in particle-strengthened materials rely on the introduction of some empirical quantity with units of stress. These are variously known as threshold stresses, obstacle glide resistances, detachment stresses and athermal stresses. The purpose of these terms is to appropriately scale the data so that parameters such as stress exponents and activation energies conform to expected values based on an assumed deformation mechanism. However, the theoretical justification and physical interpretation of these stress terms is often limited, and the experimental methods for deriving the values of these quantities is usually indirect. Advances in the application of new materials will require a more thorough fundamental understanding creep in these advanced materials coupled with appropriate models of deformation. The different interpretations of threshold stresses and obstacle strength parameters for dispersion-strengthened metals and metal matrix composites are reviewed. The experimental evidence in support of the various approaches to describing creep in particle strengthened materials and the methods for determining parameter values are examined. Particular attention is given to the assumption of constant structure creep behavior in identifying an appropriate stress exponent and the need to use a value of 8 with caution. The importance of considering mobile dislocation density and the interactions of multiple dislocations with particles is discussed. The limitations of using similar approaches for dispersion strengthened metals and metal matrix composites, which have very different particle size scales, are explored. Finally, the need to properly account for evidence of creep transients is emphasized.

11:15 AM INVITED PAPER

MICROSTRUCTURAL THRESHOLD EVENTS, BACK STRESSES AND MINIMUM CREEP RATES - A NEW WAY OF INTERPRETING CREEP IN ADVANCED ENGINEERING MATERIALS: *Gunther Eggeler*[1]; [1]Institut für Werkstoffe-Werkstoffwissenschaft, Ruhr-Universität, Bochum

Traditional approaches to rationalize creep of materials use minimum creep rate data in an attempt to study and understand their dependence on stress and temperature. Steady state creep, power law creep and the back stress approach are famous concepts which have been used to describe creep deformation mechanisms of materials over the last three decades. While these concepts have not lost any of their power in helping to analyze creep behavior there are cases where more is required to understand what actually controls creep. Thus microstructural threshold events on different size scales are important in creep of many advanced materials like (i) detachment of dislocations from oxide dispersoids in ODS alloys, (ii) breakage of fibres in short fiber reinforced MMCs, (iii) cutting of gamma prime particles in superalloy single crystals and (iv) the onset of dynamic recrystallization during creep in near gamma titanium aluminides. Creep is generally the result of the coupling of a number of interconnected microstructural processes and often microstructural threshold events play a key role. As deformation proceeds macroscopic (classical) back stresses build up and counteract the applied stress. But back stresses have a microstructural origin, they are not constant throughout the creep process and what microstructural and mechanical consequences this has is a first important point which will be discussed in the present paper. In the light of microstructural threshold events macroscopic back stresses which counteract the applied stress represent the driving forces which trigger the onset of threshold events. This is a second important point which will be highlighted in the paper. And finally, microstructural threshold events can result in microstructural changes which influence the creep process. In addition to a general analysis of how microstructural threshold events can be integrated into creep models two examples of advanced engineering alloys where microstructural threshold events are important are presented in more detail, (i) creep in short fiber reinforced aluminium alloys and (ii) creep of gamma prime strengthened super alloy single crystals. One important general conclusion is that progress in the understanding of creep mechanisms in advanced engineering alloys must be guided by state of the art microstructural analysis. Merely fitting data to classical concepts does not help to progress the field of creep of advanced engineering alloys. State of the art creep models for advanced engineering materials should reflect all important elementary microstructural processes including threshold events and not only rationalize the stress and temperature dependence of the minimum creep rate but also describe the shape of individual creep curves.

11:40 AM INVITED PAPER

THRESHOLD STRESSES IN HIGH TEMPERATURE YIELDING AND CREEP: A CRITICAL REVIEW: *M. Heilmaier*[1]; B. Reppich[2]; [1]University of Western Australia, Dept. of Mech. & Mats. Eng., Nedlands 6907 Australia; [2]University of Erlangen-Nürnberg, Institute of Mats. Sci., Erlangen D-91058 Germany

At low temperatures incoherent oxide particles introduce a yield strength increment for dislocation glide due to Orowan bypassing, called

threshold stress σ_{th}. However, at high temperatures such a true threshold does not exist. In contrast, the material deforms even under the lowest stress applied. Therefore, the Orowan process has lost its predominating microstructural significance. Instead, models for climb threshold developed in the past with respect to different particle shapes and climb geometries will be discussed by introducing a universal parameter called climb resistance R. An essential result for most oxide dispersion strengthened (ODS) alloys is that the detachment of the partially relaxed dislocation from the particle-matrix interface controls the creep kinetics. As a consequence, our concept tackles creep thresholds in two steps: first, we associate σ_{th} with the apparent particle hardening contribution σ_p to realize a creep rate in the particle-strengthened alloy equivalent to that of the corresponding single-phase matrix. Second, the course of σ_p with respect to strain rate is modelled by applying the above mentioned particle hardening mechanisms. We exemplify and verify our approach with selected ODS platinum-, nickel- and iron-based alloys. [1]Permanent address: Institute of Solid State and Materials Research Dresden, D-01069 Dresden, Germany

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Continuous Casting

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, Processing 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

Tuesday AM Room: 2
March 2, 1999 Location: Convention Center

Session Chairs: James W. Evans, University of California, Dept. of Mats. and Min. Eng., Berkeley, CA 94720 USA; Achilles Vassillcos, US Steel Technical Center, Monroeville, PA 15146 USA

8:30 AM

COMPUTATIONAL FLUID DYNAMICS MODELLING OF TUNDISHES AND CONTINUOUS CASTING MOULDS: *Ian Hamill*[1]; Tracy Lucas[2]; [1]CFX International, AEA Technology, 8.19 Harwell, Oxon OX11 ORA England; [2]Kvaerner Metals Continuous Casting, Prince of Wales Rd., Sheffield, Yorks S9 4EX England

Issues of importance in the area of continuous casting centre around product quality. This can be affected by particle inclusions transported into the mould from upstream processes and by draw-down of particles from the meniscus. Turbulence of the meniscus can increase the latter, and is also detrimental to product surface quality. Suitably validated Computational Fluid Dynamics (CFD) modelling can provide the operator with an efficient tool with which to investigate the effects of changes to the design and operation of the caster and associated plant. In this paper, examples will be presented of the use of the CFD software, CFX, to simulate: inclusion motion and removal in tundishes and continuous casting moulds using an algebraic slip model; free-surface shape and turbulence in the mould using deforming meshes; solidification in the mould using a fixed-grid, source-based method; argon gas injection through a submerged entry nozzle using a full multiphase model. The CFD simulations are complemented by detailed Particle Image Velocimetry measurements performed on a full-size water model of a thin-slab mould and upper strand. Close agreement is demonstrated between the predictions and experiments.

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SIMULATIONS OF GAS ENTRAINMENT IN A CONTINUOUS CASTING TUNDISH: *D. Morton*[1]; S. Louhenkilpi[1]; [1]Helsinki University of Technology, Dept. of Mats. Sci. and Rock Eng., P.O. Box 6200, Hut 02015 Finland

In continuous casting operations, metal is transferred from a ladle to the tundish via a submerged shrouding tube. When changing the ladle, blockage of the outlet is a common problem. In clearing such blockages, the nozzle must be removed from the ladle outlet so that an oxygen lance can be applied. Until the nozzle can be replaced, the outlet stream becomes a free jet that impacts the surface of the molten material contained in the tundish. Such impacts are undesirable since reoxidation of the metal increases the number of inclusions found in the cast product. This effect is significant enough that it often results in several tons of low-grade steel. In this paper, we shall present the results of numerical study of gas entrainment due to a continuous stream. CFD simulations have been performed using a "finite volume" approach that includes improved techniques for modell in extreme free surface deformation and the effect of surface tension. The numerical results clarify the mechanism by which a continuous stream carries an annular film of gas beneath the surface of the receiving fluid. Furthermore, the simulations predict the growth of instabilities on the surface of this film and subsequent break up to form air bubbles. Finally, the results of the study are used to make estimates of the bubble sizes entrained in the tundish when feed from a ladle occurs as a continuous open stream.

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ELECTROMAGNETIC METHODS TO REDUCE CLOGGING IN TUNDISH NOZZLES: Laszlo Kadar[2]; *J. D. Lavers*[1]; [1]University of Toronto, Dept. of Elect. & Computer Eng., Toronto, Ontario M5S 3E4 Canada; [2]Hatch Associates, 2800 Speakman Dr., Mississauga, Ontario L5K 2R7 Canada

This paper discusses the potential of electromagnetic forces to reduce, and for certain situations to eliminate, the stagnation regions that lead to clogging of tundish nozzles. In the continuous casting of aluminum killed steel the tundish nozzle often experiences clogging problems. Water model studies have shown that the clogging problem is dependent on the flow pattern in the nozzle entry region. In particular, the flow can separate at the entrance to the nozzle which leads to the development of a recirculation region. Although a nozzle can be designed such that no recirculation develops, even a small misalignment from the vertical positioning of the nozzle may render the design ineffective. To reduce the possibility of nozzle clogging, the recirculation zone must be reduced or eliminated. This may be achieved by moving the fluid downwards or raising the turbulence level in the separation region. In aerodynamics, this is achieved by blowing or introducing suction in the stagnation zone. In the case of a tundish nozzle, a similar effect can be achieved by using electromagnetic forces. To generate the required electromagnetic force, two systems are considered. The first is based on injecting a DC current; the second by using electromagnetic induction. Both methods are described in this paper. Order of magnitude calculations are provided to illustrate that both methods are essentially feasible. The performance of each method in terms of modifying the turbulent flow characteristics in the nozzle entry region is then examined using a numerical model. It is shown that both methods can greatly reduce the recirculation zone. A cylindrical nozzle was chosen for the model studies since it represents a worst case situation. However, the widely used radius entry nozzle was also considered. On the basis of the model studies, it was concluded that whereas both of the proposed methods have the potential to reduce the troublesome recirculation zone, the induction method appears to be simpler in terms of practical implementation.

9:30 AM

RECENT RESULTS OF MODELING OF METAL DELIVERY SYSTEMS USED IN EM AND DC CASTING OF ALUMINUM: W. Kinzy Jones[1]; Dong Xu[1]; *J. W. Evans*[1]; [1]University of California, Dept. of Mats. Sci. & Mineral Eng., 585 Evans Hall #1760, Berkeley, CA 94720 USA

It is well understood that the flow of liquid metal in the mold region of the ingot can have a notable effect on the overall solidification behavior. Previously our group has characterized the flow in this region in a water model using particle imaging velocimetry. The flow is significantly effected by the specifics of the metal delivery system, i.e. size, geometry, used during the cast. Recently, a time-averaged 3-D numerical model of the nozzle, combo bag and sump region has been developed and its validity has been tested against the experimental data. A deformable free surface was built into the numerical model to better represent the actual surface of the physical sump. Cases were compared for different sized combo bags as well as bags that have blocked. The results show good agreement between the physical and mathematical models.

9:50 AM

MATHEMATICAL AND PHYSICAL MODELLING OF STEEL FLOW IN TWIN ROLL/HORIZONTAL BELT CASTERS: *R. I. L. Guthrie*[1]; R. P. Taveres[1]; P. Q. Netto[1]; [1]McGill University, McGill Metals Processing Center, M.H.Wong Eng. Bldg., Montreal H3A 2B2 Canada

Near net shape casting technology is one of the most important areas for research and development in the iron and steel industry today. Two of the key elements in the successful design and operation of thin strip casting machines are metal delivery systems and productivity. Productivity, in turn, is related to the rate and net amount of heat that can be extracted by a particular machine. This work demonstrates the complex interactions that take place between metal flow and solidification, depending on the nozzle delivery system, the rate of heat extraction, and the physical configuration of the caster.

10:10 AM BREAK

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DESIGNING OF SUBMERGED ENTRY NOZZLE FOR SLAB CONTINUOUS CASTER WITH FLOW AND SOLIDIFICATION ANALYSIS: *Masafumi Morishita*[1]; Hirofumi Tai[1]; Toshiya Miyake[1]; [1]Kobe Steel, Ltd., 2222-1 Ikeda, Onoe-cho, Kakogawa, Hyogo 675 Japan

The shape of the Submerged Entry Nozzle for Kakogawa No. 4-2 slab continuous caster was optimized in order to prevent corner cracking while high through-put casting. Numerical simulations about the flow and solidification in the mold was successfully applied for the optimization.

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TWO PHASE FLOW NUMERICAL SIMULATION OF MOLTEN STEEL AND ARGON GAS IN CONTINUOUS CASTER MOLD: *Noriko Kubo*[1]; Jun Kubota[1]; [1]NKK Corporation, Materials & Processing Research Center, Steelmaking Laboratory, Kokan-cho Fukuyama, Hiroshima 721-8510 Japan

For steel continuous castings, it is important to control molten steel velocity at meniscus, since it is closely related to surface defects of the resultant products. Argon gas is injected into the mold with the molten steel to prevent clogging of the submerged entry nozzle. To investigate the influence of argon gas on molten steel flow in the mold, numerical simulations have been carried out by using a two phase CFD model. Simulation results indicate a strong impact of argo gas on the molten steel flow. As argon gas bubbles ascend near the nozzle by buoyancy, they entrench molten steel upward to the meniscus. As a result, the molten steel flows outward to the narrow face of the mold at meniscus. This flow direction is opposite to the case without argon gas, in which the molten steel flows inward from the narrow face of the mold at meniscus.

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NUMERICAL SIMULATION OF FLUID MOTION IN CONTINUOUS CASTING PROCESS WITH LINEAR MOTORS: *Ch. Strohm*[1]; Y. Couvat Du Terrail[1]; S. A. Rotelec[1]; M. C. Nove[1]; [1]EPM MADYLAM, ENSHMG, BP 95, 38402 Saint Martin d'Herdes, Cedex France

A specialized software has been developed for the numerical simulation of the liquid metal flow control in the particular case of the continuous casting processes. The device includes linear motors placed at the upper level of the ingot in order to control the metal speed and direction. Electromagnetic forces are calculated in three dimensions with a finite element program based on A, A-V formulation. Hydrodynamic in the ingot is solved in two dimensions using a finite different scheme using w-Y formulation. The weak coupling between electromagnetic and hydrodynamic is realized by interpolating the Laplacian forces on the nodes of the hydrodynamic grid: firstly as a mean efficient value on each node and secondly as a time dependant vector pulsating at low frequency. Then, a strong coupling is implemented by adding to the weak coupling two alternate models. In the first one, the induced currents are computed again in the hydrodynamic scheme, assuming no changes in the magnetic field. In the second approach all the electromagnetic variables are calculated again in the finite element program. The electromagnetic results have been compared to experimental trials with an empty ingot. Numerical comparisons with 3D fluent calculations have been done for the fluid flow validation.

11:30 AM

MODELING OF ELECTROMAGNETIC STIRRING IN CONTINUOUS CASTING OF STEEL: *Thinium T. Natarajan*[1]; Nagy El-Kaddah[2]; [1]U.S. Steel, Technical Center, 4000 Technical Center Dr., Monroeville, PA 15146 USA; [2]The University of Alabama, Dept. of Metall. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Electromagnetic stirring is widely used in continuous casting of steel as a means to improve homogeneity of cast billets and strands. The degree of mixing in the molten pool strongly depends on the stirrer design and dimensions. This paper describes a new general formulation for modeling electromagnetically driven flow in three-dimensional induction systems, and its application to modeling electromagnetic and flow phenomena in sub-mold rotary stirring of square billets. This model is based upon finite element solution of current and magnetic scalar potential equations for computing the electromagnetic field and Navier-Stokes equations together with the k-e turbulent model for velocity and turbulent parameter calculations. The computed force field in the billet revealed that the billet corners are the source of vorticity of the force field, which drives the flow in the bulk. Effective mixing was found to be confined to the region surrounded by the stirrer, and the mixing intensity diminishes rapidly beyond the edges of the stirrer. The significance of these findings on the effectiveness of rotary stirring on melt homogenization and inclusion removal will be discussed.

GENERAL ABSTRACTS: Session 4 - Fatigue, Corrosion Fatigue and Wear

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

Tuesday AM — Room: 12
March 2, 1999 — Location: Convention Center

Session Chairs: Indranath Dutta, 93943 Naval Postgraduate School, Monterey, CA 93943 USA; Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712-1063 USA

8:30 AM

THE EFFECTS OF RESIDUAL STRESSES ON FATIGUE CRACK INITIATION: Jonathan D. Almer[2]; *Jerome B. Cohen*[1]; Brian Moran[3]; [1]Northwestern University, Robert R. McCormick School of Engineering and Applied Science, 2225 North Campus Dr., MLSB/Room 2036, Evanston, IL 60208-3108 USA; [2]Linkoping University, Konstruktions Material, IKP (Mech. Eng. Dept.), Linkoping S-581 83 Sweden; [3]Northwestern University, Dept. of Civil Eng., Technological Institute, Room A332, 2145 Sheridan Rd., Evanston, IL 60208-3109 USA

X-ray diffraction and Finite Element (FE) analysis have been used to study crack initiation in the presence of residual stresses. Residual stresses were introduced into polished double-edge notched (DEN) 1080 steel specimens by prestraining and press-fit operations, and initiation was monitored during high-cycle fatigue tests using surface replicas. The local stress-strain behavior in the vicinity of the initiation sites was tracked using FE analysis. Microbeam x-ray diffraction measurements were also made in each phase of the steel to determine both residual macrostresses, which were compared with the FE computations, and microstresses. Microstresses were observed to fade rapidly during fatigue, while macrostresses relaxed less rapidly and were observed to strongly affect crack initiation behavior. This fatigue behavior was then correlated with the observed stresses using a traditional stress-life relationship which includes mean stress effects. This research was funded in part by the Office of Naval Research, Grant No. N00014-90-J-1374.

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FATIGUE OF A FORMULA I TITANIUM CONNECTING ROD: *Gregor K. Mori*[1]; Karl L. Maurer[1]; [1]Dept. of Failure Analysis, Franz-Josef-Str., Leoben 18 A-8700 Austria

During training for the Italian Formula I Grand Prix in Monza 1997 one of the racing cars had an engine defect. Investigations of the broken parts showed a fatigue failure of one of the titanium connecting rods. The crack was situated after one third of the shaft length near to the small eye at the piston. The fracture plane progressed perpendicular to the shaft axis indicating some bending or tensile stresses. However the fracture origin was at the inner edge of one of the flanges of the H-formed shaft of the connecting rod. Additionally there the fracture plane was under 45 ° to the shaft axis which indicated the presence of torsion stresses. A small crack of 150 µm length and app. 200 µm depth had to be opened to prove a torsion fatigue fracture. A failure analysis is given and the reasons for the appearance of torsion cracks in a connecting rod are presented.

9:10 AM

CORROSION-FATIGUE CRACK INITIATION MECHANISMS IN ALUMINUM ALLOYS: *P. S. Pao*[1]; S. J. Gill[1]; C. R. Feng[1]; [1]Naval Research Laboratory, Code 6312, 4555 Overlook Ave., SW, Washington, D.C. 20375 USA

The mechanisms of corrosion-fatigue crack initiation by pitting in high-strength 7000-series aluminum alloys were studied. The alloys used in this investigation were 7075-T7351 and 7050-T7451. Corrosion pits were formed in 3.5% NaCl solution by the electrochemical reaction between the constituent particles and the surrounding matrix. Depending on whether these particles were cathodic or anodic relative to the alloy matrix, the pits formed as a consequence of matrix (around cathodic particles) or particle (for anodic particles) dissolution. Because these particles tend to cluster parallel to the rolling plane in the rolling direction, significant pit growth from pit coalescence was observed following prolonged exposure in a salt water environment. The effect of pre-existing corrosion pits on fatigue crack initiation was investigated using blunt-notched fracture mechanics specimens (oriented in the short-transverse direction) to simulate rivet holes. Results to date indicate that the presence of corrosion pits can significantly shorten the fatigue crack initiation life and decrease the threshold stress intensity of the alloy by as much as 50 percent. Post initiation fractographic analyses further confirmed that, when corrosion pits were present, fatigue cracks always initiated from these pits. In the absence of pits, fatigue cracks initiated from large inclusions. The identification of the constituent particles, the mechanism of pit formation and growth, and the analyses of fatigue crack initiation kinetics are discussed.

9:30 AM

CORROSIVE WEAR BEHAVIOR OF 7075 ALUMINUM ALLOY USING SCRATCH TECHNIQUE: *Gustavo Vasquez*[1]; Shailendra K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metall. and Mats. Eng., El Paso, TX 79968-05620 USA

The corrosive wear behavior of 7075 aluminum alloy has been investigated using scratch test. Samples immersed in an electrolyte have been subjected to impact by a stylus containing a diamond Vickers indenter at its tip. The transient current developed due to the creation of a new bare surface exposed to the corrosive environment has been determined as a function of solutionizing time. The details of scratches produced by impact and continuous loading have been compared. SEM observations have been correlated to the transient current generated and grain size developed as a result of varying solutionizing time during the corrosive wear process. Results on 7075 aluminum alloys will be compared with those previously reported for 6061 and 2014 aluminum alloys under identical set of experimental conditions.

9:50 AM BREAK

10:00 AM

THE SLIDING WEAR MECHANISMS OF PTFE COMPOSITES: *Jaydeep P. Khedkar*[1]; [1]Louisiana State University, Dept. of Mats. Sci. and Eng. Program, Dept. of Mech. Eng., 2508, CEBA, Baton Rouge, LA 70803 USA

The tribological performance of polytetrafluroethylene (PTFE) and PTFE composites under fixed operating conditions has been examined. The role of individual filler material influencing the friction and the wear properties of PTFE is critically evaluated. The wear tests are carried out on a laboratory pin on disc type apparatus and these results are compared with those conducted on a mechanical seal test rig simulating industrial conditions. A detailed microstructural examination is carried out using SEM so as to detect the possible modes of failures. DTA analysis of the materials is also presented to study the relative heat absorbing capacity and thermal stability of the various composites. An attempt is made at correlating the DTA results with the tribological performance of the composites. Finally, the main objective here is to propose the dominant interactive wear mechanisms during sliding of PTFE and its composites.

10:20 AM

HOT ISOSTATIC PRESSING OF TUNGSTEN CARBIDE TO STAINLESS STEEL FOR USE IN HIGH ABRASION/CORROSION RESISTANT APPLICATIONS: *Adele Crystal Boone*[1]; [1]MIT Student, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA

To successfully Hot Isostatic Press (HIP) tungsten carbide (94-WC, 6%Co) to 17-4 precipitation hardened stainless steel for the application of high abrasion/corrosion resistant separator blades through the use of interlayers. Investigated interlayers include nickel-, silver-, molybdenum-, and copper-based foils. Metal-ceramet matrices and the tiling of carbides were also explored. Hip is of great interest because it produces a superior bond to the current technique of vacuum furnace nickel-based brazing. HIPping enhances fatigue strength and tensile ductility, reduces the scatter in creep life, and reduces the foundry scrap and inspection costs.

10:40 AM

THERMAL SPRAY AND MECHANICAL PROPERTIES OF NANOSTRUCTURED OXIDE COATINGS: *Leon L. Shaw*[1]; Ruiming Ren[1]; Daniel Goberman[1]; Maurice Gell[1]; [1]University of Connecticut, Dept.of Metall. and Mats. Eng., 97 N. Eagleville Rd., U-136, Institute of Mats. Sci., Storrs, CT 06269 USA

Nanostructured coatings can provide significant improvements in wear and erosion resistance deriving from enhanced hardness and toughness. In this paper, Al2O3 - 13 wt.% TiO2 coatings formed via thermal spray approach using reconstituted nanosized Al2O3 and TiO2 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 coatings obtained from reconstituted nano-powder feeds are discussed and related to thermalspray conditions.

11:00 AM

STRUCTURE AND PROPERTIES OF PVD-COATINGS BY MEANS OF IMPACT TESTER: *E. Lugscheider*[1]; O. Knotek[1]; Christian Wolff[1]; Stephen Barwulf[1]; [1]University of Technology, RWTH-Aachen, Augustinebach 4-22, Aachen 52062 Germany

Machine parts like rolling bearings or gears are stressed during operation in a changing mechanical strain. This causes wear by impacts and wear by rolling which is marked by the so called surface ruin. The

appearance of surface fatigue is based upon structural transformation, cracking and cracking-growth processes and ends with the separation of debris particles caused by the above mentioned permanent changing strain. The final stage, which is equivalent to the component failure, is the so called pitting on the technical surface, which is characteristically named surface fatigue. The impact tester is used for detailed research about failure mechanisms of thin films. Statements about the adherence of hard material coatings under dynamic compressive stress can be made using this test method, due to the possibility to simulate some effects of rolling strain. Therefore a hard metal ball strikes with a frequency of up to 50 Hz onto the surface. The altitude stress can be varied to get a detailed evaluation of fatigue strength under reversal strain. Selected hard material coatings were analyzed after testing with the described method applying an impact force of 300 N, 500 N and 700 N. In the framework of this presentation MSIP (Magnetron-Sputter-Ion-Plating) coatings on titanium- and chromium basis were used. The fatigue defects and the results of this study will be discussed depending on structure and morphology of thin films.

11:20 AM

DIFFUSION-BASED MICROALLOYING OF ALUMINUM VIA REACTION SINTERING: *Paul D. Bishop*[1]; M.C. Chaturvedi[2]; Georges J. Kipouros[1]; William F. Caley[1]; [1]DalTech-Dalhousie University, Mining and Metlalurgical Engineering; 1360 Barrington St., Halifax, Nova Scotia B3J 2X4 Canada; [2]University of Manitoba, Mechanical and Industrial Engineering, 15 Gillson St., Winnipeg, Manitoba R3T 5V6 Canada

In an effort to improve the properties of aluminum-based alloys, microalloying with selected elements has proven to be a viable method. Despite the broad range of alloys and microalloying additions possible considered, most rely on traditional ingot metallurgy practices. In this study an alternative technique using powder metallurgy principles and a diffusion/reaction sintering procedure is described. The basis of the method is the pressing of an outer shell of material containing the source microalloying element, in mineral form, about an aluminum pre-pressed core. Subsequent super-solidus liquid phase sintering liberated the cationic species of interest and promoted diffusion into the core material. Samples of a ternary Al-Cu-Mg alloy, as well as Al-2014 were successfully microalloyed with Sn and Ag. The resulting materials were age hardened and examined for hardness and tensile properties. As well, the microstructures were evaluated using scanning electron microscopy and X-ray diffraction, and selected samples were subjected to wear testing. Results were comparable to those obtained using ingot metallurgy techniques, yet the method offered the processing flexibility associated with powder metallurgy processes. Thus, the technique is an attractive alternative when a controlled level, composition and location of microalloying is of importance.

GENERAL ABSTRACTS: Session 5 - Physical Metallurgy: Thermodynamics, Interfaces & Diffusion

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

Tuesday AM Room: 13
March 2, 1999 Location: Convention Center

Session Chairs: Saskia Duyvesteyn, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Dennis D. Keiser, Argonne National Laboratory, Nuclear Technology, Idaho Falls, ID 83403-2528 USA

8:30 AM

CHEMICAL AND STRUCTURAL ENERGY OF B1 TYPE COMPOUND/AUSTENITE INTERFACE: *Zhi-Gang Yang*[1]; Masato Enomoto[1]; [1]Ibaraki University, Dept. of Mats. Sci., Nakanarusawa 4-12-1, Hitachi, Ibaraki 316-8511 Japan

Various inclusions in steel are B1 type compounds, cube-on-cube orientated with austenite. The interfacial energy between B1 compound and austenite may play an important role in controlling the nucleation of ferrite on inclusions. The semi-coherent interfacial energy is known as having both chemical component and structural component. The chemical energy is calculated by nearest neighbor broken bond method following Lee and Aaronson, and the structural energy is computed based on the misfit dislocation model following Spanos. The result indicates that both chemical component and structural component contribute significantly to the total interfacial energy, although the latter is larger. Especially, the anisotropy of chemical energy is much stronger than that of structural energy, consequently it dominates the wulff construction and corresponding equilibrium shape of B1 compound in austenite. The temperature dependence of the interfacial energy and the concentration profile across the interface are also studied in this work.

8:50 AM

VIBRATIONAL ENTROPY DIFFERENCES BETWEEN AUSTENITIC AND MARTENSITIC NiTi BY LOW TEMPERATURE INELASTIC NEUTRON SCATTERING: *P. D. Bogdanoff*[1]; B. Fultz[1]; S. Rosenkranz[2]; [1]California Institute of Technology, Engineering and Applied Sciences, 138-78, Pasadena, CA 91125 USA; [2]Argonne National Laboratory, Mats. Sci. Div., Argonne, IL 60439 USA

We report our measurements for the difference in vibrational entropy between the low temperature martensite and high temperature austenite phases of NiTi, as measured by low temperature inelastic neutron scattering. The low temperature phase exhibits significant anharmonicity in the temperature range 0 to 200K, as measured by the phonon density of states (DOS). The austenite DOS shows little change between room temperature and 580 K, which is consistent with previous work. Earlier work done at Caltech measured the vibrational entropy difference between austenitic and martensitic NiTi at 0.4±0.1 kB/atom using low temperature calorimetry, a result in agreement with literature values. Work supported by U.S. DOE DE-FG03-96ER45572 and US DOE, BES-MS, contract W-31-109-ENG-38

9:10 AM

ENTHALPY OF FORMATION IN BINARY LAVES PHASES: *Jiahong Zhu*[1]; C. T. Liu[1]; L. M. Pike[1]; P. K. Liaw[2]; [1]Oak Ridge National Lab., Metals and Ceramics Division, P.O. Box 2008 , MS 6115, Oak Ridge, TN 37831-6115 USA; [2]University of Tennessee, Dept. of Mats. Sci. and Eng., Dougherty Eng. Bldg., Knoxville, TN 37996 USA

Enthalpies of formation of Laves phases have been critically surveyed and reviewed. It was found that the bond characteristics in Laves phases are not necessarily metallic in nature for both non-transition metal Laves phases and Laves phases containing transition metals. There are metallic, covalent and ionic bonds, or a mixed metallic-covalent-ionic bond. A thermodynamic interpretation is offered to explain the size ratio limits for Laves phase formation. As the deviation from the ideal size ratio increases, the maximum negative enthalpy of formation decreases linearly, which is assumed to be due to the elastic strain energy expended in compressing the atoms. At RA/RB =1.03 and 1.65, the enthalpy of formation reaches zero. Further deviation in the RA/RB ratio will lead to the enthalpy of formation positive. Thus, the free energy of formation becomes positive, due to the negligible entropy of formation term. Therefore, Laves phases can only be stabilized in certain atomic size, RA/RB, ratios. The enthalpies of formation calculated by the semiempirical Miedema model is in good agreement with the available experimental data for transition-metal and Al lanthanide Laves phases. This means that Miedema's theory can be used to predict quantitatively, at least qualitatively, the enthalpies of formation of transition-metal and Al lanthanide Laves phase systems.

9:30 AM

INFLUENCE OF COHERENCY STRAIN ON INTERFACE MIGRATION: *Jong K. Lee*[1]; [1]Michigan Technological University, Dept. of Metall. Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

For a long time, coherency strain is known to play a crucial role in interface migration and thus microstructural evolution during alloy processing, but its understanding has been frustrated with the mathematical complexity associated with elasticity. This work presents some fascinating results of the Discrete Atom Method, which is predicated upon the combination of statistical mechanics and linear elasticity. It is found that coherency strain induces interfacial waves whose dynamic activities drive a coherent interface to migrate. The wavelength of these waves is proportional to the ratio of the interfacial energy to the strain energy, but strongly depends on elastic constants, interface geometry, and diffusion temperature. The waves also act as a source for the introduction of fresh ledges necessary in a ledge growth mechanism, and also responsible for the formation of pits, huts, and domes encountered in epitaxially-grown thin films. In an anisotropic system, stress concentrations are built at the regions o f elastically hard directions. In such a case, the interface is found initially to move toward the elastically-hard phase. If it is in a highly non-equilibrium state, coherency-induced wave interactions can create deep grooves on the interface, eventually splitting precipitates into smaller particles. Role of coherency strain in ordering and segregation phenomena will be also discussed.

9:50 AM BREAK

10:00 AM
TIME-DEPENDENT DIFFUSION PATHS ASSOCIATED WITH TRANSIENT PHASES IN TERNARY SYSTEMS: *Carelyn E. Campbell*[1]; William J. Boettinger[1]; [1]National Institute of Standards and Technology, Metallurgy, Bldg. 223 A153, Gaithersburg, MD 20899 USA

Unlike the time-independent composition paths associated with infinite diffusion couples, the composition paths of semi-infinite and finite diffusion couples can be time-dependent. This time dependence can result in the formation and/or dissolution of transient phases in multicomponent systems. The ability to predict the presence of transient phases during the diffusion process is important for many industrial processes, including transient liquid phase bonding. Numerical simulations of semi-infinite diffusion couples are compared to simulations of infinite diffusion couples for ternary systems with three species having equivalent diffusivities and ternary systems having one fast diffusing specie. For the Ni-Al-B system, diffusion paths through single-phase (γ-FCC), two-phase (liquid + γ-FCC) and three-phase (liquid + γ-FCC + intermetallic $Ni_{20}Al_3B_{(6\text{-}12)}$) regions are analyzed.

10:20 AM
AN INTERDIFFUSION MICROSTRUCTURE MAP FOR GAMMA/GAMMA+BETA DIFFUSION COUPLES IN THE Al-Cr-Ni SYSTEM: *Huimin Amy Chen*[1]; *John Eric Morral*[1]; [1]University of Connecticut, Dept. of Metall. and Mats. Eng., 97 North Eagleville Rd., U-136, Storrs, CT 06269 USA

ABSTRACT NOT AVAILABLE

10:40 AM
HIGHER ORDER BOUNDARIES IN MULTIPHASE DIFFUSION COUPLES: *John Eric Morral*[1]; Huimin Amy Chen[1]; [1]University of Connecticut, Dept. of Metall. and Mats. Eng., 97 North Eagleville Rd., U-136, Storrs, CT 06269 USA

ABSTRACT NOT AVAILABLE

11:00 AM
RAPID PREDICTION OF TIME-TEMPERATURE-TRANSFORMATION DIAGRAMS OF LOW AND MEDIUM CARBON MULTICOMPONENT ALLOY STEELS BY A COMPUTER EXPERT SYSTEM: *Zhenbo Zhao*[1]; *Derek O. Northwood*[2]; Cheng Liu[3]; Yunxu Liu[3]; Qihui Zhu[3]; [1]University of Windsor, Mech. & Mats. Eng., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ryerson Polytechnic University, Eng. & Applied Sci., 350 Victoria St., Toronto, Ontario M5B 2K3 Canada; [3]Jilin Institute of Technology, Mats. Eng., 76 Yanan Dalu, Changchun, Jilin 130012 PR China

A computer expert system for the rapid prediction of time-temperature-transformation (TTT) diagrams of low and medium carbon multicomponent alloy steels was established. The principle and methodology for the rapid prediction of TTT diagrams were described. Some critical points of transformation used to model the time-temperature-transformation diagrams of muticomponent alloy steels were represented as functions of alloy chemistry. The bay between the two C-curves (pearlite and bainite), as a function of alloy chemistry, can be predicted accurately in terms of the precise calculation of Bs temperature (a new equation of Bs temperature has been proposed). It is believed that the reasonable prediction can be achieved in much larger concentration ranges. The limitations of alloy chemistry in most of other models can be avoided effectively because a relatively larger range of alloy chemistry (Ni, Mn, W, Mo, Cr, Si, V, Co, Cu) were considered in most equations. The commercial application potential in the design of new multicomponent alloy steels was discussed also.

11:20 AM
THE EFFECT OF CU ON PHASE TRANSFORMATIONS IN LOW CARBON STEELS: Shaun Dilney[1]; Matthias Militzer[1]; [1]The Centre for Metallurgical Process Engineering, University of British Columbia, Vancouver, BC, V6T 1Z4 Canada

Increases in the level of residual Cu in steel scrap is forcing electric arc furnace steelmakers to process steel containing higher levels of Cu. Although Cu can be detrimental during processing, causing hot shortness, it can also be a beneficial alloying element, improving mechanical properties through precipitation hardening and grain size refinement. The aim of this work is to investigate the effect of Cu on the phase transformation kinetics of low carbon steels, containing 0.05wt% to 0.8wt%Cu. The austenite decomposition kinetics has been investigated with continuous cooling tests using a dilatometer and a Gleeble 1500 thermo-mechanical simulator. The study emphasizes the cooling conditions of a hot strip mill run-out table. Based on the experimental results, a model is proposed which accounts for the effects of composition, cooling rate, and austenite grain size on the austenite-to-ferrite transformation kinetics, the resulting ferrite grain size and the mechanical properties.

HIGH-TEMPERATURE SUPERCONDUCTORS: SYNTHESIS, FABRICATION AND APPLICATION: Applications & Tape Fabrication

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

Tuesday AM Room: 18
March 2, 1999 Location: Convention Center

Session Chairs: Judith MacManus-Driscoll, Imperial College of Science & Technology, Centre for High Temperature Superconductivity, London, England SW7 2BZ UK; H. W. Neumuller, Siemens AG, Corporate R & D, Erlangen Germany

8:30 AM INVITED PAPER
HIGH TEMPERATURE SUPERCONDUCTIVITY: PROSPECT FOR ELECTRONIC APPLICATIONS: *Donald U. Gubser*[1]; [1]Naval Research Laboratory, Mats. Sci. & Tech., Code 6300, 4555 Overlook Ave., SW, Washington, D.C. 20375-5343 USA

High temperature superconducting (HTS) films are of sufficient quality and size to permit system demonstrations of superconductivity for a variety of electronic applications. The most prominent device structures are the high Q filters. These filters are used in base stations for

cellular communications where range and signal quality are important; in radar systems where high levels of discrimination are important; and in communication systems where weak signals are received. Another electronic application area for HTS materials is in Superconducting Quantum Interference Devices (SQUID) that are used for detection of weak magnetic anomalies and in low noise signal amplifiers. A description of superconducting materials and system requirements will be given for these applications. Important to the introduction of these materials is the development of low cost refrigeration systems. Status and specifications of refrigeration systems for these applications will also be presented.

8:50 AM INVITED PAPER

DESIGN OF SUPERCONDUCTING POWER CABLES: *Rainer Wesche*[1]; Alexander Anghel[1]; Bruno Jakob[1]; Gabriel Pasztor[1]; Georg Vécsey[1]; [1]EPFL, CRPP, c/o Paul Scherrer Institute, WMHA C31, Villigen PSI, AG CH-5232 Switzerland

Superconducting power cables are expected to be one of the most promising energy applications of high-temperature superconductors. The main sources of losses in superconducting cables are ac, dielectric and thermal losses. The interplay of cable geometry, operating current and resulting losses is considered. The design studies indicate that for Ag/Bi-2223 cables with warm dielectric an operating temperature well below 77 K is favourable. The superconductor properties required for the cable application are briefly discussed. Finally, the design concept for a superconducting single-phase transmission cable with a power of 112.5 MW is presented. In 1999 a gas-cooled prototype cable of 5 m length will be constructed and tested in collaboration with Kabelwerke Brugg. Work financially supported by the Swiss Federal Office of Energy and PSEL.

9:10 AM INVITED PAPER

HIGH-T_C SUPERCONDUCTIVITY FOR POWER ENGINEERING - ACTIVITIES AT SIEMENS: *H.-W. Neumüller*[1]; [1]Siemens AG, Corporate Research & Development, P.O. Box 3220, Erlangen, D-91050 Germany

The introduction of superconductivity into electrical power equipment leads to an improvement of existing components i.e. cables, transformers, with respect to smaller size, larger power per unit and better efficiency. Novel components like fault current limiters promise more economic solutions for the distribution and control of power. A very important key role for a successful realisation of HTS-products is played by long-term and secured materials development. Vacuumschmelze Hanau, currently manufactures 2223 BSCCO tape in lengths of 400-600 m having current densities between 22 and 24 KA/cm^2 for use in cables, transformers and magnet windings. Large-area YBCO plate conductors (up to 20x20 cm^2; J_c= 1.5 - 3 MA/cm^2) are under production for the planned 1 MVA Model within our HTS fault current limiter program. Significant progress has been achieved in the field of HTS power cables: a 10 m machine-stranded cable conductor has been successfully tested under A C and DC conditions and a 50 m flexible 100 kV single phase cable is already under construction.

9:30 AM INVITED PAPER

PROGRESS IN Bi-2212 HIGH FIELD INSERT COILS: *Kenneth R. Marken*[1]; Weiming Dai[1]; Huub Weijers[2]; Qingyu Hu[2]; Yusuf Hascicek[2]; Justin Schwartz[2]; [1]Oxford Instruments, 600 Milik St., Carteret, NJ 07008-0429 USA; [2]National High Magnetic Field Lab, 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Progress has been made toward a goal of adding 3 Tesla in a 20 Tesla background using coils of BiSrCaCuO-2212 tape conductor. The conductor used is a 19 filament tape which has demonstrated short sample current density exceeding 100 kA/cm2 at 4.2 K, 20 T. Double pancake coils were wound in 3 sizes and stacked into concentric sections. A strengthened alloy conductor matrix was used in the outer sections in order to control strains in the higher stress regions of the stack. Individual coil sections, as well as the stacked magnet, will be tested in a 20 T large bore resistive magnet at the NHMFL. Test results and design details will be presented.

9:50 AM INVITED PAPER

RECENT DEVELOPMENT OF HIGH JC Bi-2212/Ag TAPES AND WIRES: *Hitoshi Kitaguchi*[1]; Hanping Miao[1]; Hiroaki Kumakura[1]; Kazumasa Togano[1]; Takayo Hasegawa[2]; Michiya Okada[3]; Jun-ichi Sato[4]; [1]National Research Institute for Metals, 1st Group, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Showa Electric Wire & Cable Co., Ltd., 2-1-1 Odasakae, Kawasaki, Kanagawa 210-0843 Japan; [3]Hitachi, Ltd., 7-1-1 Ohmika, Hitachi, Ibaraki 319-1292 Japan; [4]Hitachi Cable, Ltd., 3550 Kidamari, Tsuchiura, Ibaraki 300 Japan

Remarkable progress on Bi-2212/Ag conductor fabrication has been achieved recently. Two of newly developed process that enables to obtain high Jc Bi-2212/Ag conductors for practical applications are reported. PAIR process is the combination of pre-annealing (PA) and subsequent intermediate rolling (IR) processes and is performed prior to melt-solidification process. By performing PAIR process, Bi-2212 grain alignment and intergrain connectivity are much improved and a large Jc enhancement can be expected. PAIR processed samples have transport Jc-oxide (4.2 K, 10 T) > 500,000 A/cm2, which corresponds to Jc-conductor of 900 A/mm2 and twice higher than that for the samples melt-solidified without PAIR process. ROSAT (Rotation-Symmetric Arranged Tape-in-tube) wire has been developed. Round wire with small Jc anisotropy can be fabricated with this method. ROSAT wire of 1.6 mm in diameter carries >900 A (4.2K, 0T) which corresponds to Jc-oxide of 250,000 A/cm2 and Jc-conductor of 440 A/mm2.

10:10 AM BREAK

10:20 AM INVITED PAPER

IMPROVEMENT OF FLUX PINNING CENTRES IN MELT PROCESSED Bi-2212/Ag TAPE: *Judith MacManus-Driscoll*[1]; Alice Crossley[1]; David Caplin[1]; [1]Imperial College of Science Technology and Medicine, Centre for High Temperature Superconductivity, Prince Consort Rd., London, England SW7 2BZ UK

In the Bi-Sr-Ca-Cu-O superconductors there has been considerable success in overcoming weak link problems, and at high temperatures intra-grain flux motion is thought to be the dominant dissipation mechanism. The microstructural and electrical homogeneity of $Bi_2Sr_2CaCu_2O_{8+d}$ tape (Bi-2212/Ag) provides a good basis to study the effects of doping and secondary phase precipitates on flux pinning in a polycrystalline melt-processed system. In this study the complex Bi-2212 phase diagram has been used to quantitatively introduce secondary phase precipitates and magnetisation measurements taken in order to assess their effects on pinning within the grains of the Bi-2212. In addition to this, the effect of doping on flux pinning within the grains has been investigated.

10:40 AM INVITED PAPER

MATERIALS ASPECTS REGARDING TO PROCESSING OF BSCCO TAPES: *Peter Majewski*[1]; Andre Aubele[1]; Fritz Aldinger[1]; [1]Max-Planck-Institut für Metallforschung, PML, Heisenbergstr. 5, Stuttgart 70569 Germany

The results of detailed studies of the materials of BSCCO will be presented and discussed in terms of the optimization of the processing. The Ag sheath material of the tapes has been found to dissolve up to 0.3% Cu. This aspect is of great importance for the stoichiometry of the ceramic material, because during processing the Cu content of the ceramic decreases due to diffusion of Cu into the sheath material. The Pb solubility of (Bi,Pb)2+xSr2Ca2Cu3O10+d [(Bi,Pb)2223] has been found to vary significantly with temperature, oxygen partial pressure and presence of silver. This aspect is of great importance for the cooling of the tapes.

11:00 AM INVITED PAPER

HIGH QUALITY HTSC NANOPOWDERS: FABRICATION AND SCALE-UP: Andrei A. Zagorodni[1]; Lingna Wang[1]; Yu Zhang[1]; Kenneth Billqvist[2]; K. Venkat Rao[1]; *Mamoun Muhammed*[1]; [1]Royal Institute of Technology, Mats. Chem. Div., Teknikringen 30, Stockholm 100 44 Sweden; [2]NANOCHEM AB., Stockholm 100 44 Sweden

Since the discovery of HTSC materials, a number of bulk applications have been developed based on their novel properties. Currently, several prototype components and systems are being tested. A major challenge is the availability of high quality powder in large quantities.

The basic requirements of the high quality powders are homogeneity, precise chemical composition, and high reactivity to allow solid-state reactions at short time scales. We report on the development and scale-up of a co-precipitation method for the fabrication of several classes of HTSC nanopowders with exact composition. Large-scale computer-controlled production facility has been constructed. Batches of 3-5 kg powder precursor have been achieved. These nanopowders have been used for the fabrication of Ag-clad tapes with high Jc at very short processing time.

11:20 AM INVITED PAPER

FORECASTING TAPE PERFORMANCE FROM MICROSTRUCTURAL STUDIES IN Ag-CLAD Bi-2223 COMPOSITE CONDUCTORS: Nazarali N. Merchant[1]; *Victor A. Maroni*[1]; Albert K. Fischer[1]; Gilbert N. Riley[2]; Ron D. Parrella[2]; [1]Argonne National Laboratory, CMT, 9700 S. Cass Ave., Argonne, IL 60626 USA; [2]American Superconductor, Two Technology Dr., Westborough, MA 01581-1727 USA

Microstructural studies on transverse sections of mono- and multifilament silver-clad Bi-2223 composite conductors after first stage annealing under optimum conditions have been performed on a variety of wires and tapes over the past several years. We have found a consistent correlation between the microstructures observed by scanning electron microscopy on short anneal samples (quenced in oil) and the final Jc measurements performed on fully processed tapes. Tapes that have microstructures with controlled secondary phase growth and good texture evolution of the layered phase in the early stages of annealing, eventually exhibit high Jcs (50-70 kA/cm2) at 77K and zero field. Conversely, tapes that show extensive secondary phase growth and rapid conversion of the precursor powder to Bi-2223, tend to exhibit poor Jc performance (5-15 kA/cm2). We have also observed that tapes with reproducible conversion kinetics (via XRD), exhibit superior superconducting properties after full processing.

11:40 AM INVITED PAPER

FABRICATION AND CHARACTERISATION OF SUPERCONDUCTING Bi-2223/Ag TAPES WITH HIGH CRITICAL CURRENT DENSITIES IN KM LENGTHS : *Per Vase*[1]; [1]Nordic Superconductor Technologies, Priorparken 878, Broendby, Copenhagen 2605 Denmark

High critical current density, long length Bi-2223/Ag tapes are needed for large scale applications. In this paper we describe our recent result reaching critical current densities of 23 kA/sq.cm and engineering critical current densities of 5.2 kA/sq.cm at 77K over the whole length of the 1250 meters long Bi-2223/Ag tapes. To our knowledge this is the highest critical current density reported for Bi-2223/Ag tapes longer than 1 km. Detailed measurements of the critical current over the 1250 m long tape both by conventional four-probe method and by a specially developed continuous measurement of the remanent field by Hall probes will be reported. Various Ag alloy sheathed Bi-2223 tapes have been made by our standard production line. The approach for improving the homogeneity and the considerations of processing and handling the long length tape will be presented. Electrical, mechanical and thermal properties of our Ag alloy sheathed tapes, including Ag-Au alloy sheathed, will also be described.

HIGH TEMPERATURE COATINGS III: Overlay Coatings for Engine Applications

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, Center for Laser Applications, Tullahoma, TN 37388 USA

Tuesday AM Room: 19
March 2, 1999 Location: Convention Center

Session Chairs: John E. Morral, University of Connecticut, Institute of Mats. Sci., Storrs, CT 06268-3136 USA; Christoph Leyens, Oak Ridge National Laboratory, Metals & Ceramics Div., Oak Ridge, TN 37831-6156 USA

8:30 AM INVITED PAPER

SIGNIFICANCE OF BOND COAT OXIDATION FOR THERMAL BARRIER COATING LIFE: *W. J. Quadakkers*[1]; W. Stamm[2]; D. Clemens[1]; L. Singheiser[1]; [1]Forschungszentrum Julich, IWV-2, Julich 52425 FRG; [2]Siemens Power Generation, Mulheim FRG

In modern industrial gas turbines metallic construction materials are protected against the high combustion gas temperatures by yttria stabilized thermal barrier coatings (TBC's). The long term performance of these ceramic coatings requires excellent oxidation properties of the substrate material, because high growth rates and poor adherence of the oxide layers forming during service would lead to early failure of the ceramic TBC. Therefore the high temperature components are protected by an oxidation resistant coating, commonly of the NiCoCrAlY-type prior to applying the TBC. The choice of an optimum NiCoCrAlY bond coat for TBC's not only requires the definition of a suitable NiCoCrAlY composition but also consideration of the multiphase character of the coatings. Changes in phase distribution, determined by coating manufacturing parameters and heat treatment can significantly affect the oxidation properties of the NiCoCrAlY bond coats and consequently TBC life.

8:55 AM

DIFFUSION BARRIERS TO INCREASE THE OXIDATIVE LIFE OF OVERLAY COATINGS: *James A. Nesbitt*[1]; Jih-Fen Lei[2]; [1]NASA Lewis Research Center, Mats. Div., MS 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Army Research Center, NASA Lewis Research Center, MS 77-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Currently, most blades and vanes in the hottest section of aero gas turbine engines require some type of coating for oxidation protection. Newly developed single crystal superalloys have the mechanical potential to operate at even higher component temperatures. However, at these elevated temperatures, coating/substrate interdiffusion can shorten the protective life of the coating. Diffusion barriers between the coating and substrate are being examined to extend the protective life of the coating. A finite-difference diffusion model has been developed to predict the life enhancement due to use of total or partial diffusion barriers. This model simulates Al diffusion to the growing oxide scale as well as into the substrate. Coating failure is predicted when the Al concentration in the coating drops to a critical level. The diffusion model takes into account the reduced interdiffusion between the coating and substrate when a diffusion barrier is present. The diffusion model has been used to predict the effectiveness of diffusion barriers in extending the protective life of a NiAl overlay coating undergoing cyclic oxidation as well as that of a bond coat beneath a ceramic layer (i.e., a

TBC). Experimental results for alumina thin films deposited as diffusion barriers on superalloy substrates will also be presented.

9:15 AM

MICROSTRUCTURES RESULTING FROM COATING/SUPERALLOY INTERDIFFUSION: Fred Meisenkothen[1]; *John E. Morral*[1]; [1]University of Connecticut, Metall. and Mats. Eng., 97 N. Eagleville Rd., Storrs, CT 06269-3136 USA

When coatings and superalloys interdiffuse, there are a variety of microstructures that can form. An example will be given in which small changes in composition of an MCrAlY type coating on a Ni-Cr-Al superalloy can produce the formation of five different microstructures. The microstructures were discovered by combining experimental observations with computer simulations to form an "Interdiffusion Microstructure Map." Such maps can be used in coating design and to predict microstructural changes that will occur during coating service.

9:35 AM

KINETIC CONSIDERATIONS FOR MANUFACTURING A DIFFUSION NiAl COATING UNIFORMLY DOPED WITH A REACTIVE ELEMENT BY CHEMICAL VAPOR DEPOSITION: *W. Y. Lee*[1]; L. He[1]; J. D. Meyer[1]; G. Y. Kim[1]; [1]Stevens Institute of Technology, Dept. of Mats. Sci. and Eng., Castle Point on Hudson, Hoboken, NJ 07030 USA

Recent manufacturing advances in aluminizing by chemical vapor deposition (CVD) offer new processing opportunities to further improve the performance of diffusion NiAl and (Ni,Pt)Al coatings for advanced thermal barrier coating applications. In particular, the dynamic versatility of the CVD aluminzing process provides a potent avenue of uniformly incorporating a reactive element such as Hf in the NiAl coating matrix via proactive control of the concentration of the dopant's precursor in the gas phase. However, with the apparent lack of meaningful experimental data, considerable uncertainties exist to properly project the viability of the doping approach. In this presentation, the critical research issues associated with the doping concept will be discussed with emphasis on generating reliable and reproducible kinetic data and analyzing the kinetics of the doping process.

9:55 AM

ALUMINA/YSZ COMPOSITE COATINGS: D. W. Stollberg[1]; W. B. Carter[1]; *J. M. Hampikian*[1]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

Multi component coatings of alumina and yttria stabilized zirconia (YSZ) containing from zero percent alumina up to the eutectic composition have been deposited via liquid fuel combustion chemical vapor deposition (CVD) onto single crystal sapphire substrates. Aluminum acetylacetonate, and 2-ethylhexanoates of yttrium and zirconia were used as chemical coating precursors dissolved in toluene. The coatings will be incorporated into thermal barrier coatings (TBCs) as an interlayer between the metallic bond coat and the ceramic top coat in an effort to strengthen the bond coat/ceramic interface as measured by thermal fatigue testing. Nanoindentation measurements of the hardness and fracture toughness of the alumina/YSZ coatings will be reported.

10:15 AM BREAK

10:35 AM INVITED PAPER

HIGH ASPECT RATIO MICROSTRUCTURE- SUPPORTED SHROUD FOR A TURBINE BLADE: *K. W. Kelly*[1]; [1]Louisianna State University, Mech. Eng. Dept., 2502 CEBA, Baton Rouge, LA 70803 USA

A major component in the development of advanced gas turbine engines is the increase of turbine inlet temperatures. Associated with this drive for higher turbine inlet temperatures is the need for more effective blade cooling strategies. Current cooling technology relies primarily on a combination of internal cooling through serpentine ribbed-coolant passages that are integrally cast in the blades or film cooling where a coolant jet is injected through a series of coolant holes on the blade surfaces. In the proposed research, a new concept of significantly increasing turbine-blade heat transfer is proposed, and is based on electrodepositing a moderately dense array of microstructures (nickel or nickel-alloy) directly on the blade surface with a nickel-alloy shroud on top. Preliminary results, resulting from an ongoing research program at LSU funded by the Defense Advanced Research Project Agency (DARPA), have already demonstrated the feasibility of this approach. The manufacturing process to build this shroud will be described and the results from heat transfer tests which quantify its performance will be provided.

11:00 AM

EFFECTS OF PLATINUM ADDITIONS AND SULFUR CONTENT ON THE ADHESION OF ALUMINA SCALES TO CVD ALUMINIDE BOND COATS: *J. Allen Haynes*[1]; Ying Zhang[2]; Woo Y. Lee[3]; Bruce A. Pint[1]; Ian G. Wright[1]; Peter K. Liaw[2]; [1]Oak Ridge National Laboratory, P.O. Box 2008, M.S. 6063, Oak Ridge, TN 37831-6063 USA; [2]University of Tennessee, School of Eng., Knoxville, TN USA; [3]Stevens Institute of Technology, Castle Point on Hudson, Hoboken, NJ USA

The adhesion of alumina scales to aluminide bond coats is the life-limiting factor for some advanced thermal barrier coating systems. It has been demonstrated that scale adhesion on superalloys can be substantially improved by reducing S impurities; and scale adhesion on aluminide coatings is enhanced by Pt additions. This work investigated the effects of aluminide bond coat sulfur and platinum contents on alumina scale adhesion. Low-sulfur NiAl and NiPtAl bond coats were fabricated by aluminizing de-sulfurized René N5 using a unique low-sulfur chemical vapor deposition (CVD) process. Scale adhesion was investigated by isothermal and cyclic oxidation testing at 1150°C. Lowering the sulfur content of CVD NiAl significantly improved scale adhesion, but scale spallation eventually initiated along coating grain boundaries. Significant improvements in scale adhesion along bond coat grain boundaries were obtained after Pt additions, although sulfur levels were higher in CVD NiPtAl due to impurities in the electroplated Pt. The observed influences of Pt additions included (1) considerable reductions in scale-metal void growth, and (2) reductions in Ta-rich oxides in the scales above the bond coat grain boundaries. The influence of bond coat microstructure on scale adhesion will also be discussed.

11:20 AM

THERMOMECHANICAL FATIGUE OF A SINGLE CRYSTAL SUPERALLOY: INFLUENCE OF A PROTECTIVE COATING: *Alejandro Sanz*[1]; L. Llanes[3]; J.-P. Bernadou[2]; M. Anglada[3]; [1]Danieli Research and Development, Mats. Development, Via Nazionale 41, Buttrio 33042 Italy; [2]E.N.S.A.E., Laboratoire de Metallurgie, 10 Av. E. Belin, Toulouse, Cedex 31055 France; [3]U.P.C., ETSII, Depto. de Ciencia de Los Materiales y Metalurgia Av, Diagonal 647, Barcelona, 08028 Spain

The improvement of the gas turbine engine efficiency requires to have the highest possible Turbine Inlet Temperature (T.I.T.). Turbine's turbine blades and nozzle vanes are commonly protected against high temperature degradation with NiCoCrAlYTa coatings. A careful selection of the coating which offers sufficient chemical compatibility with the substrate and a low interdiffusivity is necessary for these high temperature systems. AM-3 single-crystals, [001] oriented, coated with a Low Pressure Plasma Spray (LPPS) NiCoArAlYTa coating were submitted to thermomechanical fatigue conditions until fracture. The thermomechanical fatigue test consisted of strain controlled cycle having four slopes and a duration of 180 seconds. The temperature range extended from 923ϒK (650ϒC) to 1373ϒK (1100ϒC). Two strain ranges of $\Delta\varepsilon_{max}/2$=0.5% and $\Delta\varepsilon_{max}/2$=0.25% were chosen for this study. This cycle schematically reproduces the strain evolution of the leading edge, blade's critical element in a civil turbine engine under normal working conditions. Using the data obtained from Energy Dispersive microprobe Spectroscopy (EDS) the diffusional phenomena between the coating and the substrate is characterised and correlated with the stress gradient and test temperature. Scanning Electron Microscopy (SEM) analysis were performed at each condition to establish the fracture mechanisms and the microstructural evolution, correlationing them to the presence of a NiCoCrAlYTa protective coating.

11:40 AM

REACTIVE PROCESSING OF A DENSE FUNCTIONALLY-GRADED INTERMETALLIC MATRIX COMPOSITE COATING: *Hexiang Zhu*[1]; Reza Abbaschian[1]; [1]University of Florida, Dept. of Mats. Sci. and Eng., 224 MAE, P.O.Box 116400, Gainesville, FL 32611 USA

A dense functionally-graded intermetallic matrix composite (FGIMC) coating on NiAl substrate has been successfully fabricated by reactive hot compaction technique. The FGIMC coating consisted of four NiAl/Al_2O_3 composite layers with alumina content varying from less than 3 vol.% to about 36 vol.%, with thickness of about 900μm. The microstructures of the coating and microhardness profiles across its thickness were characterized and compared to those of a one-layer NiAl -36 vol%Al_2O_3 composite coating on NiAl substrate. The comparison indicated that the bonding between FGIMC coating and NiAl substrate was stronger than that between single NiAl-36 vol.%Al_2O_3 composite coating and NiAl substrate. This is attributed to the reduction in residual stresses resulting from the more gradual composition transition in FGIMC coating.

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

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. and Eng., Madison, WI 53706-1595 USA; Ray Y. Lin, University of Cincinnati, Dept. of Mats. Sci. & Eng., Cincinnati, OH 45221-0012 USA

Tuesday AM Room: 14A
March 2, 1999 Location: Convention Center

Session Chairs: W. J. Boettinger, NIST Metallurgy Division, Gaithersburg, MD 20899 USA; R. Y. Lin, University of Cincinnati, Dept. of Mats. Sci. and Eng., Cincinnati, OH 45221-0012 USA

8:30 AM INVITED PAPER
MOTION BY CURVATURE AND IMPURITY DRAG: *J. W. Cahn*[1]; Amy Novick-Cohn[2]; [1]NIST, Mats. Sci. and Eng. Laboratory, Gaithersburg, MD 20899 USA; [2]Technion-IIT, Dept. of Mathematics, Haifa 32000 Israel

Diffuse interfaces between ordered domains (APB) provide a good testing ground for ideas about the interactions of solutes with moving interfaces. The free energy, thickness, and amount of adsorption can be manipulated experimentally over many orders of magnitude near critical temperatures for order-disorder transitions. Motion of such interfaces is by atomic diffusion on a common lattice, and does not require long range diffusion. We present a system of equations to model this diffusive motion and obtain a single equation for the predicted velocity that contains many factors. Interface motion is by curvature, and the velocity of motion is linear in surface free energy. However, this surface free energy factor is canceled by a term in the mobility in the limit of no adsorption, and the velocity of motion is approximately independent of the interfacial free energy and reduces to the result of Allen and Cahn. Adsorption lowers the velocity by a term that mimics what is expected for impurity drag. Near wetting transition with thick wetting layers the velocity becomes inversely proportional to their thickness. We examine the predictions near the tricritical point, and compare the results with experiments.

9:10 AM INVITED PAPER
THE THERMODYNAMICS OF INTERFACES FAR FROM EQUILIBRIUM: *Peter W. Voorhees*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

A general thermodynamical description of an evolving interface appropriate to situations far from equilibrium is developed. The theory represents a departure from theories based on classical nonequilibrium thermodynamics as we do not assume a linear relation between fluxes and forces, and we do not limit our theory to small departures from equilibrium. Two examples of this approach will be presented. The first deals with the effects of diffusion in the bulk phases, solute drag and interfacial diffusion on the thermodynamics of the interface. In the second the conditions setting the velocity of the facets that compose a fully faceted interface in an elastically stressed solid are derived. We find, for example, that the singularities in the elastic stress induced by the presence of corners do not influence the velocity of a facet.

9:50 AM BREAK

10:00 AM INVITED PAPER
PHASE FIELD MODELING OF ALLOY SOLIDIFICATION: *James A. Warren*[1]; *William J. Boettinger*[1]; [1]NIST, Metall. Division, Bldg 223/B164, Gaithersburg, MD 20899 USA

The phase field method has been used with considerable success over the past five years to model solidification phenomena. This method starts from thermodynamic principles employing gradient energies and adds reasonable kinetic postulates to yield differential equations that govern the evolution of the solidification microstructure, without explicitly tracking the interface position. These equations can, under appropriate initial conditions, describe processes such as solute trapping, solute drag, Mullins-Sekerka instabilities, cellular/dendritic growth, and dendrite sidearm coarsening, fragmentation and bridging. The microsegregation patterns associated with these phenomena can be predicted and characterized. Both two and three dimensional simulations will be discussed. A recent modification of the approach permits the modeling of the solidification of grains with different orientations.

10:40 AM INVITED PAPER
SOLUTE DRAG IN ALLOY SOLIDIFICATION: *Michael J. Aziz*[1]; [1]Harvard University, Div. Eng. & Applied Sci., 29 Oxford St., Cambridge, MA 02138 USA

Several models for the kinetics of interface motion in binary alloys are based on the pioneering work of Hillert and Sundman. Some find a solute drag effect, as suggested by Hillert and Sundman for the migration of interphase boundaries, whereas others have not. Some of these models have been applied to binary alloy solidification and tested experimentally. The implications of the experimental results for kinetic models of interface motion will be discussed.

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Interfacial Reaction

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 Corp, San Jose, CA 95134 USA; Ephraim Suhir, Bell Labs., Murray Hill, NJ 07974 USA

Tuesday AM Room: 17A
March 2, 1999 Location: Convention Center

Session Chairs: K. N. Tu, University of California, Dept. of Mats. Sci. and Eng., Los Angeles, CA 90095, USA; K. N. Subramanian, Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA

8:30 AM INVITED PAPER
AN IMPROVED NUMERICAL METHOD FOR PREDICTING INTERMETALLIC LAYER THICKNESS DEVELOPED DURING THE

FORMATION OF SOLDER JOINTS ON Cu SUBSTRATES: S. Chada[1]; *R. A. Fournelle*[1]; D. Shangguan[2]; [1]Marquette University, Mats. Sci. Program, P.O. Box 1881, Milwaukee, WI 53201-1881 USA; [2]Ford Motor Company, Visteon Automotive Systems, 17000 Rotunda Drive, Dearborn, MI 48121 USA

An improved numerical method has been developed for calculating the thickness of intermetallic layers formed between Cu substrates and solders during the soldering process. The improved method takes into account intermetallic dissolution during heating and intermetallic precipitation during cooling and requires as input (1) the temperature-time profile for the soldering process, (2) the experimentally determined isothermal growth parameters for the growth of the intermetallic layer into Cu saturated molten solder, (3) the experimentally determined Nernst-Brunner parameters for the dissolution of Cu into molten solder, (4) the experimentally determined solubility of Cu in molten solder and (5) assumptions about the thickness of the boundary layer in the liquid ahead of the growing intermetallic. Calculations show that the improved method predicts intermetallic growth between Cu substrates and 96.5Sn-3.5Ag and 62Sn-36Pb-2Ag solders during reflow soldering better than a previously developed method, which did not take into account dissolution during heating and precipitation during cooling. Calculations further show that dissolution has a significant effect on intermetallic growth, while precipitation does not.

8:55 AM INVITED PAPER

INTERFACIAL REACTIONS IN THE Ag-Sn/Cu COUPLES: Yee-wen Chen[1]; *Sinn-wen Chen*[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., Kuang-Fu Rd., Hsinchu 30043 Taiwan

Two types of diffusion couples, Sn-3.5 wt.%Ag/Cu and Sn-25.0 wt.%Ag/Cu, have been studied. The phases formed at the interfaces and their thickness have been examined. The reaction temperatures were at 240°C and 450°C, and the reaction time varied from 5 minutes to 72 hours. At 240°C, only Cu_3Sn and Cu_6Sn_5 phases are formed. At 450°C, Cu_4Sn, Cu_3Sn, and Cu_6Sn_5 phases formed in the Sn-3.5 wt.%Ag/Cu couple; while Cu_4Sn, Cu_3Sn, Cu_6Sn_5, and Ag_3Sn phases formed in the Sn-25 wt.%Ag/Cu couple. The interfaces between the molten solder and the solid intermetallic phases had a wavy morphology; while those between the solid intermetallic phases and the solid Cu substrate were relatively planar. The isothermal sections of the Ag-Sn-Cu system at 240°C and 450°C have been assessed. The interfacial reaction paths are proposed. It is concluded that the Cu_6Sn_5 and Ag_3Sn phases formed during solidification not by interfacial reactions.

9:20 AM

INTERFACIAL REACTION BETWEEN Ni, Pd and Sn-Bi AND Sn-Ag EUTECTIC SOLDERS: *G. Ghosh*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Metallization schemes using Ni and Pd are becoming increasingly popular in electronic packaging. Both two-layer and four-layer Ni-Pd coatings are being used for soldering of various components using Pb-Sn solders. However, there is a growing interest in the use Pb-free solders. This paper deals with a systematic study of interfacial reaction between bulk Ni and Pd substrates and Sn-Bi and Sn-Ag eutectic solders. The evolution of interfacial microstructures due to both liquid- and solid-state reactions will be presented. The products of interfacial reaction are characterized by SEM, TEM and AEM. The microstructural evolution at the interface and the diffusion path will be discussed in terms of the calculated isothermal sections of the corresponding ternary system at the experimental temperature of interest.

9:40 AM

CHARACTERIZATION OF THE GROWTH OF INTERMETALLIC INTERFACIAL LAYERS OF Sn-Ag and Sn-Pb EUTECTIC SOLDERS AND THEIR COMPOSITE SOLDERS ON Cu SUBSTRATE DURING ISOTHERMAL LONG-TERM AGING: *S. Choi*[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Mats. Sci. & Mech., Eng. Bldg. # 3536, East Lansing, MI 48824-1226 USA

The intermetallic layer formation between solder and substrate indicates a firm bonding of solder material with substrate. However, the solid state growth of intermetallic interfacial layers have been known to adversely affect the mechanical properties and reliability of solder joints, leading to fracture at or near the interfacial layer. Single shear lap joints were made with four different solders, Sn-Pb and Sn-Ag eutectic solders, and their composites containing about 20 vol% in-situ Cu_6Sn_5 intermetallic phases about 3-8 micrometers in diameter. Two sets of experiments were performed: First, all of the above four solder joints were aged at 150°C for periods ranging to 4000 hours and the intermetallic growth was monitored periodically. Second, each of the above four solder joints was aged at five different temperatures for 4000 hours. The interfacial layers between solders and the Cu substrate were examined using optical microscopy and scanning electron microscopy. The kinetics of growth of intermetallic interfacial layers formed between solders and Cu substrate was characterized. Effects of in-situ Cu_6Sn_5 intermetallic phases on the growth rate were also characterized. The intermetallic layers grew much slower in the composite solders for the first 350 hours for the Sn-Pb composite and for the first 1200 hours for the Sn-Ag composite as compared to the corresponding non-composite solders. Thereafter, the growth rates were more similar, and the thickness became nominally similar in the 150ϒC specimen after about 4000 hours, when the reinforcements were also quite large.

10:00 AM INVITED PAPER

Pb-FREE SURFACE-FINISH ON ELECTRONIC COMPONENT TERMINALS FOR Pb-FREE SOLDER ASSEMBLY: H. Tanaka[1]; M. Tanimoto[1]; A. Matsuda[1]; *S. Shiga*[1]; [1]Furukawa Electric Co., Ltd., R&D Division, Metal Research Center, 500 Kiyotaki, Nikko 321-0942 Japan

Terminals of electronic components such as IC's, connectors and condensors are presently surface-finished with Sn-Pb alloys in general in order to keep solderability without Sn-whisker occurrence. NCMS's Pb-free Solder Project reports that Sn-Pb alloys have some disastrous effects on the melting behaviors in case of Pb-free solder assembly, some of which are due to the formation of low melting phases that may generate when Pb-free solders with Bi and/or In are mixed with Sn-Pb coatings. In the final report published in Aug. 1997, written is that the much larger topic of eliminating Pb from solderable surface finishes will need to be addressed before Pb-free alloys are implemented. In Japan, developed were two types of Pb-free solderable finishes, one of which is simple Ni/Pd double layer mostly IC lead frames, while Au-flash can be top-coated in order to enhance solderability. Although showing a variety of benefits, both technical and economical, this system have some retarding aspects of Pd's supply and price situation along with hazardous corrosion characteristics in case of its application to ferrous base metals such as Fe-Ni alloys. Different from the precious metal system, the second system consists of two layers, thicker Sn underlayer and thin Sn-Bi alloy over layer, which are electroplated in succession. In comparison with the monolayer of Sn-Bi which reacts hazardously with base metals such as copper, this double layer system shows superior performances similar to the conventional Sn-Pb with regard to wetting time and temperature, shelf life of solderability, whisker occurrence and so on. This is due to the surface localization of a small amount of Bi. Along with diffusion rates and metallographies, some practical usages and related data will be presented.

10:25 AM BREAK

10:40 AM INVITED PAPER

INTERACTION BETWEEN COPPER AND AN EUTECTIC Pb-Sn/HIGH Pb COMPOSITE SOLDER FOR MICROELECTRONIC PACKAGING : *A. S. Zuruzi*[1]; C. Chiu[1]; K. M. Chua[2]; W. T. Chen[1]; S. K. Lahiri[1]; [1]National University of Singapore, Microelectronics Materials, Processes and Packaging Programme, Blk S7, Level 3 119 260 Singapore; [2]Gintic Institute of Manufacturing Technology, 71 Nanyang Dr. Singapore

Use of composite solders comprising eutectic Pb-Sn and higher lead solders is an option for joining chips to substrates at lower temperatures. In this work the reaction between such composite solders and copper, which results in Cu-Sn intermetallic compound formation, during solder reflow processes was investigated for both flip chip and BGA types of applications. The thickness of the intermetallic compound was found to increase with the square root of the number of reflows, which suggests that the compound growth is dominated by a diffusion controlled mechanism. This result is similar to observations made in constant temperature annealing studies. The similarity leads to a simple method to assess

how the compound growth rate is affected by the temperature-time profile.

11:05 AM
DISSOLUTION AND REACTION KINETICS OF THE SOLDER-BALL PADS IN BGA DURING REFLOW SOLDERING: *C. R. Kao*[1]; J. A. Ho[1]; [1]National Central University, Dept. of Chem. Eng., Chungli Taiwan

The solder-ball pads in a Ball-Grid-Array (BGA) package are made up of several metal layers. The Au/Ni/Cu tri-layer structure is the most common pad metallization. In this structure, the first layer which is to be in direct contact with solder is the Au layer, which has a thickness of about 1 micron. Below this Au layer is the Ni layer, whose thickness is about 3 microns. The Cu layer is part of the internal wiring within the BGA package, and its thickness is often greater than 20 microns. During reflow soldering, the Au layer dissolves into the solder rather quickly, and the Ni layer is then exposed to the solder. In this study, we report the dissolution kinetics of the Au layer and the reaction kinetics of Ni layer with solder in commercial BGA substrates. The spatial distribution of the dissolved Au atoms in solder is also determined. These information are helpful in determining the optimal reflow temperature profile.

11:25 AM
INTERFACIAL MICROSTRUCTURE EVOLUTION OF Pb-Sn SOLDER JOINTS ON Ni/Pd/Cu METALLIZATIONS: *G. Ghosh*[1]; [1]Northwestern University, Dept. Mats. Sci. and Eng., 2225 N.Campus Dr., Evanston, IL USA 60208-3108

This paper will examine the interfacial microstructure of the Pb-Sn solder joints on Ni/Pd/Cu metallizations. Both liquid-state and solid-state reactions have been investigated using the eutectic (62Sn38Pb) and a high-lead (95Pb5Sn) solders. A two-layer (Pd/Ni) and a four-layer (Pd/Ni/ni-Pd/Ni) metallization schemes on Cu are used as substrates. The interfacial microstructures have been characterized using a variety of electron microscopy techniques, such as SEM, TEM and AEM. The formation of voids at interface and the complex interfacial microstructures due to interdiffusion and phase transformation will be discussed in detail.

11:45 AM
ANALYSIS OF THE UPPER PEAK OF WETTING FORCE-TIME CURVE USING Sn-37Pb, Sn-3.5Ag SOLDERS: *Jae Yong Park*[1]; Jae Pil Jung[2]; Choon Sik Kang[1]; [1]Seoul National University, Dept. of Mats. Sci. and Eng., San 56-1, Shillim-dong, Kwanak-Ku, Seoul 151-742 Korea; [2]University of Seoul, Dept. of Mats. Sci. and Eng., 90, Jeonnong-dong, Dondaemun-Ku, Seoul 130-743 Korea

The meniscograph/wetting curve is a very useful tool for quantifying the wettability of solder, but its meaning and mechanism have not been fully developed yet, especially the upper peak of the wetting curve which can be shown when Cu plate is detached from solder bath. To evaluate this peak, two kinds of experiments were performed: 1) Cu plates from which two adjacent corners are cut out in various sizes of squares were used to compare with the regular rectangular Cu plate; 2) the immersion depth of the regular rectangular Cu plate to be dipped into solder bath varied from 0.5mm to 9.5mm. Sn-37Pb and Sn-3.5Ag solders were used for both experiments. In the case of the first experiment using cut out plates, the upper peak of the wetting curve changed to a rather round shape and a very short horizontal segment appeared after the peak, followed by a downfall. This horizontal segment represents the point of transition where the sliding solder reached the edge of the cut out Cu plate. According to the second experiment, the amount of time needed for the curve to reach the peak increases in proportion to the immersion depth of Cu plate while the span of time taken for the drop of the curve remained independent of the depth. The force value on the horizontally steady line of the wetting curve was inversely proportional to the immersion depth while the force value on the pinnacle of the upper peak remained constant. This means that the latter is influenced by the buoyancy force while the former is not. It can be concluded from the results that: (1) ascending segment of the upper peak represents the sliding of solder on Cu plate; (2) the pinnacle of the upper peak stands for the state in which the sliding solder meets the edge of the Cu plate.

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Twinning in Electronic Materials

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

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

Tuesday AM — Room: 17B
March 2, 1999 — Location: Convention Center

Session Chairs: Bhakta B. Rath, Naval Research Laboratory, MS & CT Directorate, Washington, D.C. 20375-5320 USA; David O. Welch, Brookhaven National Laboratory, Mats. Sci. Div., P.O. Box 5000, NY 11973-5000 USA

8:30 AM INVITED PAPER
STACKING FAULTS AND TWIN GENERATED ANTI PHASE DOMAINS IN SEMICONDUCTOR HETEROSTRUCTURES: *Aristos Christou*[1]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., Bldg. 090, Room 2135, Stadium Dr., College Park, MD 20742-2115 USA

The formation of antiphase domains may be ideally investigated through the growth of a polar on non-polar semiconductor such as GaAs on silicon. Such investigations have been carried out and we now have a clear model on the generation of APD as a function of interfacial strain at the interfaces of such semiconductors. Through transmission electron microscopy, initial misfit strain is usually accommodated through the generated or initiated at stacking faults or twins which proceed to enlarge as the growth process continues. The APB boundary is therefore a twinned of SF boundary leading to eventual three dimensional growth. We have isolated single SF randomly distributed and not at all affected by the distribution of misfit dislocations. These SF grow into twin boundaries as the growth process continues and are affected directly by the specific growth conditions. Electrically, film mobility is greatly reduced due to the presence of APDs, and the degree of reduction depends on compensating impurities. Twins in lattice matched heterosystems are also presented. In such samples, twins are always initiated at growth abnormalities close to the interfaces of systems such as the InGaAs/InP configuration. The twin density may be affected again by growth kinetics.

9:05 AM INVITED PAPER
THE PHENOMENOLOGY AND THERMODYNAMICS OF THE STRUCTURE OF TWINS AND TWIN BOUNDARIES IN $YBa_2Cu_3O_x$ SUPERCONDUCTORS*: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Mats. Sci. Division, Bldg. 480, P.O. Box 5000, Upton, NY 11973-5000 USA

Twins and twin boundaries play some important roles in the high-T_c superconductor $YBa_2Cu_3O_x$. Among other things, twin boundaries serve as pinning centers for magnetic flux and as diffusion paths for oxygen. The organization of the twin structure serves to relieve elastic strain in confined microstructures such as in superconducting films on substrates and in composite conductors. In this paper, I will review what is known about the local atomic structure and composition of twin boundaries and their dependence on oxygen content and alloying additions, together with the systematics of the dependence of twin density on grain size, oxygen content, and alloying additions. I will then discuss thermodynamic and atomistic models which can be used to describe the compositional and strain distributions near twin boundaries, as well as their effect on the density of electronic holes, which plays a vital role in several superconducting properties. *This research was supported by the U.S.

Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

9:40 AM INVITED PAPER

TWINNING IN $YBa_2Cu_3O_{7-x}$ SUPERCONDUCTORS: *Yimei Zhu*[1]; [1]Brookhaven National Laboratory, Dept. of Applied Sci., Bldg. 480, Upton, NY 11790 USA

The most prominent structural defects in YBa2Cu3O7-x (x<0.5) are twins. Below about 750ϒC, YBa2Cu3O7-x undergoes a tetragonal-to-orthorhombic structural phase transition, resulting in twinning on the {110} planes. Twins are formed to reduce strain energy due to the change in shape and volume resulting from the phase transformation. The spacing of the twin lamella is determined by minimizing the total energy associated with the strain energy at the grain boundaries where the twins terminate and the interfacial energy of the twin boundary. High-resolution electron microscopy and electron energy-loss spectroscopy studies suggest that there are two types of twin boundaries in YBa2Cu3O7-x: one with a lattice translation (~d110) along the boundary for fully oxygenated samples (x~0.0), and the other, without a lattice translation, for oxygen-deficient ones. Electron diffraction and fringe analysis of the twin boundaries show that the former is an alpha-delta interface, while the latter is a pure delta-inter face. In-situ experiments indicate that the structure of the twin boundaries can change from one type to the other. This structural transition was analyzed using the observed twinning dislocations and steps within the framework of a modified coincidence-site-lattice model. Work supported by the U.S. DOE DE-AC02-98CH10886.

10:15 AM BREAK

10:25 AM INVITED PAPER

DOMAINS IN FERROELECTRIC THIN FILMS AND HETERO-STRUCTURES: *Ramamoorthy Ramesh*[1]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., Bldg. 090, Rm. 2135, Stadium Dr., College Park, MD 20742-2115 USA

Ferroelectric materials undergo a phase transition from the paraelectric (high temperature) to the ferroelectric (low temperature) phase, accompanied by a change in crystal structure and symmetry. The strain accompanying these structural and electric phase transformations is generally accommodated by the formation of domains, that are separated by domain walls. Due to the fact that the anisotropy in these materials is rather high and the exchange interaction rather weak, these domain walls are very thin (of the order of a few atomic spacings). A typical case is that of the cubic-tetragonal transformation in the lead zirconate titanate (PZT) system, accompanied by the formation of 90 degree domains. Domain wall in ferroelectrics can move under the influence of electric or stress fields (ferroelastic). Since these domain walls can exist in thin films of ferroelectric materials, that are likely to be used in non-volitale memories, piezoelectric actuators and pyroelectric sensors, it is important to understand the statics and dynamics of domain evolution in them. In these presentation, I will review some of the work on-going in our program that is aimed at establishing the structure-property-processing interrelationships relevant to domain formation in ferroelectric thin films.

11:00 AM

RELAXATION OF COHERENCY STRAIN VIA TWINNING IN Ge-C AND Ge-Si-C EPITAXIAL THIN FILMS: *Mohan Krishnamurthy*[1]; Bi-Ke Yang[2]; *Jong K. Lee*[1]; [1]Michigan Technological University, Metall. and Mats. Eng., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]IRF, Inc., Temecula, CA USA

In thin films, plastic relaxation of coherency strain due to a lattice mismatch is accommodated typically by misfitting and/or threading dislocations. There is, however, a strong indication that coherency strain is also relaxed through twinning in Ge1-xCx and Ge0.8-xSi0.2Cx (nominal x < 0.1) films grown epitaxially on Si(100) subatrates. Low temperature (~200ϒC) molecular beam epitaxy can engineer the immiscible Ge-C system to form a dilute binary with substitutional C fractions typically ~ 1-2 atomic%. X-ray and transmission electron microscopy analyses of such epitaxial films indicates that the presence of C diminishes the role of dislocations and increases the tendency for the formation of stacking faults and twins. The density of twins increases with increasing C concentrations and, in the case of GeSiC films, increasing Si fractions. Details of the experimental results as well as a theoretical model based on the discrete atom method will be discussed for the role of twinning in such epitaxial thin films.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Alloys and Properties

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

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

Tuesday AM Room: 8
March 2, 1999 Location: Convention Center

Session Chairs: MIchael H. Loretto, The University of Birmingham, IRC, Birmingahm, West Midlands B15 2TT UK; Donald L. Anton, United Technologies Research Center, MS-129-22, 411 Silver Lane, East Hartford CT 06108 USA

8:30 AM INVITED PAPER

ALLOY AND PROCESS IMPROVEMENTS FOR CAST GAMMA TIAL ALLOY APPLICATIONS: *Paul A. McQuay*[1]; [1]Howmet Research Corporation, Advanced Technology, 1500 South Warner St., Whitehall, MI 49461-1895 USA

Recent cast alloy development efforts have focused on the improvement of creep resistance through alloying and micro-alloying additions. Specifically, micro-alloying additions of carbon have been shown to reduce the time to 0.2% primary creep strain by up to an order of magnitude, while only slightly reducing the room temperature ductility. Additionally, the Howmet Corporation has developed several net or near-net shape casting processes which produce components which meet industry requirements for metallurgical integrity and performance. Using these processes, component development efforts have focused on diesel engine turbocharger wheels, and small structural and airfoil components for turbine engines. The status of these manufacturing technologies, microstructure and property relationships of selected alloys, and examples of near-term production opportunities will be reviewed and discussed.

9:00 AM

STUDY OF MICROSTRUCTURE TO IMPROVE CREEP PROPERTIES OF CAST TiAl-Fe-V-B ALLOY: *Sadao Nishikiori*[1]; Kenji Matsuda[1]; [1]Ishikawajima-Harima Heavy Industries Co., Ltd., Mat. Tech. Dept., 3-1-15, Kotou-ku, Tokyo 135 Japan

A gamma titanium aluminide, Ti-46.7Al-Fe-V-B with duplex structure, has been developed as a casting material for high temperature structural use in aero-engines. This alloy is characterized by the addition of Fe and V which improve castability. In order to apply this alloy to various rotating parts operated at temperatures up to 750ϒC, further improvements of creep strength will be required. In general, it is widely known that lamella structure, coarse grains and/or increase of degree of serration at grain boundaries have the advantage of improving the creep strength. So, viewed in these metallographical behavior, the possibility of modifying microstructure in TiAl-Fe-V-B system was investigated. Then, as compared with creep properties results, effects of alloying elements and heat treatment process were estimated with metallografic characterization.

9:20 AM

MECHANISMS OF LAMELLAR REFINEMENT DURING PRIMARY CREEP OF NEAR - GAMMA DUPLEX TiAl: *Thomas R. Bieler*[1]; Dong Yi Seo[1]; [1]Michigan State University, Dept. of Mats. Sci. and Eng., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Primary creep has been measured in a number of near-gamma TiAl alloys with duplex microstructures. Two stages of primary creep deformation occur, an early and rapid process that exhausts itself after about 0.2-0.5% strain, and a different process that dominates the deformation up through the minimum creep rate. When creep data are fit to functions that are complex enough to accurately represent the measured strain-time data, these two stages are easily observed in a strain-rate vs. strain (or time) plot. From interrupted creep tests, the early process causes lamellar refinement, i.e. a reduction in lamellar spacing, and the second process does not alter the lamellar spacing significantly. The primary creep resistance is dominated by the rate at which the early process is exhausted. Since lamellar refinement is a metric of this early process, simulations of the refinement process have been made to examine possible mechanisms that could account for the observed refinement. The simulation is made by starting with a set of lamellar spacings that represent the undeformed lamellar spacing frequency distribution. The thickest lamellae are subdivided in a manner that changes the frequency distribution to match the deformed distribution by assuming that twins and/or strain induced gamma-alpha2 phase transformations occur parallel to lamellar planes. The effects of lamellar spacing bin size, and the relative fractions of twinning or gamma-alpha2 shear transformations are examined to consider how different deformation histories affect the resulting deformed distribution, and the resulting local strains in the lamellar microstructure are computed.

9:40 AM

MICROSTRUCTURAL EFFECTS ON THE CREEP, LOW CYCLE FATIGUE AND CRACK PROPAGATION BEHAVIORS OF Ti-47Al-2W-0.5Si: *Valentino Lupinc*[1]; Massimo Marchionni[1]; Mohamed Nazmy[2]; Giovanni Onofrio[1]; Mark Staubli[2]; [1]CNR-TEMPE, Via Cozzi 53, Milan 20125 Italy; [2]ABB Power Generation, KWTM.D, Baden, CH-5401 Switzerland

In the present investigation, the effect of microstructure on the tensile, creep, low cycle fatigue and fatigue crack properties is studied in the cast and HIP'ed Ti~47Al-2W-0.5Si alloy. Two heat treatment schedules were applied to produce two different types of microstructures, i.e. the duplex (globular gamma and lamellar alfa2/gamma) and the nearly lamellar types. The tensile strength and creep behaviour of this alloy, in the temperature range of 700-850ϒC, have been determined and correlated to the corresponding microstructures. In addition, the low cycle fatigue and fatigue crack propagation behaviours in this alloy have been studied at different temperatures. The results on the creep behaviour showed that the alloy with lamellar microstructure has an improved creep strength as compared with that of the duplex microstructure. The static and dynamic mechanical properties of this alloy were also compared with those of nickel base superalloys.

10:00 AM

K5 WROUGHT TiAl ALLOYS: DESIGN, PROCESSING, AND PROPERTIES: *Young-Won (Y-W.) Kim*[1]; [1]UES, 4401 Dayton-Xenia Rd., Dayton, OH 45431 USA

Significant improvements in tensile properties in wrought gamma TiAl K5 alloys have been made through controlling lamellar grain-size (GS) and spacing (LS) and grain-boundary (GB) morphology. GS refinement has been achieved by several methods, including: small boron additions, novel processing routes, two-phase-field heat treatments, and the combinations. LS was varied by controlling cooling-rate and scheme which are often chemistry- or processing-specific. The microstructural refinements were found to also enhance creep-resistance, fatigue-strength and damage-tolerance at temperatures below BDTT; however, at a certain expense of higher-temperature deformation/fracture resistance. LS refinement tends to reduce GB serration thereby lowering RT ductility and fracture resistance. Recently, progresses have been made in reducing the refinement-induced deficiencies through chemistry modification and/or microalloying and process/heat-treatment control. This progress report: 1) discusses designing K5 alloys based on the experimental findings and fundamental knowledge; 2) evaluates K5-series alloys with specific property data set; and 3) assesses the issues for further improvements.

10:30 AM INVITED PAPER

EFFECTS OF MINOR ALLOYING ADDITIONS ON MIROSTRUCTURES AND MECHANICAL PROPERTIES OF LAMELLAR TiAl ALLOYS: *C. T. Liu*[1]; P. J. Maziasz[1]; D. J. Larson[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

This paper provides a comprehensive review of our recent work on the effects of minor alloying additions on lamellar structures and mechanical properties of dual-phase TiAl alloys. The base alloy Ti-47Al-2Cr-2Nb (at. %) was alloyed with equal and less 1% of B, W, Mo, and/or Ta, and all the alloys were prepared by powder and ingot metallurgy. The microstructural features were controlled by hot-extrusion temperature, and mechanical properties were evaluated by tensile testing at temperatures to 1000ϒC. Microstructural features, including colony size, interlamellar spacing, interfacial segregation and particle composition, were characterized by optical microscopy, electron microprobe analyses, transmission electron microscopy, and atom probe field ion analyses. The Hall-Petch equations are found to be applicable to both the yield strength and tensile ductility at room and elevated temperatures. Colony size is a major parameter controlling the tensile ductility, and the yield strength is solely controlled by interlamellar spacing. The strengthening effect of minor alloying additions essentially comes from their refinement of lamellar spacing, rather than from a classic solute hardening effect.

11:00 AM

THE EFFECT OF COMPOSITION ON THE STABILITY AT 700°C AND ON THE MECHANICAL PROPERTIES OF GAMMA TITANIUM ALUMINIDES: *Richard Raymond Botten*[1]; Alastair Bryan Godfrey[1]; Michael H. Loretto[1]; [1]University of Birmingham, IRC in Materials for High Performance Applications, Edgbaston, Birmingham, West Midlands B15 2TT UK

Recent research has shown that the fully lamellar microstructure is not thermally stable at its predicted use temperature (700ϒC). Decomposition of the alpha-2 laths occurs, along with the formation of precipitates rich in alloying elements. The current research concentrates on two alloys one based on the Ti-47at.%Al system with low alloying additions, 1-2at.% of each individual alloying element, and on the Ti-44at.%Al system with higher alloying additions, 4-8at.% of each individual alloying element. Both alloys have been heat treated to produce the fully lamellar microstructure and then, exposed in air at 700ϒC for 3000h. Microstructural changes have been investigated using scanning and transmission electron microscopy. Mechanical properties have been assessed in terms of room temperature tensile properties and creep rates at 700ϒC under a stress of 200MPa.

11:20 AM

CREEP OF GAMMA TITANIUM ALUMINIDES: ENGINEERING OBSERVATIONS AND REQUIREMENTS FOR COMPONENTS: *Ian J. Perrin*[1]; [1]Mechanical Engineering Centre, GEC ALSTHOM, Cambridge Rd., Whetstone, Keicester Le8 6LH UK

Alloys based on the gamma titanium aluminide intermetallic compound have a combination of properties which offer many benefits for low pressure turbine blades of industrial gas turbines. These benefits are discussed and it is identified that creep strength is particularly important for such components which are required to operate at high loads for long periods of time. These creep strength requirements are assessed using simple models. This enables absolute strength requirements to be defined and identifies the most crucial aspects of the creep response. This information should help to guide future alloy development. In addition, the creep response of gamma-TiAl alloys is reviewed from an engineering perspective. Features discussed include basic behaviour (under both uniaxial and multiaxial states of stress), creep models and life assessment methods. The various concepts and methods are illustrated with typical data for a gamma-TiAl alloy.

11:40 AM

IMPROVEMENT OF METALLURGICAL PROCESSING PARAMETERS FOR EPM TiAl-1.4Mn-2Mo ALLOY: J. K. Kim[1]; *Sun-Keun*

Hwang[1]; S. W. Nam[2]; N. J. Kim[3]; [1]Inha University, Metall. Eng., 253 Yonghyeon-Dong, Nam-Gu, Incheon 402-751 Korea; [2]KAIST, 373-1 GuSeong-Dong, Daejeon, ChungNam 305-701 Korea; [3]POSTECH, Mt. 31, HyoJa-Dong, Pohang, GyungBug 709-784 Korea

Laboratory alloy TiAl-1.4Mn-2Mo was synthesized by elemental powder Metallurgy (EPM) and the tensile properties and the creep resistance were evaluated. The mechanical properties were comparable to or better than other gamma alloys made by ingot metallurgy. In the EPM processing of the laboratory alloy, it was important to select a fine Mo powder and to conduct a proper pre-hot extrusion treatment in order to ensure minimal porosity and uniform microstructure. Controlling the Mo powder size was necessary because of concerns on Kirkendall porosity and ¥â-phase formation. Pre-hot extrusion treatment was required to stabilize intermediate phases such as Ti2MoAl and TiAl3. Various post-extrusion heat treatments were devised to refine the final lamellar microstructure to a range of 30§≤ in packet size. Overall the present work supports the viability of the PM approach in developing gamma alloys.

12:00 PM

PROPERTIES OF FORGED GAMMA-TiAl COMPRESSOR BLADES: *Thomas Haubold*[1]; *Dan Fagaraseanu*[1]; [1]BMW Rolls-Royce GmbH, Mats. Tech., Hohemarkstr. 60-70, Oberursel, Hessen 61440 Germany

TiAl compressor blades and vanes in aeroengines would offer a potential for weight saving and increased work temperature above superalloys commonly used for this application (Inco718). Extrusion and isothermal forging has been investigated as processing routes for manufacturing. The blade life depend mainly on material fatigue behaviour, the static and dynamic stress fields in the blade, the load history and environment. Hence main properties for component design such as fatigue behaviour (HCF/LCF), crack growth, tensile and creep properties have been tested for different processing routes for temperatures up to 700ϒC. The fracture surface and microstructure were characterised by light and scanning electron microscope analysis. Component vibrations test are planned to prove the blade resonant mode behaviour and correlate the results with material test data. This research has been supported by BMBF (MaTech) 03N 3025

TUESDAY AM

LEACHING THEORY PROCESS DEVELOPMENT & INDUSTRIAL PRACTICE: Copper 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

Tuesday AM Room: 1B
March 2, 1999 Location: Convention Center

Session Chairs: Dr. A. Alfantazi, Falconbridge, Ltd Falconbridge Technology Centre, Falconbridge, Ontario P0M 1S0 Canada.; Dr. G. Demopoulos, McGill University

8:30 AM

AMMONIA LEACHING OF COPPER CONCENTRATES - COMMERCIAL APPLICATION: *Nathaniel Arbiter*[1]; [1]Nathaniel Arbiter Associates, Inc., 6300 S. Upper Valley Rd., Vail, AZ 85641 USA

First commercial plants for ammonia leaching (Alaska-copper carbonate ores; Michigan-native copper tailings) were followed later by development of flowsheets for sulphide concentrate leaching. Two of these resulted in full scale plants. 1) Annconda's Arbor Plant started up in 1974 with a design capacity of 35,000 tons/year of cathodes, produce by ammonia leaching (AL) with oxygen, followed by solvent extraction (SX) and electrowinning (EW). This shut down in 1977 after experiencing operating problems due to sulphate disposal as gypsum. 2) BHP's Coloso plant in Chile leached part of Eacondida's concentrates, using AL/SX/EW but with $O_{2/}$ from air to avoid mulphate production. Starting up in late 1994, it shut down in mid-1998, after failing to reach design capacity and experiencing problems with its technology. This paper will review the flowsheets, suggesting changes to improve metallurgy. Disposal of ammonium sulphate produced by sulphide oxidation was and is critical. Alternatives are to find markers for it; or to use line for precipitating $CaSo_4$, and recovering ammonia, other alternatives will also be discussed.

8:50 AM

COMPUTATION OF AQUEOUS SPECIES AND ACTIVITIES AND SOLUTION EQUILIBRIA IN HEAP LEACHING OF A LOW-GRADE SULPHURIC COPPER ORES: *Tero Kolhinen*[1]; Heikki Jalkanen[1]; [1]Helsinki University of Technology, Laboratory of Metallurgy, Vuorimiehentie 2, Espoo, P.O. Box 6200, Espoo FIN-02015 Finland

The aim of the study was to establish a thermodynamically consistent route for heap leaching of sulphide ore of copper using ferric iron as a leaching agent. The computer program, CHEMSAGE, was used for predicting the equilibrium behaviour of the leach solution. Pitzer's model was included to account for the expected non-ideal model. This model is a virial coefficient expansion of the Debye-Hückel equation and is considered suitable for the high ionic strengths chracterestic of heap solutions. The leach solution was modelled using heat capacity functions for aqueous species. Pitzer model parameters were taken from the literature or estimated from solubility data for sulphates. Gradual dissolution of copper sulphide minerals and precipitation ranges for metal sulphates have been calculated within a temperature region of 0 to 75°C. These results have been used to provide a basis for investigating the thermodynamic stability of various routes for the heap leaching process.

9:10 AM

RESULTS OF LABORATORY AND PILOT TESTWORK: LEACHING AND PROCESSING OF COMPLEX CONCENTRATES CONTAINING ZINC, LEAD AND COPPER: *L. Filip*[1]; T. Velsa[2]; A. Mezei[3]; [1]Ceronef S.A. Buin, Maro Romania; [2]IAMN S.A., Bucarest, Romania; [3]Lakefield Research, Ltd., Canada

The roast leach electra-recovery process for zinc was modified to treat two typical complex concentrates containing zinc, lead, copper, gold and silver. Laboratory testwork results indicated that the optimum roasting temperature ranged from 700 to 800ϒC in order to produce acceptable residual sulphur levels. The calcined concentrate was subjected to a three stage lench sequence. The resulting leach solution was purified by hernattic precipitation and directed in the recovery of copper and zinc by solvent extraction and electrolysis. The lead and precious metals were recovered by alkaline smelting and subsequent electrolytic purification. A flowsheet was produced based on the laboratory testwork results, and verified in a large scale pilot plant. The recoveries produced during the pilot plant operation ranged from 90 to 92% for zinc and copper, 85% for lead and 90% for gold and silver, respectively.

9:30 AM

ATOMIC FORCE SPECTROSOPIC AND ELECTROCHEMICAL IMPEDANCE SPECTROSCOPIC CHARACTERIZATION OF SULFUR LAYER DURING ACIDIC LEACHING OF CHALCOPYRITE: *M. Misra*[1]; K. Narayanan[1]; B. K. Jena[1]; [1]University of Nevada, Dept. of Chem. & Metall. Eng./MS 170, Reno, NV 89557 USA

The utilization of Atomic Force Microcopy (AFM) along with Electrochemical impedance Spectroscopy (EIS) is a new approach in the characterization of the sulfur layer formed during chalcopyrite leaching. The AFM imaging was performed in conjunction with EIS to analyze sulfur growth, grain size, roughness and peak height. In addition the critical resistance of the sulfur layer and rate law for sulfur layer formation were calculated.

9:50 AM

FEASIBILITY OF SELECTIVE EXTRACTION OF NICKEL AND COPPER FROM COMPLEX SULPHIDE ORE BY PRESSURE LEACHING: *J. X. Guo*[1]; [1]Queen's University, Kingston, Ontario K7L 3N Canada

The typical feed to pressure leach process, as practiced today in nickel sulfide industry, is intermediate nickel-bearing materials such as anode nickel, nickel-copper mattes, or at least nickel sulfide concentrates. Flotation continues to be an indispensable step without which the mining and the subsequent metallurgy of low-grade and complex ore bodies would be prohibitively expensive, However, it has been gradually recognized that the importance of flotation may decline as other methods such as hydro and pyrometallurigal processes may be better qualified to treat refractory ores in which the useful mineral is very finely disseminated and adequate liberation from the gangue is not possible. This paper summarizes the laboratory study in applying pressure leach process to the selective extraction of copper and nickel directly from a complex sulfide ore which mainly consists of chalcopyrite, pentlandite and pyrrhotite. The results indicate significant differences in the relative metal-releasing rates between different minerals by controlling process variables. Nickel-bearing sulfide metals decompose more readily than chalcopyrite while most of iron sulfides remain in the residue in the form of hermatite. Temperature was found to exhibit the strongest impact on selectivity, while a reduced oxygen concentration in combination with a suitable between retention time also yielded very effective selective extraction rates. A two stage pressure leach process was desired and tested at laboratory scale resulting in satisfactory selectivity and overall recovery.

10:10 AM

OPTIMISATION OF THE LEACH CONDITIONS FOR A COPPER/URANIUM ORE: *R. J. Ring*[1]; D. E. Collier[1]; L. Tan[1]; A. Day[1]; S. J. Macnaughton[1]; [1]Australian Nuclear Science and Technology Organization, Environment Division, PMB 1, Menai, NSW 2234 Australia

The Olympic Dam deposit in South Australia contains 24 copper and 0.06% uranium, which are present as bornite, chalcopyrite, uraninite, brannerite and coffinite. The copper is principally recovered using flotation. The floatation tailings, which contain the bulk of the uranium, and typically 0.3% copper, are leached using sulfuric acid and ax oxidant at ambient pressure. This paper describes the results from a detailed investigation of the leaching behaviour of this ore. The influence of acid strength, ferric ion concentration, redox potential and temperature on the dissolution of uranium, copper and gangue minerals was studied. Experimental data was obtained from SEM analysis of individual mineral grains, extensive mineralogical studies and batch leach tests. The maximum extractions of copper and uranium determined experimentally were in good agreement with those predicted from SEM determination of the proportions of refractory chalcopyrite and brannerite in the ore. The leaching rate of copper was found to be independent of acidity over the range 20 g L-1 to pH 2.0, but was strongly dependent on temperature and ferric ion concentration. Uranium extraction showed a weak dependence on acidity, Arrhenius temperature dependence and a complex co-dependence on ferric ion concentration and redox potential. A kinetic model has been developed that predicts copper and uranium dissolution and also accounts for gangue dissolution. This model is being incorporated into a more comprehensive model that can be used to predict leach performance and reagent consumption for specific ore samples.

10:30 AM

FLUOROCHEMICAL SURFACTANT USE IN HEAP LEACHING: THEORY AND PRACTICE: *Michael J. Sierakowski*[1]; [1]3M Performance Chemicals & Fluids Laboratory, 3M Bldg. 236-2A-01, St. Paul, MN 55144 USA

Surfactants play a vital role in the mining industry and flurochemical surfactants, in particular, have demonstrated utility in several hydrometallurgical operations including their use leach operations. Surfactant aided wetting to increase lixiviant contact with one can result in accelerated leach kinatics and increased metal recovery. The critical aspects of establishing fluorochemical surfactant utility by determining their mode of best use, determining amenable ore types, the development of column testing procedures to overcome sample variability, and the nuances of large-scale field testing followed by partnering efforts with mining operations to gain industry acceptance illustrate the commitment required to bring a product from concept to commercial use in this industry.

LIGHT WEIGHT ALLOYS FOR AEROSPACE APPLICATIONS V: Ultra Light Weight Materials

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 Materials, Pohang 790-330 Korea

Tuesday AM Room: 9
March 2, 1999 Location: Convention Center

Session Chair: K S Shin, Seoul National University, Dept. of Mats. Sci. and Eng., Kwanak-ku, Seoul Korea

8:30 AM

ELEVATED TEMPERATURE DEFORMATION OF NANO-SCALE DISPERSION STRENGTHENED ALUMINIUM ALLOYS: *Emma Jane Minay*[1]; Richard J. Dashwood[1]; Henry McShane[1]; [1]Imperial College, Mats. Process. Group, RSM, Prince Consort Rd., South Kensington, London SW7 2BP UK

Nano-scale reinforced aluminium alloys are being developed for applications at 523-573K as a lighter alternative to titanium based materials in the aerospace industry. Results of an investigation of the deformation behaviour of dispersion strengthened aluminium based materials produced by mechanically alloying aluminium and aluminium 0.35wt% lithium 1wt% magnesium powders with 13nm diameter Al_2O_3 powder and 23nm diameter TiO_2 powders are reported. Constant strain rate compression tests were performed at temperatures of 523, 623, 723 and 823 K and strain rates of $5x10^{-5}$ to $10^{-1}s^{-1}$. The effect of volume fraction of dispersoid, type of dispersoid and solute additions on the deformation behaviour is reported. The materials were found to work soften and undergo compressive failure. The deformation behaviour is extremely sensitive to temperature and insensitive to strain rate and is shown to be associated with a threshold stress resulting from a particle dislocation interaction. The strain rate-stress relationship has been compared to the detachment model of Rosler and Arzt. By introducing a variable value for the detachment stress it is possible to reflect the correct temperature dependance of the stress-strain rate relationship.

8:55 AM

THE JOMINY END QUENCH FOR LIGHT WEIGHT ALLOY DEVELOPMENT: *Joseph William Newkirk*[1]; [1]University of Missouri-Rolla, Dept. of Metall. Eng, Rolla, MI 65409 USA

The Jominy end quench test is well known as a method of measuring hardenability in steels. In light weight alloys there is a desire to determine the effect of quenching on final properties after heat treating. The Jominy end quench test offers a method for studying many quenching conditions with a minimum of samples. The potential of the Jominy End Quench and a modified end quenching apparatus for developing new understanding of the complex response of light weight alloys to processing conditions, especially quenching, will be presented. In addition, several examples will be illustrated.

9:20 AM

PROCESSING AND CHARACTERIZATION OF Be-Al ALLOYS: *Xiao-Dong Zhang*[1]; Jorg Wiezorek[1]; Fritz Grensing[2]; Glyn Meyrick[1]; Harry Lipsitt[1]; Hamish Fraser[1]; [1]The Ohio State University, Dept. Mats. Sci. Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Brush Wellman, 14710 West Portage River South Rd, Elmore, OH 43416 USA

Be-Al alloys, due to their high elastic modulus, low density and a relatively high melting point, are one of the most promising light weight materials for aerospace and high speed ground transport applications. It has been reported recently that Be-Al alloys can be cast and extruded successfully, and exhibit reasonably promising mechanical properties. However there is lack of fundamental understanding of the physical metallurgy of these alloys, such as the formation of the cast microstructure which has previously been attributed to the existence of a submerged metastable monotectic reaction in high Be-Al compositions. In the present study we will present and discuss the results on the microstructural characterization on both as-cast and extruded alloys using SEM and TEM. The fracture and deformation mechanisms observed from tensile and compressive test samples will also be presented and discussed in terms of the mobility and the nature of dislocations in Al and Be phase respectively. More recent work on the alloy development will also be presented. This work has been supported by a grant from the Office of Naval Research with Dr. Steven J. Fishman as program manager.

9:45 AM

STRENGTHENING EFFECTS OF Al AND Mn ON WROUGHT Mg-6WT%Zn ALLOYS: *S. C. Park*[1]; K S Shin[1]; [1]Seoul National University, School of Mats. Sci. and Eng., Shinrimdong, Center for Advanced Aerospace Mats., Seoul 151-742 Korea

In order to evaluate the effects of Al and Mn additions on the strengthening of the extruded Mg-6wt%Zn alloys, the age hardening response and tensile properties were examined with different amounts of alloying elements and heat treatments. The microstructures of the aged specimens were examined by STEM. After the double aging treatment the peak hardness was found to increase after shorter aging time. The optimum tensile properties were obtained with the addition of 1 wt% Mn. The addition of 2 wt. % Al to the Mg-6 wt % Zn alloy increased UTS with a small increase in YS. in the double aging condition. The tensile elongation was found to increase remarkably with the Al addition after solution heat treatment and double aging treatment. The effects of Al and Mn addition will be discussed in the light of the experimental results.

10:10 AM

THE MECHANICAL PERFORMANCE OF METAL MATRIX COMPOSITE JOINTS: *Damon D. Brink*[1]; S. A. Waltner[1]; C. G. Levi[1]; F. A. Leckie[1];[1]University of CA at Santa Barbara, Mats., Santa Barbara, CA 93106 USA

The mechanical performance of joints in MMCs will be discussed with regard to the interplay between metal-ceramic interfaces, metal constraint and the geometry of composite -monolith transitions. Model joints consisting of aluminum matrix composite subelements separated by a thin metal interlayer were fabricated by pressure infiltration of an Al-4.5% Mg alloy into preforms of polycrystalline alumina fibers containing planar discontinuities, thus minimizing processing defects and ensuring interface integrity. The high degree of constraint and concurrent build up of large hydostatic stresses in the metal layer allows simple butt joints to support applied loads in excess of 3 times the metal yield strength. The composite /interflayer interface fails at these stress levels due to the coalescence of debonded regions at the fiber ends. Increasing metals constraint to the order of the fiber diameter achieved by reinforcing the interlayer with particles inhibits this mode of failure and results in strength increases of up to 50%. The failure mechanisms operating in these joints will be discussed in terms of the relevant length scales of plasticity associated with differences in interfacial microstructure. Changes in the evolution of the interlayer stress state associated with various levels of constraint will also be considered.

10:35 AM

DEVELOPMENT OF STEEL FOAM AS A LIGHT WEIGHT MATERIAL FOR AEROSPACE APPLICATIONS: *Mike Yu*[1]; Harald Eifert[1]; Markus Knuwer[2]; Markus Weber[2]; [1]Fraunhofer Resource Center, Mats., Newark, DE USA; [2]Fraunhofer Institute for Applied Materials Research, Bremen Germany

A new powder metallurgy process for the production of metallic foams was developed for a range of alloys including Al and steel. This method allows for a direct net-shape fabrication of foamed parts with relatively homogeneous and isotropic pore structure. Metallic foam made by this approach has a high volume fraction of porosity and exhibits a closed-cell microstructure. This type of microstructure is particularly attractive for applications requiring high specific stiffness and energy absorption. This paper will report the result of a project sponsored by the ONR in developing steel foam for lightweight structure. To date, the obtained foam steel has a range of density from 3-5 gm/cm3. The advantage in weight reduction in steel may enable its use for aerospace applications.

11:00 AM

DEFORMATION BEHAVIOUR OF 7075Al/SiCp COMPOSITE DURING MULTI-PASS DEFORMATION AT HIGH TEMPERATURES: A. Razaghia[1]; D. Yu[1]; H. Asanuma[2]; T. Chandra[1]; [1]Department of Materials Engineering, Wollongong University, Wollongong, NSW, 2522, Australia; [2]Chiba University, Department of Mechanical Engineering; Chiba City, 263, Japan

Hot Deformation behavior of 7075 aluminium alloy containing 15 vol% of SiC particles (average size of 14 μm) and the monolithic alloy was studied at 300 and 400°C at constant strain rate of $1s^{-1}$ under condition of uniaxial compression. The effect of delay between two consecutive passes on the high temperature mechanical strength and microstructural development was examined. The results showed that the fractional softening (%FS) increased in both reinforced and monolithic alloys when the deformation temperature increased from 300 to 400°C, but monolithic alloy showed a slightly higher FS compared to composite under identical deformation conditions. TEM examination revealed that the monolithic alloy and composite contained almost similar substructures after either single or double pass deformation at a given temperature irrespective of interpass hold time. However, some subgrain growth was observed in these materials during holding after deformation at 400°C, but this was not the case at 300°C. The absence of subgrain growth at lower temperature can be attributed to pinning effect by fine dispersions present in the matrix. The structural study also showed that static recrystallization did not occur in these materials during hold time between passes, and the fractional softening occurs mainly due to static recovery.

11:25 AM

CORROSION FATIGUE BEHAVIOR OF THE HIGH-STRENGTH MAGNESIUM ALLOY AZ 80: M. Hilpert[1]; L. Wagner[1]; [1]Technical University of Brandenburg at Cottbus, P.O. Box 10 33 44, 03013 Cottbus, Germany

The high-strength magnesium alloy AZ 80 was received as extruded bar. Specimens were machined with the load axis parallel to the extrusion (L) direction as well as parallel to the radial (R) direction. Axial fatigue tests were performed on electrolytically polished hourglass shaped specimens in fully reversed loading (R=-1) using a resonance testing machine at frequencies of roughly 100 Hz. Tests were performed in an aequious 3.5% NaCl solution and in lab air. In addition, some tests were conducted in vacuum. To improve the fatigue performance, mechanical surface treatments were applied. Results on shot peened and roller-burnished specimens will be compared with the electrolytically polished reference. Fatigue performance of the various conditions will be interpreted in terms of crystallographic texture, surface roughness, work hardening and residual compressive stresses.

MATERIALS PROCESSING FUNDAMENTALS: Thermodynamics and Kinetics

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

Program Organizers: W.D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Huimin Liu, UES, Inc., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Lab, P.O. Box 2008, Bldg. 4508, Oak Ridge, TN 37831-6083 USA

Tuesday AM Room: 5A
March 2, 1999 Location: Convention Center

Session Chairs: Weol D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; J. B. Hisky, University of Arizona, Dept. of Mats. Sci. and Eng., Tuscon, AZ 85721 USA

8:30 AM

THERMODYNAMIC STUDY OF ZINC-IRON INTERMETALLICS BY MASS SPECTROMETRY: Kazuaki Mita[1]; *Masafumi Maeda*[1]; [1]University of Tokyo, IIS, 7-22-1 Roppongi, Minato, Tokyo Japan

There are various intermetallic compounds in Zn-Fe system. We synthesized those at about 500ϒC. Specimen was examined chemically and Xray diffraction was applied to ensure the formation of intermetallics compounds. Material containing two phases was placed in a knudesen type effusion cell and mass spectrum studied to evaluate the vapor pressure of Zinc. Thermodynamics of intermetallics are then evaluated through measured intensity.

8:55 AM

SIMULTANEOUS SULFATION OF CALCIUM OXIDE AND MAGNESIUM OXIDE IN CALCINED CMA (CALCIUM MAGNESIUM ACETATE): *Dong Hoon Han*[1]; Hong Yong Sohn[1]; [1]University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA

The kinetics of the simultaneous sulfation by sulfur dioxide and oxygen of CaO and MgO in calcined CMA (calcium magnesium acetate) were investigated experimentally and compared with a rate equation obtained based on the kinetics of each reaction. The experiments were carried out in the temperature range from 700 to 830ϒC and under the S0[2] concentrations of 0.3 to 1 mol%. The oxygen concentration and the particle size were 5% and-325+400 mesh (37-44um), respectively Each sample was calcined completely by heating it at about 10ϒC /min up to 1000ϒC under a nitrogen atmosphere, before the temperature was lowered to the sulfation temperature. According to the ranges of partial pressure of sulfur dioxide and temperature, there exist three regions for the sulfation reaction: region I in which neither oxide is sulfated, region II in which only CaO is sulfated, and region III in which both oxides are sulfated. To investigate separate sulfation kinetics of each solid in calcined CMA, the sulfation kinetics of the CaO content were obtained in region II. The MgO sulfation kinetics were obtained in region III after the CaO content was fully sulfated to CaSO4. The experimental results were in good agreement with the theoretical predictions according to the combined simultaneous rate equation.

9:20 AM

REDUCTION OF CALCIUM SULFATE BY HYDROGEN TO PRODUCE CALCIUM SULFIDE AS A REDUCTANT OF SULFUR DIOXIDE TO ELEMENTAL SULFUR: *Byung-Su Kim*[1]; Hong Yong Sohn[1]; [1]University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112-0114 USA

The reaction between calcium sulfate and hydrogen is of interest both as a means of regenerating calcium sulfide as a reductant of sulfur dioxide to elemental sulfur and for producing elemental sulfur from gypsum. The kinetics of this reaction were measured using a thermogravimetric analysis technique in the absence and presence of nickel catalyst, and the reactivity of regenerated calcium sulfate investigated. The reaction temperature was varied between 973 and 1153K, while hydrogen partial pressures of 2~ 86 kPa were utilized. At 1073K in the hydrogen partial pressure of 86 kpa, 60% of the original calcium sulfate in the absence, and 95% in the presence, of the nickel catalyst was converted to calcium sulfide in one hour. A nucleation and growth model was found to fit the reaction rate reasonably well. The reactivity of the regenerated calcium sulfate, with or without the catalyst, remained similar to that of the original sample even after three cycles.

9:45 AM

PSEUDO-BINARY PHASE DIAGRAM OF THE CUSPIDINE - CAF2 SYSTEM - RELATING TO MOLD FLUX FOR CONTINUOUS CASTING OF STEEL: *Hiroyuki Fukuyama*[1]; Takashi Watanabe[1]; Masahiro Susa[2]; Kazuhiro Nagata[1]; [1]Tokyo Institute of Technology, Dept. of Chem. and Mats. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan; [2]Tokyo Institute of Technology, Dept. of Metall. Eng., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Recently, the role of cuspidine, $3CaO2SiO_2CaF_2$, has been of considerable practical concern in mold flux for the continuous casting process of steel. Cuspidine crystallizes in almost all kinds of flux films during the casting of steel and it affects the thermal properties of the flux. Therefore, the crystallization causes a non-uniform heat flux between the strand and the mold, which may lead to the surface defects of slab due to the thermal stress. However, very little work is currently available in the published literature on the fundamental physicochemical properties of cuspidine, such as the phase diagram, the free energy of the formation and the thermal conductivity, etc. In the present paper, one of the most important data to design mold powder, i.e. the phase diagram of the system cuspidine - CaF_2 has been studied by electron probe X-ray microanalysis (EPMA) and X-ray diffractometry (XRD).

10:10 AM BREAK

10:20 AM

KINETICS OF DEHYDRATION OF $FeSO_4$-$7H_2O$ UNDER DIFFERENT ATMOSPHERES: *N. Kanari*[1]; I. Gaballah[1]; C. Mathieu[2]; N. Neveux[2]; O. Evrard[2]; [1]LEM, Associated to CNRS UMR 7569, Mineral Process. and Environmental Eng. Team, ENSG, INPL, Vandceurve BP 40, 54501 France; [2]Universite Henri Poincare Nancy I, Laboratoire de Chimie du Solide Minerale, Vandceuvre Bp 239, 54506 France

Hydrated ferrous sulfate is a by-product of titanium and surface treatment industries. The majority of this product has to be waste disposed that is considered as a supplementary cost for these industries. Recently, the ferrous sulfate partially dehydrated was successfully used for the synthesis of a new superoxidant material containing iron in hexavalent state (Fe^{VI}). The kinetics of dehydration of FeSO4~7H2O under neutral, oxidizing and chlorinating atmospheres were studied by thermogravimetric and differential scanning calorimetry analysis. The obtained products were analyzed by X-ray diffraction and Mossbauer spectroscopy. The effects of temperature, gas velocity, type of sulfate, etc. on the dehydration rate were defined.

10:45 AM

KINETICS OF OXYCHLORINATION OF MAGNESIUM OXIDE: *N. Kanari*[1]; E. Allain[2]; I. Gaballah[1]; [1]LEM, CNRS UMR, Mineral Process. and Environmental Eng. Team, ENSG, INPL, Vandceure BP 40, 54501 France; [2]University of Missouri-Rolla, School of Mines and Metallurgy, Center for Pyrometallurgy, 215 Fulton Hall, Rolla, MO 65401 USA

Kinetics of oxychlorination of MgO by Cl_2+$0_2$2 were in the temperature range of 850ϒC to 1025°C using thermogravimetric analysis. The effects of $C1_2/0_2$ ratio, gas velocity, temperature and partial pressure of reactive gases reaction rate were investigated. The apparent activation energy of MgO oxychlorination was about 214 kJ/mol. The reaction orders with respect to Cl_2+0_2, Cl_2 and 0_2 at 950ϒC were about 0.65, 0.98, and -0.37, respectively. Some data concerning the oxychlorination of MgO were compared with those of Cr_20_3 and $MgCr_2O_4$ contained in

chromite mineral. Efficiency of using oxychlorination for the removal of iron oxides contained in magnesia was demonstrated.

11:10 AM
KINETIC STUDY ON THE LIME ENHANCED REDUCTION OF CHALCOCITE WITH CARBON: *M. C. Ruiz*[1]; R. Padilla[1]; [1]University of Concepcion, Dept. of Metall. Eng., Edmundo Larenas, Concepcion 270 Chile

The conventional pyrometallurgical methods to treat copper sulfide concentrates include generally one or more oxidizing steps where the formation of $S0_2$ gas can not be prevented from the economical point of view and the nature of some of the processes in which SO_2 is produced, its complete capture is not feasible; therefore, an important fraction is released to the atmosphere as fugitive emissions. In this research, the reduction of chalcocite was studied in the presence of lime as an alternative method which overcomes the pollution problems related to SO_2 emissions. The effects of temperature, time, concentration of the reactants carbon and CaO on the reductions rate were studied. From these variables, temperature affects most the reduction rate. Conversions over 95% can be obtained in less than 20 um at temperatures over 1000ϒC. The measured partial pressures of the gaseous components indicated that the chalcocite reduction proceeds through the gaseous intermediate species CO, $C0_2$, and that the overall kinetics was controlled by the Boudouard reaction. The kinetic model ln(1 -X)= kt fits well the experimental data, and an apparent activation energy of 314 kJ/mol was calculated for the temperature range 800ϒC-1050ϒC.

11:35 AM
KINETICS OF SCORODITE FORMATION: *Preeti Pande*[1]; J. B. Hiskey[1]; [1]University of Arizona, Dept. of Mats. Sci. and Eng., AZ USA

The kinetics of high-pressure, hydrothermal crystallization of the arsenic containing mineral, scorodite is investigated. An amorphous FeAsO4 particulate is formed in solution, and its transformation into the As-stabilized, crystalline scorodite phase is followed both by a selective dissolution method and by XRD. The effects of treatment temperature and of the iron and arsenic concentrations on the kinetics of the transformation are discussed.

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

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

Tuesday AM Room: 14B
March 2, 1999 Location: Convention Center

Session Chairs: Tresa M Pollock, Carnegie Mellon University, Dept. of Mats. Sci and Eng., Pittsburgh, PA 15213 USA; Hael Mughrabi, University Erlangen-Nuernberg, Erlangen, Bavaria D-91085 Germany

8:30 AM INVITED PAPER
LONG-RANGE INTERNAL STRESSES: THE COMPOSITE MODEL AND ITS CONSEQUENCES: *Hael Mughrabi*[1]; [1]University Erlangen-Nuernberg, Werkstoffwissenschaften, Martensstr 5, Erlangen, Bavaria D-91058 F.R. Germany

The original discussion about the role of long-range internal stresses in strain-hardened (single-phase) metals was based on dislocation pile-ups as the classical sources of long-range internal stresses. The problems encountered were related to the fact that, while there was abundant evidence in favor of deformation-induced long-range internal stresses, dislocation pile-ups were rarely observed. This problem was resolved theoretically by the introduction of the composite model of heterogeneous dislocation distributions which could also be verified experimentally. The main idea of the composite model is that any heterogeneous dislocation distribution, i.e. also a dislocation cell structure, bears in it long-range internal stresses as a necessary consequence of strain compatibility, leading to internal forward stresses in the hard and internal back stresses in the soft regions, respectively. In order to develop the composite model further, some points need more work. First of all, there is a need to express the microstructural parameters of the composite model in terms of the macroscopic parameters measured in a mechanical test. Next, the composite model is a static consideration of the stress-applied state. It lacks all aspects of dislocation dynamics and thermal activation. It cannot, therefore, explain some other experimental observations such as, for example, the fact that the glide paths in stage II extend over, typically, 5 to 10 bundle/wall spacings. The idea of the composite model which considers the local stresses in soft and hard regions has consequences with respect to existing work-hardening models such as the long-range stress, the meshlength or the forest theories. These aspects will be discussed

9:00 AM
SEQUENCES OF DISLOCATION PATTERNS: *Frank R. Nabarro*[1]; [1]University of Witwatersrand, Johannesburg, Div. of Mats. Sci. and Tech. P.O. Box 395, CSIR, Pretoria 0001 South Africa

It is generally accepted that continued plastic deformation of a metal occurs in a sequence of stages. In each stage, the dislocation pattern shrinks, while remaining roughly self-similar, and the transition between successive stages is fairly abrupt. Possible causes for these changes of pattern are discussed. It is emphasized that the changes occur under an imposed stress. A simple model is presented in which, as the applied stress is increased through a critical value, a structure with a uniform dislocation density gives way to one with a higher average density but a lower total energy.

9:20 AM
DYNAMIC RECOVERY BY LOMER GLIDE: *Vasily V. Bulatov*[1]; Ali S. Argon[1]; [1]Massachusetts Institute of Technology, Dept. of Mech. Eng., Room 3-382, Cambridge, MA 02139 USA

Transition from stage II (linear hardening) to stage III (parabolic hardening) in FCC metals has been of considerable interest for decades. Although phenomenology of this transition is well established experimentally, its mechanistic interpretation remains an issue. Despite its wide acceptance, the well known Seeger's theory of dynamic recovery by cross-slip is not consistent with available experimental observations. We suggest a different mechanism for dynamic recovery in which dislocation density in the cell walls is reduced by recombination, concurrently with storage of new density. The recovery mechanism involves thermally-activated removal of the Lomer-Cottrell dislocation segments anchoring the excess dislocation density in the cell walls under the action of the applied stress and the active dislocation flux. Under normal conditions, in stage II, the LC locks are sessile, as is usually assumed. However, at higher stress levels typical of stage III deformation conditions, the locks can move in the {001} planes by the nodal glide mechanism discovered by Karnthaler[1]. Although nodal glide should be very slow compared to the motion of glissile dislocations, it provides a path for LC dislocation segment removal which will, in turn, trigger massive recombination of the excess dislocation density in the cell walls by spontaneous glide. We present results of large scale 3-D atomistic simulations of nodal glide in Al and Cu which demonstrate feasibility of the proposed scenario for dynamic recovery in stage III. [1]Karnthaler, H. P., 1978, Philos. Mag. A, 38, 141-156.

9:40 AM
BOUNDS AND INTERMEDIATE MODELING OF LARGE ELASTIC-VISCOPLASTIC BEHAVIOR OF POLYCRYSTALS: *Said Ahzi*[1]; [1]Clemson University, Dept. of Mech. Eng., Clemson, SC 29631 USA

The aim of this work is to propose a simple intermediate model for large elastic-viscoplastic deformations that could predict the copper and brass texture components in FCC metals as well as the texture transition. First, we propose a new formulation of the Sachs model as extension of this model to large elastic-viscoplastic deformations. In light of state variable based modeling, we introduce a one parameter weight function to formulate an intermediate model. This proposed model combines both bounds, Taylor and Sachs models. Our motivation for this investigation is to develop simple intermediate modeling other than the self-consistent models. In the applications, we concentrate on axisymmetric and plane strain compression tests. The results from all these models will be shown and compared with each other and with experimental results. A preliminary discussions concerning single parameter, used in the intermediate model, is given. This constitute a first attempt for modeling the effect of non-uniformity of deformation in polycrystals using simple intermediate modeling.

10:00 AM BREAK

10:15 AM INVITED PAPER
REALISTIC CONSTITUTIVE RELATIONS FOR METAL PLASTICITY: *U. Fred Kocks*[1]; [1]Los Alamos National Laboratory, Center for Materials Science, Mail Stop K765, Los Alamos, NM 87545 USA

In 1975, Ali Argon edited a book "Constitutive Equations in Plasticity". In it, two of the now widely acknowledged mainstays were presented: that plasticity must be expressed in state-variable form; and that different regimes of behavior exist that require different relations. Since then, this framework has been filled with a wide set of quantitative relations for a number of (mostly fcc) metals, with respect to both the kinetics of flow and the evolution of the state parameters in macroscopic bodies. The relations are not always expressible by closed-form equations, but by nomograms or graphs, derived by interaction with experiment and simulation. In this way, it is assured that the relations are actually executable in practice, and that they reflect real material behavior for a specific case. The most recent form of the underlying models will be given explicitly.

10:45 AM INVITED PAPER
A COMPARATIVE ANALYSIS OF LOW TEMPERATURE DEFORMATION IN B2 ALUMINIDES: NiAl, RuAl AND FeAl: *Tresa M. Pollock*[1]; David Lu[1]; Xiaoli Shi[1]; Khenlak Eow[1]; [1]Carnegie Mellon University, MSE Dept., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Aluminides with the B2 structure are an interesting group of compounds which could be useful as structural materials. However, these ordered compounds often lack adequate low temperature tensile ductility. In this study, macroscopic and microscopic aspects of deformation have been studied in a series of B2 aluminides with a wide range of melting temperature and varied levels of toughness and/or ductility. Deformation kinetics have been measured with the use of strain rate change experiments in the temperature range of 77K to room temperature. Dislocation substructures have been characterized in detail in critical regimes of temperature and strain rate. A wide variety of deformation processes have been observed, and their relationship to macroscopic properties will be discussed.

11:15 AM
DEFORMATION MICROSTRUCTURES IN SINGLE CRYSTALS AND POLYCRYSTALS AND WORK HARDENING STAGES: *N. Hansen*[1]; D. A. Hughes[2]; [1]Risoe National Laboratory, Mats. Research Dept., Roskilde DK-4000 Denmark; [2]Sandia National Laboratories, Center for Mats. and Applied Mechanics, Livermore, CA 94550 USA

The evolution of dislocation structures in single crystals and polycrystals with increasing strain is described within a framework of grain subdivision by dislocation boundaries and high angle boundaries. The microstructures evolving are characterized with emphasis on morphology and the changes in the misorientation across and the spacing between the deformation induced boundaries. These parameter changes are modelled over a large strain range using scaling methods of relevance to microstructural and micromechanical modelling. The dislocation boundaries are further analyzed using Franks formula. It is shown that the dislocations in the boundaries originate in part from active slip systems which are predicted by a Schmid factor analysis or a Taylor-Bishop-Hill model. In addition, these analyses show that supplementary slip systems are activated due to the presence of the deformation induced boundaries. These different types of analyses lead to a discussion of the mechanisms controlling the microstructural evolution and of correlations between the microstructural transformations and the work hardening stages.

11:35 AM
THE EFFECT OF PARTICLE DISTRIBUTION ON DEFORMATION AND DAMAGE IN TWO-PHASE ALLOYS: K. Conlon[1]; P. Poruks[1]; *D.S. Wilkinson*[1];J.D. Embury[1]; [1]McMaster University, Dept of Materials Science and Engineering, Hamilton, Ontario L8S 4L7

In materials containing large (>1 micron) particles, which strengthen by continuum load transfer from the matrix, a non-uniform particle distribution can have a profound effect on both strength and ductility. We have studied this using a combination of modelling (based on self-consistent analysis) and experiments (based on model materials). The models are best able to treat severe cases of clustering when an essentially bimodal distribution exists. These show that inhomogeneity enhances the efficiency of load transfer. However, this effect is negated once damage commences. The models have been tested against experiments involving powder processed Al-Cu alloys containing a bimodal distribution of CuAl2 particles, and against spherodized steels in which a bimodal carbide distribution can be introduced through thermomechanical processing. The tensile and compressive behavior of these materials is broadly consistent with the theory.

MILTON BLANDER INTERNATIONAL SYMPOSIUM ON "THERMODYNAMIC PREDICTIONS AND APPLICATIONS": High Temperature Experimentation

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. Eng., Tuscaloosa, AL 35487 USA; Dr. A. D. Pelton, Montreal, Quebec H3C3A7 Canada

Tuesday AM Room: 4
March 2, 1999 Location: Convention Center

Session Chairs: Robert A. Osteryoung, North Carolina State University, Dept. of Chem., Raleigh, NC 27695-8204 USA; Kjell Hagemark, STATOIL, Research Centre, Trondheim, Ranheimsveien 10 Norway

8:30 AM INVITED PAPER
HIGH-TEMPERATURE THERMOCHEMISTRY; FROM LIQUID ALLOYS TO MOLTEN SALTS: *Ole J. Kleppa*[1]; [1]James Franck Institute, The University of Chicago, 5640 S. Ellis Ave., Chicago, IL 60637 USA

This paper will begin with a historical part. It starts with the first calorimetric study of liquid alloys by Kawakami carried out at Tohoku University in 1927-30. It will then touch on the calorimetric work on intermetallic compounds of Kubaschewski and Walter in Germany, first published in 1939. A study of this paper in due course led the author to the development of his first high-temperature reaction calorimeter in 1952. This new calorimeter was used very extensively in studies relating

to low-melting liquid alloys in the 1950's. A fairly detailed review of this work on liquid alloys was presented to the ASM in the symposium on "Liquid Metals and Solidification" published in 1958. However, the author had spent 1956-57 as a Visiting Professor at his alma mater in Trondheim, Norway. His office in Trondheim was next door to that of Professor Hakon Flood, the founder of the Norwegian School of Molten Salt Chemistry. His many discussions with Professor Flood led the author to conclude that calorimetry of molt en salt mixtures would be a very desirable new enterprise. However, the calorimeter used in his work on liquid alloys was not very suitable for studies of molten salts. Fortunately, in the meantime Calvet and Prat had in 1956 published their new monograph "Microcalorimétrie". Development of a series of new Calvet type calorimeters for temperatures which ranged from 350°C to 1100°C provided the obvious solution to detailed calorimetric studies of molten salt mixtures, from liquid nitrates to liquid fluorides. A review will be presented of our work in this field.

9:10 AM

DEVELOPMENT OF LIYO2 AS A SOLID ELECTROLYTE FOR LITHIUM: Luis Yamarte[1]; Aniceta Skowron[1]; *Anthony Petric*[1]; [1]McMaster University, Dept. of Metall. and Mats. Sci., Hamilton, Ontario L8S 4L7 Canada

The properties of lithium yttriate were studied and the crystal structure of monoclinic LiYO2 was determined using neutron diffraction. The mobility of Li ions in the YO2 framework is discussed. A cell was devised to determine the activity of lithium in liquid alloys. The lithium yttriate electrolyte was prepared by solid state reaction of lithium carbonate and yttrium oxide. The cells were made by slip casting the electrolyte powder into tubes and sealing them to Y2O3 lids. The following cell configuration was used for both titration of lithium and measurement of the E.M.F.: (-)Ta/Li/LiYO2/Li-Zn(liq)/Ta (+). The activities of lithium in molten Li-Zn alloys at 823K show moderately negative deviations from Raoult's law over the composition range studied. LiYO2 was found to have good stability in both air and molten lithium environments.

9:30 AM

PHOSPHATE CAPACITIES OF CaO SLAGS: *Ramana G. Reddy*[1]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

The phosphate capacity of slag is a measure of ability of a slag to remove phosphorous from molten alloy. The phosphate capacities of $CaO\text{-}CaF_2$ and $CaO\text{-}AlO_{1.5}$ slags were experimentally determined. The melts were equilibrated in a graphite crucible with CO-Ar mixtures and Ag-P alloys between 1450 and 1600°C. The phosphate capacities model for predicting C_{PO4} in slags "a priori" was developed. The predicted data are in very good agreement with the experimental capacity data. The phosphorous distribution ratio was calculated using the capacity data for slags.

9:50 AM

THERMODYNAMICS OF HYDROGEN ABSORPTION AND DESORPTION IN Pd-Rh-Co TERNARY ALLOYS: *David F. Teter*[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory, MST-6 (Metallurgy), MS: G770, Los Alamos, NM 87545 USA

The effect of varying the Rh and Co compositions on the thermodynamics of hydrogen absorption behavior, hydride formation and decomposition, and hydrogen capacity of several Pd-Rh-Co ternary alloys has been investigated using pressure-composition (PC) isotherms. The trends in the thermodynamic properties of the alloys as a function of Rh and Co content will be correlated to x-ray diffraction measurements of the lattice parameters as well as other physical properties of the alloys. An empirical model, based on binary Pd-based alloys, has been developed which accurately predicts the thermodynamic properties of hydrogen solution, hydride formation and decomposition, and the hydrogen capacity of the ternary Pd-Rh-Co alloys. Also, the effects of cycling on the thermodynamic parameters will be presented. Results show that cycling decreases the enthalpy of hydride formation and slightly increases the enthalpy of hydride decomposition.

10:10 AM BREAK

10:30 AM

THERMODYNAMICS OF IRON AND EARTH'S CORE: *Surendra K. Saxena*[1]; Leonid S. Dubrovinsky[1]; Peter Lazor[1]; [1]Uppsala University, Earth Sciences, Geocentrum, Villavägen 16, Uppsala S-75236 Sweden

There are four well-known structural polymorphs of iron, namely alfa and delta (body centerd cubic), gamma (face centered cubic) and epsilon (hexagonal closest packed). The possible occurrence of a new iron phase was suggested by [(1)]; the evidence was indirect, obtained through laser heating of iron above pressures of about 40 gigapascal (GPa). We found that the FCC phase (face centered cubic), when heated in its stability field, is quenchable at high pressures. Using this technique, we determined that the structure of the quenched phase at high pressures is DHCP (double hexagonal closest packed). Several experiments done using in-situ heating and x-ray have now confirmed the presence of this new phase [(2)]. Although experimental data are sparse, it is possible to obtain a fairly quantitative thermodynamic description of all the iron phases and the double hexagonal closest packed (DHCP) beta iron. Earth's core beginning at depths of 2900 Km (133 GPa) and continuing to the center (6730 Km, 360 GPa) consists principally of iron. Until experiments become feasible on iron above 200 GPa, extrapolation of thermodynamic data on iron is necessary for the study of the core. The assessed data on iron is based on experimental data on a) melting to 200 GPa, b) the location of the triple point HCP-DHCP-FCC at 36 GPa and 1450K and c) the location of the triple point DHCP-FCC-melt close to 60 GPa and 2800K. If no other phase transition intervenes, the melting of beta-iron at 360 GPa takes place at temperatures less than 5000K, which constrains the temperature of Earth's center to be less than that. However there are some shock-wave data that cannot be easily reconciled with the current data obtained with the diamond-anvil cell technique unless there is yet another high P phase transition of the beta phase at about 200 GPa to increase the melting temperature by about 1000 at the center. A search for such a phase is now on. [1]S. K. Saxena, Shen, G. & Lazor, P. Science, 260, 1312-1314, (1993). [2]S. K. Saxena, L. S. Dubrovinsky and Häggkvist, P. Geophys. Res. Lett., 23, 2441-2444 (1996). [3]O.L. Anderson, Rev. Geophys. Suppl., 429-441, (1995).

11:10 AM

PROPERTIES OF MONAZITE (LaPO4) BASED CERAMICS: *Robert M. Housley*[1]; Peter E.D. Morgan[1]; and Janet Davis[1]; [1]Rockwell Science Center, 1049 Camino dos Rios, Thousand Oaks, CA 91360.

Successful development of oxidation resistant ceramics with good strength and toughness properties at high temperatures could increase the efficiency of energy generation, with consequent major economic and environmental advantages. Monazite has a number of properties which suggest that it will be a valuable component in high temperature ceramic composites. Monazite-La (LaPO4) melts at 2074 C and is phase compatible with alumina and alumina based fibers to at least 1600 C. It is also soft and readily deforms by cleavage, twinning, and dislocation motion to quite low temperatures. Fiber pullout has been well documented. For these reasons we have been studying the synthesis of monazite and the fabrication of monazite based ceramics. Most synthesis methods yield a phosphorus rich product. Ceramics made from materials containing even a fraction of a percent excess phosphorus frequently exhibit giant grain growth and have little strength. We have developed a procedure for making stoichiometric monazite powder precursor. Ceramics made from it remain fine grained and tough even at high temperatures. Microstructures will be compared.

11:30 AM

MODELLING STUDIES IN MOLTEN SALT ELECTROREFINING OF PLUTONIUM: *B. Mishra* [1]; D.L. Olson[1]; A. Raraz[2]; [1]Kroll Institute for Extractive Metallurgy, Dep't. Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO 80401; [2]CMT 205, Argonne National Laboratory, 9700 S. Cass Avenue, Argonne, IL 60439-4837

The study and optimization of process parameters in some of the processes involved in the production of plutonium metal is extremely difficult. Due to the dynamic nature of the systems operating at high temperatures under protective atmospheres, safety and health-hazard concerns, waste-related issues and the cost considerations, the use of actinide metals directly to investigate the processes is inappropriate. This study discusses the use of various metals and alloys to simulate the

pyrometallurgical characteristics of plutonium. Design parameters relating to component arrangement in process-reactors, process parameters relating to chemical behavior of plutonium and operating parameters relating to reactor conditions, such as the temperature, stirring, current density, metal recovery, etc. have been studied through modeling. The validity and justification of these measurements have been discussed.

11:50 AM

EXPERIMENTS IN THE REMOVAL OF COPPER FROM IRON WITH OXIDE AND SULFIDE-MODIFIED OXIDE FLUXES: *Adam Cohen*; Argonne National Laboratory, 9700 S. Cass Ave., ET/Bldg 212, Argonne, IL 60439-4838.

If an economically viable method were available for removing copper from scrap iron, steelmakers could turn high-copper-content scrap (above 0.1 wt.%) into useful products. Molten salt mixtures of charge-asymmetric cation species show a significant drop in the activity coefficient for monovalent cation species in the presence of trivalent or higher cation species. This phenomenon can be used to design a flux for the removal of copper as a (monovalent) cuprous species from iron-based metal. Distribution coefficients of copper (i.e., the ratio of the weight percent copper in a matte to that in the metal) have been determined by various authors between a sulfide flux and iron. However, the values have not been high enough to make their use on a commercial scale economically viable, largely because a sulfide flux would result in a high sulfur content in the treated metal. A sulfide-based process would likely need a distribution coefficient of 100 or higher to be a good candidate for a commercial process. In this study, experiments were conducted to determine if distribution coefficients between an oxide flux (consisting of alumina, silica, calcia, and ferrous oxide) and iron would be increased by the charge-asymmetric effect described above. In the limited number of tests conducted, a maximum distribution coefficient of 0.5 was found at 1,923 K. While this distribution coefficient is significantly higher than the equilibrium constant required for the reaction between ferrous oxide and copper to form cuprous oxide (K » 0.025), it is not high enough for the process to be developed commercially. Finally, distribution coefficients were determined between a sulfide-modified oxide flux (consisting of alumina, silica, ferrous sulfide, and dilute amounts of calcia, magnesia, calcium sulfide, or magnesium sulfide) and iron with various concentrations of carbon at 1,823 K. A maximum distribution coefficient for copper of 6.8 was found for iron with 0.1 wt.% carbon; distribution coefficients increased as carbon content decreased. While that level is still too low for commercial applications, further research with the mixed flux is strongly recommended because significantly higher distribution coefficients should be possible.

11:50 AM

RARE EARTH CONTAINING HALIDE SOLUTIONS AND PHASE DIAGRAM DETERMINATION: *Zhiyu Qiao*[1]; [1]University of Science & Technology Beijing, Dept. of Phys. Chem., Beijing 100083 China

China is very rich in rare earths (RE). Fundamental research work on RE containing halide solution models and phase diagram determinations is of great importance for better understanding their structures and properties as well as for RE applications. As a key project supported by the National Natural Science Foundation of China in cooperation with CRCT, Ecole Polytechnique de Montreal, Canada, RE containing halide solution models have been studied and the phase diagrams of the RE halide and divalent iodide systems have been determined by the CALPHAD technique in combination with accurate experimental measurements. Several models for describing the Gibbs energies of RE liquid halide solution, especially the polynomial series in equivalent fractions and a modified quasichemical theory developed by M. Blander and A. Pelton, are discussed for phase diagram optimization. Several series of binary RE containing halide and divalent iodide phase diagrams have been optimized and calculated, agreeing with experimental data quite well. As well, a series of ternary phase diagrams of the RE containing chloride systems has been successfully predicted by using the FACT system. In order to determine reliable measured phase diagram, many experimental improvements have been made and are also discussed. (Project supported by National Natural Science Foundation of China.)

MINIATURE STRUCTURES & COMPONENTS UNDER CYCLIC LOADING; FATIGUE & INTERNAL FRICTION: Session I

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee; Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee; ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: H. D. Merchant, Gould Electronics, Inc., Eastlake, OH 44095-4001 USA; Thomas R. Bieler, Michigan State University, Dept. of Mats. Sci. & Mech., East Lansing, MI 48824-1226 USA; James C. Earthman, University of California, Dept. of Chem. Eng. & Mats. Sci., Irvine, CA 92717-2535 USA; M. Wuttig, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20743-2115 USA

Tuesday AM Room: 11B
March 2, 1999 Location: Convention Center

Session Chair: James Earthman, University of California - Irvine, Dept. of Chem. Eng. and Mats. Sci., Irvine, CA 92717-2535 USA

8:30 AM INVITED PAPER

RESIDUAL STRESSES AND THERMOMECHANICAL FATIGUE RESPONSE IN MINIATURE STRUCTURES AND COMPONENTS: *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Room 4-140, Cambridge, MA 02139-4307 USA

The measurement of residual stresses and thermomechanical fatigue characteristics of small-volume structures and components will be examined. Theoretical, computational and experimental results for extracting the magnitude and sign of residual stresses in thin films and surface layers of thick materials will be presented. Independent and complementary experimental methods for the extraction of plastic yield properties of thin films and patterned thin lines on substrates will then be considered by recourse to curvature measurements, nanoindentation and x-ray diffraction. Theoretical formulations which facilitate the determination of curvatures, volume-averaged stresses and/or thermal fatigue loading conditions for the onset of plastic yielding will then be considered along with experimental results for glass, aluminum and copper films/lines on silicon substrates. The effects of microstructural, crystallographic, and physical size effects on the interpretation of various properties will also be highlighted.

9:10 AM INVITED PAPER

MECHANICAL FATIGUE OF THIN COPPER FOIL: *Harish D. Merchant*[1]; Melvin G. Minor[1]; Y. N. Liu[2]; [1]Gould Electronics, 34929 Curtis Blvd., Eastlake, OH 44095-4001 USA; [2]University of Kentucky, Light Metals Laboratory, Anderson Hall, Lexington, KY USA

Copper foils, 12um to 35um thick, are subjected to the bending/unbending fatigue around a mandrel in the flex and fold modes. Electrodeposited and rolled foils are employed. The strain-based fatigue is evaluated over a wide range of strain amplitudes. The fatigue life versus strain amplitude curve in the high strain amplitude (low cycle) and low strain amplitude (high cycle) regimes is developed for each foil type prior to and following recrystallization anneal. The parameters of Coffin-Manson relationship are utilized to calculate the cyclic strain hardening parameter and fatigue ductility. The initiation and propagation of fatigue crack through the foil thickness and across the sample width are related to the unique fine grain structure for each foil type, pancaked grains for the rolled foil and equiaxed grains for the electrodeposited foil. The variations in electrical resistance, microhardness, grain structure and dislocation structure with fatigue are monitored. The fatal

fatigue failure is associated with an increase in electrical resistance beyond a preassigned threshold which corresponds with the convergence of through-thickness and across-the-width fatigue crack.

9:40 AM INVITED PAPER

A DYNAMIC MODEL FOR SOLDER AND THE PROBLEM OF ACCELERATED LIFE TESTING: *Stan Russell*[1]; Anne Gringals[1]; Ed Clemente[1]; [1]Motorola, Space and Systems Technology Group, 8220 E. Roosevelt St., Scottsdale, AZ 85252 USA

Solder joint reliability under thermal cycling is a key problem in electronic packaging. Accelerated life testing (few cycles larger temperature excursions) is a practical necessity in predicting fatigue life in field environments (many cycles, smaller temperature excursions). Complex solder behavior with marked temperature dwell and cycle time influence at slower frequencies makes this a difficult problem. A dynamic model is presented which couples micro instability of coarsened grain band evolution with changes in macroscopic constitutive behavior. Key features of the model include effects of shear band width to total solder joint thickness, pertinent to small scale design, and frictional resistance at slow load rates. Model fit and interpretation of published data are discussed as well as implications for accelerated life test design.

10:10 AM BREAK

10:20 AM INVITED PAPER

AUTOMATED INSTRUMENTATION FOR DETERMINING THE DAMPING CAPACITY OF MATERIALS AND SMALL STRUCTURES: *James C. Earthman*[1]; [1]University of California - Irvine, Dept. of Chem. and Biochem. Eng. and Mats. Sci., Irvine, CA 92697-2575 USA

Novel instrumentation interfaced to a percussion probe will be presented that can quantitatively determine the damping capacity of material samples and structures including biomedical implant components. The loss coefficient is used to characterize mechanical damping capacity of a material or structure. Values of this property for dental prostheses will be presented and compared with those for natural teeth. It will be shown how these values may be used to evaluate mechanical integrity as well as being used in the assessment and design of prosthetic structures. A further assessment of the measured stress wave response of small structures will also be discussed.

10:50 AM INVITED PAPER

AFM ANALYSIS OF CUMULATIVE FATIGUE DAMAGE IN CU THIN FILMS: Donald Kramer[1]; Alex Volinsky[1]; Gina Sandvick[1]; Yosef Katz[2]; William Gerberich[1]; [1]University of Minnesota, Chem. Eng. and Mats. Sci., 151 Amundson Hall, 421 Washington Ave. SE, Minneapolis, MN 55455 USA; [2]Nuclear Research Center, POB 9001, Negev, Beer-Sheva 84190 Israel

Established techniques for evaluation of slip-step evolution in bulk copper have been applied to micron size thin copper films. Sputter deposited copper films on titanium substrates have been annealed from 230ϒC to 310ϒC and reverse-bend fatigue cycled. Damage rates are identified as the fraction of accumulated plastic strain emerging at the free surface measured by atomic force microscopy. These are compared to bulk samples of copper which have been cold worked and annealed to corresponding temperatures. While the bulk samples can be interpreted in terms of a microstructurally-based low-cycle fatigue law, constraint by the substrate requires additional insight to model thin film behavior. Possible micromechanisms are discussed.

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

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.CBox 7909, Raleigh, NC 27695-7909 USA; R. Thompson, Iowa State University, Ames Laboratory, Ames, IA 50011 USA

Tuesday AM Room: 16A
March 2, 1999 Location: Convention Center

Session Chairs: K. L. Murty, North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695-7909 USA; Thomas J. Mackin, University of Illinois, Dept. of Mech. and Indust. Eng., Urbana, IL 61801 USA

8:30 AM INVITED PAPER

NON-DESTRUCTIVE EVALUATION OF THE EFFECT OF AGING ON TENSILE PROPERTIES OF ALLOY 625: *M. D. Mathew*[1]; K. L. Murty[1]; K. B. S. Rao[2]; S. L. Mannan[2]; [1]North Carolina State University, Dept. of Nuclear Eng., Raleigh, NC 27695-7909 USA; [2]Indira Gandhi Centre of Atomic Research, Mats. Development Division, Kalpakkam, TN 603 102 India

The effect of aging on the tensile properties of Alloy 625, a solid solution strengthened nickel base superalloy, was investigated using the non-destructive Stress-Strain Microprobe (SSM) system. SSM is based on automated ball indentation (ABI) technique, and involves strain-controlled multiple indentations by a small spherical indenter at a single penetration location on the material surface. The technique permits evaluation of tensile deformation parameters such as yield strength, ultimate tensile strength, strength coefficient and strain hardening exponent, as well as elastic-plastic fracture toughness parameter Kjc. Alloy 625 was aged at six different temperatures in the range of 873 to 1173K for 500 hours each. ABI tests were carried out at room temperature and at 473K. The variation of yield and ultimate tensile stresses with aging temperature showed a peak in strength at 973K. The peak stress was two times the strength of the unaged material, whereas the strength after aging at 1173K was nearly equal to that of the unaged material. Microstructural studies showed that the peak in strength resulted from the precipitation of "g" precipitates. The fall in strength due to aging above 973K is attributed to the precipitation of d-phase. ABI is a non-destructive testing technique to determine changes in mechanical properties of materials and components due to aging.

9:00 AM

THE APPLICATION OF PASS THROUGH FLUX FOR NON-DESTRUCTIVE TESTING AND PROCESSING CONTROL OF MAGNETIC SPUTTERING TARGETS : *Hao Zhang*[1]; Jeff Hart[1]; Tim Newport[1]; [1]Tosoh SMD, Inc. , R&D, 3600 Gantz Rd., Grove City, OH 43123 USA

The magnetic permeability and its consistency are the key factors determining the performance of a magnetic sputtering target. The performance of a magnetic target can be significantly improved by decreasing the magnetic permeability and increasing the pass through

flux (PTF), which measures the magnetic field transmitted through a ferromagnetic material. Typically, magnetic permeability is measured on miniature samples by destructive methods. It is inapplicable to measure permeability and its consistency for every target, particularly in manufacturing. In this paper, the correlation among the magnetic permeability, PTF, target thickness, crystallographic texture and fabrication process of Co targets was studied. The permeability and PTF of the Co targets fabricated by different processes were measured, and the 3-D PTF mapping was constructed to show its consistency across the targets. The results showed that the permeability strongly depended on fabrication process, microstructure and texture. The PTF increased with decreasing permeability. By control the microstructure and texture of the Co targets through processing, the permeability and PTF can be optimized. The application of the PTF for non-destructive testing and in-situ processing control in manufacturing was also studied. It has demonstrated that the PTF testing is an effective NDT tool in determining the magnetic properties, microstructure, texture and their consistency in manufacturing, and is an effective tool for quality control.

9:30 AM INVITED PAPER

ESTIMATING FATIGUE LIFETIMES OF DAMAGED COMPOSITES USING THERMOELASTICITY: *Gavin P. Horn*[1]; Peter Kurath[1]; Thomas J. Mackin[1]; [1]University of Illinois, Mech. and Industrial Eng., 140 MEB, 1206 W. Green St. MC 244, Urbana, IL 61801 USA

A new experimental method is presented for quantifying impact damage in composites, rationalizing fatigue data following impact, and for estimating the current state and remaining lifetime in composite materials. The procedure was demonstrated using samples of randomly oriented, continuous glass fiber reinforced polyurethane and epoxy. Composite samples were impact-damaged using an Instron/Dynatup 8250 Drop Tower system. Thermoelastic stress analysis (TSA) was then used to quantify the impact damage by assigning a stress concentration factor to the damaged site. Following impact and TSA imaging, the samples were fatigued to failure using a range of stress amplitudes. The stress concentration factors measured using TSA provided a rational understanding of the resulting fatigue lifetimes. Stress-life predictions were made for a significant range of damage over three decades of life, displaying a correlation coefficient closely resembling that of baseline, unimpacted specimens. In addition, the thermoelastic method was used to detect damage induced by machining test samples. These tests demonstrated the general utility of TSA as a non-destructive method for imaging and quantifying composite damage.

10:00 AM

DYNAMIC MODULUS AND POISSON RATIO MEASUREMENT OF Ti/TiB COMPOSITES BY IMPULSE EXCITATION: *Revti R. Atri*[1]; K. S. Ravichandran[1]; [1]University of Utah, Dept. of Metall. Eng., 135 S 1460 E Rm. 412, Salt Lake City, UT 84112 USA

The elastic modulus, shear modulus and the Poisson ratio of four different Ti-TiB composites, along with monolithic Ti and TiB were measured using the impulse excitation of vibration technique. This dynamic test method is specifically appropriate for the material being studied and requires the use of simple operating procedures. Most importantly this method can be used on geometrically simple specimens and is non-destructive in nature. Three specimens of Ti, TiB and Ti-TiB composites of size 60X15X3mm were cut by EDM. The Ti-TiB composite compositions consisted of Ti-20TiB, Ti-40TiB, Ti-60TiB, and Ti-80TiB. The elastic modulus for monolitic Ti and TiB were 116.8 and 426.0, respectively. In the composite materials there was an increase in elastic modulus and shear modulus with increasing volume fraction of TiB. The average shear modulus for monolithic Ti and TiB were 44.1 and 185.4, respectively. The Poisson ratio value was 0.32 for monolithic Ti and 0.15 for TiB and decreased with increasing volume fr action fo TiB in the composite materials. The values of elastic modulus measured from impulse excitaion were in good agreement with experimental data.

REACTIVE METALS: General Session I

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: John N. Hryn, Argonne National Laboratory, 9700 S. Cass Ave., Bldg. 32, Argonne, IL 60439 USA

Tuesday AM Room: 5B
March 2, 1999 Location: Convention Center

Session Chairs: John N. Hryn, Argonne National Laboratory, Energy Systems Div., Argonne, IL 60439 USA; James A. Sommers, Oremet Wah Chang, Albany, OR 97321 USA

8:30 AM

SYNTHESIS OF INTERMETALLIC COMPOUNDS BY REDUCTION OF MULTICOMPONENT SOLUTIONS OF LIQUID AMMONIA: *Hongmin Zhu*[1]; Donald R. Sadoway[1]; [1]Massachusetts Institute of Technology, Dept. Mats. Sci. and Eng., 77 Massachusetts Ave., Rm 8-109, Cambridge, MA 02139-4307 USA

Sodium dissolves in liquid ammonia to give Na^+ and a solvated electron. The result is a medium that is very potent for metallothermic reduction. The production of tantalum powder by reaction of a solution of $TaCl_5$ dissolved in liquid ammonia with a solution of Na dissolved in liquid ammonia has been demonstrated. Beyond this, co-precipitation/co-reduction of metals by first dissolving multiple compounds of the metals in liquid ammonia and then reducing the solution to promote the precipitation of mixed metal product has been demonstrated. Various intermetallic compounds have been produced in this manner. Sponsorship of the research from Cabot Performance Materials is gratefully acknowledged.

8:55 AM

APPLICATION OF LITHIUM IN MOLTEN SALT REDUCTION PROCESSES: *K. V. Gourishankar*[1]; E. J. Karell[1]; [1]Argonne National Laboratory, Chemical Technology Division, 9700 S. Cass Ave., Argonne, IL 60439 USA

At the Argonne National Laboratory (ANL), we have developed a pyrochemical process that uses lithium to reduce oxide spent nuclear fuel. The reduction is carried out at 650°C in a molten salt (LiCl) bath. Li_2O, produced during the reduction of the fuel, dissolves in the molten salt. At the end of the reduction step, the lithium is regenerated from the salt by an electrowinning process. The lithium and the salt from the electrowinning step are then reused for reduction of the next batch of oxide fuel. The process cycle has been successfully demonstrated on a large-scale in a specially designed pyroprocessing facility. The process demonstrates the applicability of lithium as a reductant in a molten salt system and can be adapted for the extraction of other metals. An important feature of this process is that the salt and the lithium are recycled for use in the next reduction cycle. This presentation will describe the role of lithium in molten salt reduction processes with specific reference to our process that has been developed for the reduction of actinide oxides in spent nuclear fuel.

9:20 AM

A SPECTROSCOPIC AND ELECTROCHEMICAL STUDY OF TITANIUM ELECTROREFINING: *Luis Ortiz*[1]; Donald R. Sadoway[1]; [1]Massachusetts Institute of Technology, Dept. Mats. Sci. and Eng., 77 Massachusetts Ave., Rm 8-109, Cambridge, MA 02139-4307 USA

Metallothermic reduction of $TiCl_4$ produces commercial grade titanium in the form of sponge. For microelectronics applications, high-purity metal can be produced from sponge by electrorefining in a melt composed of chlorotitanates dissolved in alkali chlorides. The relevant physical chemistry of the electrorefining process is under investigation. Melt chemistry is being studied by Raman spectroscopy. Electrode reactions are being studied by various transient techniques including a.c. voltammetry and electrochemical impedance spectroscopy. Electrolysis trials are being conducted in laboratory-scale cells. Recent results will

be reported in conference. Sponsorship of the research from The ALTA Group, a Johnson Matthey company, is gratefully acknowledged.

9:45 AM
SENSORS FOR USE IN SYSTEMS CONTAINING MULTIPLE REACTIVE METALS: *Jeffrey W. Fergus*[1]; [1]Auburn University, Mats. Research and Education Center, 201 Ross Hall, Auburn University, AL 36849 USA

Many reactive metals form ionic compounds that can be used as electrolyte or electrode materials in electrochemical sensors. Such sensors can conveniently be used to measure the concentration of a particular reactive element in a non-reactive environment. When multiple reactive elements are present, however, competitive exchange reactions can occur and lead to instability of the sensor and/or mixed response of the sensor. In this paper, some of the issues and approaches involved in developing sensors for use in systems containing multiple reactive metals will be discussed. Examples include magnesium + strontium or magnesium + sodium in molten aluminum, antimony in molten zinc and barium + cesium vapor.

10:10 AM BREAK

10:20 AM
PRODUCTION OF Ti-Al ALLOY-ALUMINA COMPOSITE PARTICLES: *P. C. Maity*[1]; [1]National Institute of Foundry and Forge Technology, Foundry Technology Dept., P.O. Hatia, Ranchi, Bihar 834 003 India

Titanium dioxide (TiO_2) particles (2 wt.%) were incorporated into commercially pure aluminum melt at 700°C by vortex method. After complete addition of the particles, the melt was treated with 0.3 wt.% hexachloroethane to extract the reaction products of Al and TiO2. The extracted particles contained Ti - 37%, Al - 15% and Al_2O_3 - 48% approximately.

10:45 AM
SYNTHESIS AND CASTING OF A LITHIUM-BISMUTH COMPOUND FOR AN ION-REPLACEMENT ELECTROREFINER: *Sean M. McDeavitt*[1]; [1]Argonne National Laboratory, Chemical Technology Division, 9700 S. Cass Ave., Argonne, IL 60439 USA

Recent ion replacement electrorefining experiments at ANL required the lithium-bismuth intermetallic Li_3Bi. Approximately 3.5 kg of Li_3Bi were synthesized using a high-temperature induction casting furnace. Small-scale (20 g) experiments were used to develop the synthesis method. Four larger-scale castings (500 to 1250 g) were completed by combining high purity lithium and bismuth in a tantalum crucible. Pure Li and Bi metals have relatively low melting points (Li: 180.5°C, Bi: 271.3°C), but the intermetallic has a relatively high melting point (1145°C). The metals were heated slowly to melt the charge. Upon melting of the bismuth, the formation reaction (3Li + Bi -> Li_3Bi) proceeded vigorously and the furnace power was temporarily turned off at this time. After several minutes, the tantalum crucible would stop glowing and the furnace temperature would be increased to 1200°C to melt and homogenize the compound. Liquid Li_3Bi was cast into cold stainless steel molds.

11:10 AM
MICROSTRUCTURAL DEVELOPMENT AND EVOLUTION IN UAlx, U-Al-Si, AND U-Al-Ca-O ALLOYS FOR THE DISPOSITION OF DOE SPENT NUCLEAR FUELS: *Thad M. Adams*[1]; Harold B. Peacock[1]; Fred C. Rhode[1]; Natraj C. Iyer[1]; [1]Westinghouse Savannah River Company, Savannah River Technology Center, Bldg. 773-41A, Rm. 151, Aiken, SC 29808 USA

The melt-dilute treatment technology program is focused on the development and implementation of a treatment technology for diluting highly enriched (>20% ^{235}U) aluminum spent nuclear fuel to low enriched levels (<20% ^{235}U and qualifying the LEU SNF form for geologic repository storage. In order to reduce the enrichment of these assemblies prior to ultimate geologic repository disposal, the melt-dilute technology proposes to melt these SNF assemblies and then dilute with additions of depleted uranium. The emphasis within the development program to date has been on determining the process definition and basis, developing process cycle options, and demonstrating the versatility and adaptability of the process for the treatment of all types of U-Al SNF (UAl_x, Al-U_3O_8, and Al-U_3Si_2). In determining the process definition and basis a wide range of alloys, representative of those expected in the SNF form, have been fabricated an d their product characteristics, namely microstructure, homogeneity, phase composition, etc. have been analyzed using SEM/EDS and XRD. The development of ternary isothermal sections for the U-Al-Si system using the standard Gibbs triangulation technique with validation from experimental solidification and dissolution data has been performed. Lastly, thermodynamic calculations assessing the possible reaction paths in the U-Al-O system with additional dissolution kinetics experiments using depleted U_3O_8 and Al or Al-Ca alloys have been conducted with the Al-Ca alloy being more effective at U_3O_8 reduction. Additionally, partitioning during alloy solidification of the radionuclides species (Cs, Tc, Pd, Sr, Zr, Mo, Y, Ce, Se, Te) which are present from the fission process in minor concentrations has been investigated as well as the microstructural and phase evolution of these alloys under repository storage conditions.

11:35 AM
MICROSTRUCTURE OF THE ZIRCONIUM-8 WT% STAINLESS STEEL ALLOY: *Daniel P. Abraham*[1]; James W. Richardson[2]; [1]Argonne National Laboratory, Chem. Tech. Div., Bldg. 205, Room A167, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave., Argonne, IL 60439 USA

The Zirconium-8 wt% Stainless Steel (Zr-8SS) alloy is being developed by Argonne National Laboratory as a metal waste form to contain radioactive isotopes isolated from metallic spent nuclear fuel. The microstructure of the Zr-8SS alloy has been examined by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The lattice parameters and volume contents of phases have been obtained by neutron diffraction. The major phase in the as-cast alloy is α-Zr; small amounts of the $Zr_3(Fe,Ni)$, $Zr_2(Fe,Ni)$, $Zr_2(Fe,Cr)$ and $Zr(Fe,Cr)_2$ intermetallics are also present in the alloy. On heat treatment, the amount of α-Zr, $Zr_2(Fe,Ni)$ and $Zr_2(Fe,Cr)$ decreases, whereas the amount of $Zr_3(Fe,Ni)$ and $Zr(Fe,Cr)_2$ increases. The relative stabilities of the various phases will be discussed with reference to the Zr-Fe phase diagram.

STRUCTURAL SILICIDES: Processing, Mechanical Properties, Precipitation

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 50011 USA

Tuesday AM — Room: 16B
March 2, 1999 — Location: Convention Center

Session Chairs: C. T. Liu, Oak Ridge National Laboratory, Metals and Ceramics Div., Oak Ridge, TN 37831-6115 USA; J. Mundy, 10720 Game Preserve Road, Gaithersburg, MD 20879-3106 USA

8:30 AM INVITED PAPER
A REVIEW OF NIOBIUM SILICIDE-BASED IN-SITU COMPOSITES: *B. P. Bewlay*[1]; M. R. Jackson[1]; [1]GE Corporate Research and Development, Niskayuna, NY 12309 USA

This paper will describe processing and properties of high-temperature Nb silicide based in-situ composites. These composites consist of

high-strength Nb-based M_3Si and M_5Si3 silicides together with a modest strength, high-toughness Nb-based metallic phase. Preliminary in-situ composites were derived from the binary Nb- Nb_3Si eutectic and Nb Nb_5Si_3 eutectoid reactions. While these composites displayed an attractive balance of high- and low-temperature mechanical properties, they possessed very poor environmental resistance. In order to obtain a more complete balance of properties, the more recent composites are derived from higher order alloys that contain additions such as such as Ti, Hf, Mo, Al and Cr. These in-situ composites have been produced by a number of approaches including arc melting, direction solidification, and physical vapor deposition. The role of secondary processing operations, such as extrusion and forging, has also been studied. This paper will describe composite microstructures, room-temperature fracture toughness and elevated-temperature property data (tensile strength, creep performance, and oxidation behaviour). These composites have promising high-temperature strength and oxidation resistance at temperatures of 1315ϒC (2400F) when compared with other intermetallic-based systems. The high temperature mechanical properties and oxidation behavior will be also compared with the most recent Ni-based superalloys.

9:00 AM INVITED PAPER

AN OVERVIEW ON THE PROCESSING OF MOLYBDENUM SILICIDES AND THEIR COMPOSITES: *S. C. Deevi*[1]; [1]Philip Morris USA, Development and Eng. Center, Richmond, VA 23234 USA

Silicides based on molybdenum have received considerable attention as opposed to the other structural ceramics due to their excellent oxidation resistance, and ductility at high temperatures. Silicides allow incorporation of reinforcements, and are stable with many additives even at high temperatures allowing the process engineer to design a structural ceramic utilizing the unique metallic properties of molybdenum silicides. In this paper, we present the processing methodologies adopted for the processing of molybdenum silicides using Mo and Si powders to obtain $MoSi_2$, Mo_5Si_3, and other composites, and compare them with the conventional techniques such as casting, hot pressing and hot-isostatic pressing. An attempt will be made to generalize the features of processing among the different techniques, and their effect on the fracture toughness, oxidation strength, pesting, and high temperature creep of the silicides. In addition, we will also present the unique aspects of composites based on $MoSi_2$-Si_3N_4 composites, and the processing challenges involved to make the composites a commercial reality.

9:30 AM INVITED PAPER

DEVELOPMENT OF MOLYBDENUM SILICIDES + MOLYBDENUM COMPOSITES FOR HIGH-TEMPERATURE STRUCTURAL APPLICATIONS: *M. G. Mendiratta*[1]; P. R. Subramanian[1]; T. A. Parthasarathy[1]; J. Simmons[1]; R. Wheeler[1]; D. Dimiduk[2]; [1]UES, 101-000, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Mats. and Manufact. Directorate, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Research is in progress on the Mo_5SiB_2 + Mo_3Si + α-Mo in-situ composites for structural applications in the 800-1400ϒC temperature range. Composites of various compositions are prepared by casting of small buttons for investigations of phase relations, microstructural evolution and oxidation behavior. Large cast billets are hot-extruded for mechanical property determinations. The mechanical behavior studies include measurements of toughness, strength, ductility and creep from room temperature to 1400ϒC. In addition, alloying studies are in progress involving additions of Cr, V, Nb, W, Ge, and Al to alter the equilibrium phases to improve oxidation resistance. Results will be presented and discussed in relation to other high-temperature material systems currently under development for structural applications. UES, Inc.; Research being performed under Contract F33615-96-C-5258.

10:00 AM BREAK

10:20 AM

FORMATION OF SILICIDE PRECIPITATES IN Nb-Si-BASED IN-SITU COMPOSITES: *B. P. Bewlay*[1]; R. J. Grylls[2]; H. L. Fraser[3]; [1]GE Corporate Research and Development, 1 Research Circle, Schenectady, NY 12309 USA; [2]GE Aircraft Engines, 1 Neumann Way, Cincinnati, OH 45215 USA; [3]The Ohio State University, Dept. of Mats. Sci. and Eng., Columbus, OH 43210 USA

In-situ composites based on Nb-Si alloys are being considered as future materials for high- temperature structural applications. The directionally-solidified in-situ composites studied in this work possess a microstructure consisting of dendrites of a niobium solid solution, surrounded by a eutectic mixture of niobium and Nb_3Si. In both the as-cast and heat-treated conditions, precipitates are observed within the niobium dendrites, and the object of this paper is to provide a detailed characterization of these precipitates, both in binary and higher-order hypoeutectic alloys. It is found that the precipitates have the Nb_3Si stoichiometry, but they do not possess the stable Nb_3Si crystallography. The precipitates possess a metastable orthorhombic crystal structure which is related to the niobium matrix via a simple orientation relationship. The precipitates are typically seen as semi-coherent faceted needles, with dimensions ranging from 2nm to several microns, depending on the heat-treatment. It will be shown that the precipitates form on cooling from the heat-treat temperature due to the reduction in Si solubility in the niobium on cooling. Hardness data indicate that these precipitates provide some strengthening of the niobium matrix, and the possibility for using these precipitates as a strengthening phase will be discussed.

10:40 AM

OBSERVATIONS OF THE FRACTURE BEHAVIOUR OF Nb-Si IN-SITU COMPOSITES USING CROSS-SECTIONAL ELECTRON MICROSCOPY: *Bernard P. Bewlay*[1]; R. J. Grylls[2]; H. L. Fraser[3]; [1]GE Corporate Research and Development, 1 Research Circle, Schenectady, NY 12309 USA; [2]GE Aircraft Engines, 1 Neumann Way M85, Cincinnati, OH 45215 USA; [3]The Ohio State University, Dept. of Mats. Sci. and Eng., Columbus, OH 43210 USA

Composites based on Nb-Si alloys are attractive candidates for use as structural materials at the very high temperatures required for future generation aircraft engines. Binary Nb-Si alloys are limited by their low room-temperature fracture toughness, but in-situ composites have been developed which have fracture toughness values of > 20 $MPam^{1/2}$. In this study the fracture behavior of a range of in-situ composites has been investigated using cross-sectional transmission electron microscopy (CTEM) of the fracture surface of specimens subjected to four-point bending. The composites studied in this work were produced by directional solidification, which gives an in-situ composite microstructure consisting of Nb_3Si type dendrites surrounded by a eutectic of a niobium solid solution and a niobium silicide. Using CTEM it is possible to view directly the micromechanisms of fracture, including the details of slip transmission between phases, microcracking, crack bridging, ductile-phase pull-out, and interfacial debonding. Using CTEM it has been possible to observe significant differences in the fracture behavior of these composite materials, and to correlate these observations with the mechanical properties.

11:00 AM

PHASE STABILITY AND HARDNESS OF Mo-SILICIDE ALLOYS WITH ADDITIONS OF Ni, Al, AND B: *J. W. Cohron*[1]; E. P. George[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831 USA

A preliminary investigation of phase stability and hardness changes as a function of Ni, Al and/or B in Mo-silicides has been performed. Phases and their relative amounts were obtained using X-ray diffraction (XRD). Hardness measurements were made on each alloy. The propensity for crack formation, coupled with the measured hardness numbers, gave an indication of the relative toughness of each alloy. In the absence of B and with increasing additions of Al and Ni, hardness and cracking decreased. Presumably, this "softening" is due to the increasing formation of the NiAl phase, which is evident in the XRD patterns. With the addition of B, hardness and crack formation increased. This decrease in toughness has been associated with the formation of a brittle $Mo_5(Si,B)_3$ phase, again, as indicated by the XRD data. Primary and secondary phases and their relative stability are discussed as a function of alloy composition. Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

11:20 AM
SYNTHESIS AND PROPERTIES OF IN-SITU (Mo,W)Si_2 COMPOSITES: *Dae Kyu Jang*[1]; Reza Abbaschian[1]; [1]Korea Institute of Geology, Mining and Mats., Resources Utilization & Mats. Division, 30, Kajung-Dong, Yusung-ku, Taejon 305-390 Korea

(Mo,W)Si_2 composites were prepared by vacuum hot pressing from elemental Mo, W and Si powders at various temperatures. The Microstructure and properties of these materials were characterised by using X-ray diffraction, scanning electron microscopy, energy dispersive x-ray spectroscopy and Vicker's technique. The synthesis of in-situ (Mo,W)Si_2 composites did not show the presence of any unreacted elements. Microstructural and x-ray analysis showed that (Mo.W)Si_2 alloys formed. The grain size of the (Mo,W)Si_2 was relatively uniform. The grain size of the (Mo.W)Si_2 composites is smaller than that in monolithic $MoSi_2$. The room temperature hardnesses of the composites increase with increasing reaction temperature.

11:40 AM
STRENGTH AND FRACTURE TOUGHNESS OF CAST Mo-12Si-8.5B (AT. %) INTERMETALLICS: *Joachim H. Schneibel*[1]; C. T. Liu[1]; Cecil A. Carmichael[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

Alloys with the composition Mo-12Si-8.5B (at. %) were prepared by arc-melting. They contained approximately 40 vol.% of α-Mo inclusions in a brittle matrix of 30 vol.% Mo3Si and 30 vol.% Mo_5SiB_2 (T2). The room temperature flexure strength and fracture toughness of the as-cast alloys were 450 MPa and 7 MPa $m^{1/2}$, respectively. A heat treatment of 1 day at 1873 K in vacuum increased the room temperature fracture toughness to 10 MPa $m^{1/2}$. Consistent with ductile phase toughening, the fracture surfaces revealed plastic deformation as well as debonding of the α-Mo. Testing at 773 K resulted in flexure strengths as high as 700 MPa. All these experiments suggest that the mechanical properties of the α-Mo solid solution are critical in determining the mechanical properties of these Mo-Si-B alloys. This research was sponsored by the Fossil Energy Advanced Research and Technology Development (AR&TD) Materials Program, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Nanostructured Materials

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

Tuesday AM Room: 7B
March 2, 1999 Location: Convention Center

Session Chairs: Delcie R. Durham, National Science Foundation, 4201 Wilson Blvd., Arlington, VA 22230 USA; F. Huisken, Max-Planck-Institut für Stromungsforschung, Brunsenstr 10 D-37073, Göttingen Germany

8:30 AM INVITED PAPER
NANOSTRUCTURED COATINGS: *J. M. Aikens*[1]; H. Sarkas[1]; R. W. Brotzman[1]; [1]Nanophase Technologies Corporation, 453 Commerce St., Burr Ridge, IL 60521 USA

The development of nanostructured coatings with improved abrasion resistance, EM shielding, thermal conductivity, refractive index, and transparency requires careful manipulation of material composition, nano-structure size, and composite structure. The selected materials must be processable and yield the desired physical properties. The correct nanostructure size is important because often multiple physical properties are desired, i.e., conductivity or abrasion resistance with transparency. The correct composite structure must be selected for the desired physical property from uniform surface distribution for abrasion resistance to connected structures for electrical conductivity. Nanostructured coatings formed by the addition of dense, crystalline nanoparticles to a continuous second phase will be discussed. The particles are manufactured by gas phase condensation and the continuous phase varies from epoxy polymers to silica. Particle size, material composition, and surface treatments are manipulated to control composite properties and structure. Transparent/conductive, transparent/abrasion resistant, and transparent/high refractive index coatings will be discussed with respect to a broad range of market applications including electronic materials, optical and magnetic devices, catalysts, and structural materials.

8:55 AM
THIN LIQUID FILM ELECTRO SPRAYING- A NOVEL COATING TECHNOLOGY: *Gunnar Sorensen,* Institute of Physics and Astronomy, Aarhus University, 8000 DK Aarhus C, Denmark

A novel coating technology has been developed for suspensions and solutions in a dielectric medium. A thin liquid film of this medium is flowing to a spraying nozzle at a high voltage of 2-20 kV. A glass cylinder acts as an electrostatic lens surrounding the spraying system which in a simple version can be a modified hypodermic syringe needle. Ultrafine powder down to 0.2 micron can be collected on a grounded substrate. Various spraying nozzæes will be discussed, and examples of depositing ultrafine powders on surfaces will be presented. Examples comprise deposition of nano-diamond powder, carbon 60 and the biomolecule azoadenine for sputtering C-N compounds

9:10 AM INVITED PAPER
LASER-INDUCED PRODUCTION AND CHARACTERIZATION OF CRYSTALLINE SILICON NANOPARTICLES WITH NARROW SIZE DISTRIBUTION: *F. Huisken,* M. Ehbrecht, and B. Kohn, Max-Planck-Institut für Strömungsforschung, Bunsenstr. 10, D-37073 Göttingen, Germany

Silicon clusters and nanoparticles are produced by CO_2-laser-induced gas phase reactions in a flow reactor. In contrast to conventional techniques, the particles are expanded, directly after production, through a conical nozzle into a high vacuum chamber and then transferred into a molecular beam machine where they are analyzed in situ with a time-of-flight mass spectrometer. The analysis reveals that the flow reactor emits, besides small silicon clusters, also high-purity silicon crystallites with diameters between 1 and 10 nm. It is found that the particles* velocity strongly correlates with their mass. This feature and the fact that the particles are produced in a pulsed mode (by using a pulsed CO_2 laser) enable us, by introducing a chopper into the cluster beam, to considerably reduce the dispersion of their size distribution and to perform size-selected low-energy cluster deposition on various substrates. High resolution transmission micrographs demonstrate the capabilities of the new apparatus. The monodispersed silicon films have been further characterized by studying their luminescence and Raman scattering behavior. As predicted by theoretical models, the peak of the luminescence curve shifts with decreasing particle size to smaller wavelengths (higher energies). In addition, it is found that the efficiency curve has a sharp maximum for particles with 4 nm diameter.

9:35 AM INVITED PAPER
RECENT DEVELOPMENT IN LASER-ASSISTED CHEMICAL VAPOR DEPOSITION OF NANOPOWDERS AND COATINGS: *Rodica Alexandrescu*[1]; [1]National Institute for Laser, Plasma and Radiation Physics, P.O. Box MG-36, Bucharest Romania

Laser-assisted chemical vapor deposition (CVD) method plays an important role in producing solid-state materials because of its potentialities to be applied to a wide variety of compounds (nitrides, carbides and metal-based products) together with its ability to control the chemical and physical properties of the new produced material. Although related by similar experimental technique, the differences between thin film deposition and the powder synthesis by laser CVD rely in the

magnitude of the growth time (or residence time in the laser beam) and in the density of nucleated particles. Fast heating and cooling processes and high particles densities are characterized nanophase powder synthesis. In this paper the synthesis of iron-based and carbon nitride napophase powders and thin films are discussed, in order to demonstrate the multiple possibilities provided by laser assisted CVD processing for the production of materials with tailored properties. For iron-based deposition, the gas-phase decomposition of iron pentacarbonyl is an area of current interest. Iron carbides fine powders were obtained by the CO2 laser pyrolysis of sensitized (SF6) mixtures and different carrier gases (C2H4, C2H2) for Fe(CO)5 vapors: non-aggregated particles with mean size in the range 6 nm were obtained for acetylene/iron pentacarbonyl mixtures. As concerning iron-based film deposition, a comparative analysis of coatings obtained by photolytic and thermal laser-induced processes indicate that specific growth mechanism influence the film structure and chemical composition. The carbon nitride thin film and nano powders were synthesized from hydrocarbon/ammonia mixtures. The CNx thin films were obtained at two irradiation wavelengths on different substrates. The CN powders were produced by the IR pyrolysis of acetylene/ammonia/nitrous oxide mixtures. The different chemical and crystallographic phases of the obtained materials are discussed.

10:00 AM INVITED PAPER

AN INVESTIGATION OF THE RELATIONSHIP BETWEEN STRUCTURE, SURFACE ROUGHNESS AND HARDNESS BY TiN THIN FILMS DEPOSITED BY RF SPUTTERED PVD PROCESS: *Delcie R. Durham*[1]; Carl Carney[1]; [1]National Science Foundation, 4201 Wilson Blvd., Room 530, Arlington, VA 22230 USA

The growth of a thin film on a substrate is dependent upon the process, the substrate preparation and any additional “drivers” to the system. An ECR plasma enhanced reactive sputtering technique was used to deposit the TiN films of 1 - 2 micrometers thickness on Inconel 718 polished substrates. The PVD process is enhanced using Electron Cyclotron Resonance (ECR) which allows broader options in selecting the basic processing conditions. The properties of the TiN thin films have been shown to be dependent upon the temperature of the substrate, substrate bias, flow conditions within the plasma and the pressure in the chamber. For example, lower deposition temperatures are desirable to reduce substrate microstructural changes and to reduce the thermal stresses developed in the films. Typically, lower pressures are also advantageous, since they provide for longer mean free path movement of the species being deposited. A 24 designed experiment was conducted to investigate the individual and combined effects of substrate temperature, substrate bias, system pressure and nitrogen flow. An additional investigation included the effects of microwave power. The films obtained were polycrystalline, with varying crystallographic structures, grain size, and surface roughness, and hardness. After deposition, the films were characterized using the x-ray diffractometer, the SEM and the AFM, a Digital Instruments nano-indentor as well as microhardness tests and standard profilometry. Characterization of the film surface was accomplished by standard profilometry and by atomic force microscopy (AFM) techniques. RMS roughness and fractal roughness information was obtained for the films produced. The fractal dimension for the film surface was determined by AFM methods, providing data at the nanoscale level. The fractal dimension can provide significantly more detail regarding fine variations in the surface topography than RMS information can provide. When the RMS roughness and the fractal number data were compared for the set of tests, there is no clear correlation between these two methods of describing surface roughness. There appears to be a stronger relationship between fractal roughness and hardness than between RMS roughness and hardness. The relationship with crystal orientation is not strong, although when [200] growth was suppressed, nanohardness of over 30 micrometers was obtained. Those films which exhibited fine, or very fine columnar grains had the higher hardness as expected.

10:25 AM BREAK

10:40 AM INVITED PAPER

DESIGN OF NANOSTRUCTURED THIN FILMS FOR TRIBOLOGICAL APPLICATIONS: *H. Holleck*[1]; [1]Institut fur Material-und Festkorperforschung 1, Kemforschungszentrum Karlsruhe, Postfach 3640, Karlsruhe 1 W-7500 Germany

Nanoscaled coatings for tribological applications can be subdivided into nanostructured multilayer films, nanomodulated superlattice films, nanocrystalline films, nanostabilized single and multilayer films and nanograded films. Aside from materials selection and deposition characteristics, the interface volume, grain size, single layer thickness, surface and interface energy, texture and the epitaxial stress and strain are principal factors determining constitution, properties and performance of these coatings. The functional and structural design of multilayer coatings can result in tailored multifunctional coatings as it is shown for nanoscaled TiN/TiAlN, TiC/TiB2 , TiN/MoS2 and TiC/C multilayer films. The influence of the modulation of composition, structure, stress and strain in so called superlattice films on the properties is discussed in dependence on the modulation period and the material selection. Nanocrystalline coatings (e.g. TiC/TiB2 , TiC/C or TiN/MoS2 films) are well suited to combine film materials with similar or extremely different bonding characteristics to a multifunctional composite coating. New structures for thin film materials or specific film textures can be stabilized by epitaxial growth in nanostabilized multilayer coatings. The stabilization of fcc AlN, fcc SiC, crystalline C3N4 and wurtzite type BN are examples for materials which operate successfully or are in the state of development. New metastable nanocrystalline films can be deposited by vapor quenching. A thermodynamic and kinetic modeling results in new film materials (e.g.TiBCN), tailored with respect to constitution, properties and performance.

11:05 AM

DIAMOND-LIKE NANOCOMPOSITE COATINGS FOR SPACE APPLICATIONS: *Craig A. Outten*[1]; Daniel Kester[1]; Chandra Venkatraman[1]; Donald Bray[1]; Chris Halter[1]; [1]Advanced Refractory Technologies, 699 Hertel Ave., Buffalo, NY 14207 USA

In recent studies, Diamond-Like Nanocomposite (DYLYN®) coatings were found to possess resistance to atomic oxygen, tailorable electrical conductivity, and excellent mechanical properties. Therefore, several potential applications are anticipated on critical optical, electronic, thermal, and mechanical components for commercial and military Low Earth Orbit spacecraft systems. The coatings were deposited with a proprietary plasma-assisted chemical vapor deposited process. In particular, electrically conductive coatings (as low as 1800 ohms per square) were deposited on flexible plastic substrates, e.g. Kapton® and Mylar®. The coated substrates were subjected to ground based atomic oxygen testing. The results indicated that DYLYN® coatings reduced erosion yields on Kapton® and Mylar® by 130X and 50X, respectively. Furthermore, the coatings on plastics exhibit high flexibility and were bent to a radii less than 1/32" with no damage to the coating. Further mechanical testing is presented to demonstrate the coating’s durability. A brief discussion is included of the DYLYN® coatings which were flown on the NASA Optical Properties Monitor (OPM) Experiment.

11:20 AM INVITED PAPER

NANOSCALE CONTROLLED GROWTH, PROPERTIES, AND NOVEL DEVICE APPLICATIONS OF EPITAXIAL THIN FILMS OF CONDUCTIVE MAGNETIC OXIDE SrRuO3: *Chang-Beom Eom*[1]; [1]Duke University, Dept. of Mech. Eng. and Mats. Sci., Durham, NC 27708 USA

Oxide materials possess an enormous range of electrical, optical, and magnetic properties. For instance, insulators, high quality metals, dielectrics, ferroelectrics, piezoelectrics, semiconductors, ferromagnetics, transparent conductors, colossal magnetoresistance materials, superconductors, and nonlinear optic materials have all been produced using oxide materials. Therefore, thin films and heterostructures of oxide materials have great potential for novel device applications. For many of these device applications, it is necessary to have epitaxial growth of conductive oxide thin films in a single heterostructure. We have grown epitaxial thin films and heterostrcutures of conductive magnetic oxides, SrRuO3(1) in situ by 90Ῠ off-axis sputtering and pulse laser deposition. SrRuO3 is a distorted perovskite with a GdFeO3-type structure and undergoes a ferromagnetic transition at 160K. Single crystal epitaxial SrRuO3 thin films were obtained on vicinal (001) SrTiO3 substrates with a large miscut angle and miscut direction close to the [010] axis. Scanning tunneling microscopy revealed that the growth mechanism changed

from two dimensional nucleation to step flow growth as the miscut angle increased(2). The electrical transport, magnetoresistance and novel ferroelectric(3) and magnetic device applications of the conductive ferromagnetic oxide thin films and heterostructures will be discussed. (1) C. B. Eom et al., Science, 258, 1766 (1992); (2) R.A. Rao, Q. Gan, and C.B. Eom, Appl. Phys. Letts. 71, 1171 (1997); (3) C. B. Eom et al., Appl. Phys. Letts., 63, 2570 (1993). This work was supported by the ONR Grant No. N00014-95-1-0513, NSF Grant No. DMR 980244, the NSF Young Investigator Award (CBE) and the David and Lucile Packard Fellowship (CBE).

11:45 PM

MECHANICAL AND TRIBOLOGICAL PROPERTIES OF NANOSTRUCTURED DIAMOND FILMS ON Ti-6Al-4V SUBSTRATES: *Shane A. Catledge*[1]; Yogesh K. Vohra[1]; [1]University of Alabama at Birmingham, Dept. of Physics, Birmingham, AL 35294-1170 USA

Microwave plasma chemical vapor deposition (MPCVD) was used to deposit diamond films onto Ti-6Al-4V substrates at high density plasma processing conditions which can be tailored to result in a range of microstructures ranging from crystalline to nano-crystalline diamond. Micro-Raman spectroscopy, glancing-angle x-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to characterize the structure of the films. The average surface roughness of the nano-crystalline films as measured by profilometry was 40 nm and the average hardness/ elastic modulus of the films as measured by nano-indentation was 90 GPa/ 700 GPa. The adhesion of the films produced by our high density plasma processing technique is significantly better than films produced by conventional processing conditions as qualitatively observed by the lack of spallation for substrate temperatures up to 850 ?C. Further quantitative adhesion and wear-resistance tests were performed on the diamond films. The improved adhesion, low surface roughness, and high hardness of these diamond films make them attractive for a variety of tribological applications.

12:00 PM

NANOSCALE MECHANICAL, FRICTION, AND WEAR PROPERTIES OF MATERIALS FOR MEMS APPLICATIONS: *Oden L. Warren*[1]; L. L. Kuhn[1]; D. A. Crowson[1]; T. Wyrobek[1]; [1]Hysitron, Inc., 5251 West 73rd St., Minneapolis, MN USA

The primary goal of many emerging device technologies, such as microelectromechanical systems (MEMS), is to achieve macroscale functionality from microscale components. Further miniaturization of these device technologies will result in progressively smaller clearances between moving parts, which will inevitably increase the occurrence of tribological contacts between surfaces. Because MEMS devices often lack sufficient reserve power to shear adhesive junctions, even the first contact event can lead to irreversible formation of an interface. Therefore, optimization of device design parameters will eventually require knowledge of mechanical, friction, and wear properties of surfaces down to the atomic scale. Quantification of these properties down to the nanoscale is now achievable due to recent improvements in depth-sensing nanoindentation instrumentation. Here, we discuss new developments in lateral displacement actuation and lateral force measurement with a capacitance-based nanoindenter. Coupling this novel self-contained technology to a scanning probe microscope results in unprecedented utility for tribological and mechanical properties investigations at the nanoscale. Results of case studies relating to materials for MEMS applications will also be presented.

12:15 PM

SYNTHESIS OF THIN FILMS OF SILICON NANOPARTICLES WITH SIZE CONTROLLED BY REACTION PARAMETERS: *S. Botti*[1]; R. Coppola[1]; E. Massetti[1]; [1]ENEA, Dipartimento Innovazione, Divisione Fisica Applicata, Centro Ricerche Frascati, Rome Italy

Nanoscale silicon is attracting considerable interest due to its electronic quantum-size effect. The CO2 laser induced pyrolisis of gas phase reactants has proved to be a viable tool for producing ultrafine particles and has been applied to the synthesis of nanosized silicon particles. The powders synthesis occurs by heating the reactant gases up to the dissociation limit with a 500 W CO2 laser. The reactants interact in a laminar flow confined by a carrier beam (He, Ar) in a collision environment. For the silicon synthesis the silane provides the coupling with laser radiation due its strong absorption at 10.6 ?m. The silicon particle growth is controlled by on-line optical technique and the silicon particle diameter has been scaled in a wide size range (2-100 nm). By this technique it is possible also to fabricate thin film comprised of size selected nanoparticles. By a secondary nozzle the reaction products are extracted at right angle to both the gas flow and laser beam and are expanded in a differential chamber before the deposition on heated substrate. Photoluminescence properties of thin film and powders synthesized

SYNTHESIS OF LIGHTWEIGHT METALS III: Semi-Solid 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, Structural Mats. Ctr, Farnborough, Hampshire GUI14OLX UK; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Neqev Israel; P. G. McCormick, University of W. Australia, Res. Ctr for Adv. Min. & Mat. Proc., Nedlands, W.A. 6907 Australia

Tuesday AM Room: 10
March 2, 1999 Location: Convention Center

Session Chairs: Robert Dax, Concurrent Technologies Corporation, Johnstown, PA 15904 USA; Helen Atkinson, The University of Sheffield, Dept. of Eng. Mats., Sheffield, South Yorkshire S1 3JD UK

8:30 AM INVITED PAPER

EFFECT OF SEMI SOLID PROCESSING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ALUMINUM MATRIX COMPOSITES - PART I: *F. Robert Dax*[1]; Tim Freidhoff[1]; Juan J. Valencia[1]; [1]Concurrent Technologies Corporation, 1450 Scalp Ave., Johnstown, PA 15904 USA

Metal matrix composites (MMC) have not been used as extensively as forecast for a variety of reasons. One is the extreme difficulty of machining and grinding MMCs because of the hard second phase. Semi solid metalworking (SSM) offers the potential to form these materials into very complex parts (net shape) while maintaining a homogeneous distribution of the hard second phase within the part. This paper will provide an overview of SSM processing of various aluminum matrix composites, including aluminum A357 + SiC, 6092 + SiC, A356 + SiB6, and in situ Al+25%Si+4%Cu. In addition, the effect of SSM on microstructure and mechanical properties and will also be presented. This work was conducted by the National Center for Excellence in Metalworking Technology operated by Concurrent Technologies Corporation under contract No. N00140-92-C-BC49 to the U.S. Navy as part of the U.S. Navy Manufacturing Technology Program.

8:50 AM INVITED PAPER

EFFECT OF SEMI SOLID PROCESSING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ALUMINUM MATRIX COMPOSITES - PART II: *F. Robert Dax*[1]; Tim Freidhoff[1]; Juan J. Valencia[1]; [1]Concurrent Technologies Corporation, 1450 Scalp Ave., Johnstown, PA 15904 USA

9:10 AM

ADVANCES IN THIXOMOLDING: *D. Matthew Walukas*[1]; Raymond F. Decker[1]; Stephen E. LeBeau[1]; [1]Thixomat, Inc., 717 E. Huron St., Ann Arbor, MI 48104 USA

Thixomolding® is a one step that produces net-shape parts through high speed injection molding of semi-solid thixotropic alloys. A de-

scription of the process will be presented. Microstructural effects and reduced porosity result in improved properties of magnesium alloys. Mechanical properties and microstructures of Thixomolded® AZ91D will be presented. Data on enhanced creep and corrosion properties of Thixomolded® AZ91D will be compared to die AZ91D. Preliminary developments in aluminum Thixomolding® will be covered.

9:30 AM
SOME CRITERIA FOR MATERIAL SELECTION FOR SEMISOLID PROCESSING: *Antonios Zavaliangos*[1]; Evangelos Tzimas[1]; [1]Drexel University, Dept. of Mats. Eng., 32 and Chesnut St., Philadelphia, PA 19104 USA

The important microstructural parameters that determine whether a material is suitable for thixoforming are (i) adequate volume fraction of solid, (ii) equiaxed morphology of the solid grains, and (iii) minimum entrapped liquid. In this paper we introduce a simple criterion for processability in the semisolid range based on the sensitivity of the volume fraction of solid with respect to minor temperature variations. In addition we propose that the processability in the semisolid state can be enhanced using microsegregated material.

9:50 AM BREAK

10:05 AM
EXPERIMENTAL DETERMINATION OF PARAMETERS REQUIRED FOR MODELLING OF SEMI-SOLID PROCESSING: *Helen Valerie Atkinson*[1]; [1]The University of Sheffield, Dept. of Eng. Mats., Sir Robert Hadfield Bldg., Mappin St., Sheffield, South Yorkshire S1 3JD UK

Numerical modelling of the filling of dies in semi-solid processing could help to ensure defect-free products and to avoid costly and time-consuming trial and error with die design and process variables. All numerical models require the input of experimental data on material parameters. These data can be obtained from rheological investigations in rheometers. However, semi-solid processing is generally carried out with fractions of solid above 0.5 and rheometers can only function with fractions of solid below about 0.4. Alternatively, rapid compression experiments can be used with high fractions solid but these have inherent drawbacks. This paper will review the various approaches to obtaining data on material properties as required for numerical modelling.

10:25 AM
EFFECT OF ULTRASONIC TREATMENT ON SUBSEQUENT SEMI-SOLID PROCESSING: *Nico DeSmedt*[1]; B. Bishoff[2]; R. Wagstaff[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]Wagstaff Engineering, N. 3910 Flora Rd., Spokane, WA 99216 USA

Use of ultrasonic treatment can lead to a grain refinement and reduction in secondary dendrite arm spacing (SDAS) in direct chill (DC) aluminum billets. In this paper the effect of a number of parameters including alloy type, grain refiners, billet size and positioning of the ultrasonic transducer on the grain size and SDAS will be discussed. Consideration will also be given to the behavior of ultrasonically treated billets on subsequent semi-solid processing.

10:45 AM
HIGH TEMPERATURE CREEP RESPONSE OF THE AZ91 MAGNESIUM ALLOY PRODUCED BY THIXOFORMING: E. Evangelista[1]; M. Cabibbo[1]; S. Spigarelli[1]; A. Rosen[2]; University of Ancona, [1]INFM/Department of Mechanics, Ancona, Italy 60131; [2]Technion, Dept. of Materials Engineering, Haifa, Israel 32000

The creep response of an AZ91 alloy produced by thixoforming was investigated at 120, 135 and 185°C. The microstructure of the alloy after thixoforming consisted in a distribution of a globules separated by a divorced eutectic composed by small a particles surrounded by b brittle phase. The creep curves exhibited a short primary region, followed by a minimum creep rate range and an extended tertiary stage; the maximum rupture elongation approached 20%. The minimum creep rate dependence on applied stress showed a reduction of the m parameter ($m=\P\log e_m/\P\log s)_{T=cost}$ with stress, suggesting a variation of the creep controlling mechanism. The activation energy for creep ($Q=\P\ln e_m/\P(1/T))_{s=cost.}$ was 111 and 145 kJ/mol at 80 and 100 MPa respectively. Both the minimum creep rate dependence on applied stress and the reduction of activation energy for creep with stress were in close analogy with the creep response of die cast AZ91 having similar microstructure; this result and the presence of a distribution of fine a grains, in turn, support the idea that in the low stress region grain boundary sliding could become rate controlling.

THE MARTIN E. GLICKSMAN SYMPOSIUM ON SOLIDIFICATION AND CRYSTAL GROWTH: Solidification: Modeling

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 Mats. Sci. & Eng., Evanston, IL 60208 USA

Tuesday AM — Room: 11A
March 2, 1999 — Location: Convention Center

Session Chairs: Sam R. Coriell, NIST, Dept. of Metall., Gaithersburg, MD 20899 USA; Ranga Narayanan, University of Florida, Dept. of Chem. Eng., Gainesville, FL USA; Reza Abbaschian, University of Florida, Dept. of Mats. Sci. and Eng., Gainesville, FL 32611 USA

8:30 AM INTRODUCTION

8:45 AM
SEGREGATION SUBSTRUCTURES IN DILUTE Al-Cu ALLOYS DIRECTIONALLY SOLIDIFIED: *O. Fornaro*[1]; H. Palacio[1]; H. Biloni[2]; [1]IFIMAT-CIC-UNICEN, Tandil Argentina; [2]LEMIT-CIC, La Plata Argentina

At low and moderate solidification velocities, the decrease of the parameter GL/VCo (GL: Thermal gradient in the liquid in front of the Solid-Liquid interface, V: S-L interface velocity, Co Alloy composition) controls the evolution of the S-L interface from plane to dendritic. Even the evolution of the instabilities is continuous, with the increase of the constitutional supercooling (CS) the following stages, in f.c.c. alloys, may be defined: (i) planar interface; (ii) nodes or depressions of the S-L interface; (iii) elongated or bidimensional cells; (iv) regular hexagonal cells; (v) distorted or branched cells and (iv) dendritic cells. Extensive research, both experimental and theoretical has been done by several authors on the evolution of the S-L interface, both in dilute alloys and organic transparent alloys, using the linear stability theory where the CS is a particular case. Considering that after the passage of the solidification front, a variation of composition remains on a length scale characteristic of the cellular or dendritic growth, that is called microsegregation, we stress that a careful and suitable metallographic analysis can give good information in 3D symmetry on the mechanisms involved in the evolution of the segregation substructure, where still the transition between cellular and dendritic substructures at low and intermediate velocities are not well understood. Under this frame, unidirectional growth of dilute Al-Cu alloys between 0.2 and 0.5 wt. % Cu, were performed under controlled conditions of the GL and V using a quenching technique of the S-L interface. The metallographic analysis of the segregation patterns behind the frozen interface in longitudinal and transverse sections gives information about the effect of the local solidification conditions, which results in small wall instabilities during the lateral growth of the cells. These instabilities seems to control the formation or disappearance of cellular walls, a mechanism necessary for the evolution of the substructure through the different stages above

mentioned. At the same time a more complete microsegregation map associated with the bulk crystalline growth of the alloys has been obtained.

9:15 AM
EVOLUTION EFFECTS IN DENDRITIC SOLIDIFICATION: *Arnon Chait*[1]; Vladimir Pines[2]; Marianne Zlaatkowski[2]; Matthew Koss[2]; Martin Glicksman[2]; [1]NASA Lewis Research Center, Brook Park Rd., Cleveland, OH 44135 USA; [2]R. P. I., Dept. of Mats. Sci. and Eng., Troy, NY 12180 USA

We examine evolution effects predicted from the recently developed two-parameter scaling theory in dendritic growth (Pines, Chait, Zlatkowski). For sufficiently small levels of supercooling, it was demonstrated that an additional scaling parameter is necessary to completely solve the dendrite selection problem. The new parameter was shown to be related to the initial characteristic nucleus size. We propose a novel microgravity experiment (Transient Dendritic Solidification Experiment) to study dynamic events in dendritic growth. Using rapid pressurization/depressurization we can obtain fast changes in the effective supercooling via the Clapeyron effect on the melting temperature. A hypothetical cycle of increasing/decreasing pressure is examined using a fully numerical nonlinear simulation. We explore the potential role of the initial nucleus size via a direct inspection of hysteresis effects in the dendrite operating state (tip radius and velocity) at the same supercooling level originating from different initial conditions.

9:35 AM INVITED PAPER
FLOW-INDUCED MORPHOLOGIES: *Stephen Davis*[1]; [1]Northwestern University, Evanston, IL 60208 USA

Consider the directional solidification of a dilute binary alloy. The front can be unstable to cellular structures in the absence of melt flow. When the melt undergoes fluid flow, the rejected solute can be redistributed so as to create new morphologies. In the presentation, examples will be discussed of hydrodynamic instabilities interacting with solid-melt interfaces (Benard convection, Taylor-Couette flow), forced flows on propagating interfaces, and some recent results on convective flows.

9:55 AM INVITED PAPER
DENDRITIC GROWTH-DEVIATIONS FORM THE IDEAL: *Robert J. Schaefer*[1]; R. E. Napolitano[1]; [1]NIST, Dept. of Metall., Gaithersburg, MD 20899 USA

Much of our understanding of dendritic growth comes from studies under well-controlled laboratory conditions: in isothermal, supercooled liquids or in constant gradients moving at constant velocity. However, when single crystal superalloy components are produced for use in aircraft engines, the dendrites encounter more complex, transient thermal environments which may lead to defective structures. A predictive model for the development of such defects needs to include several components, including a model describing the thermal field, a model describing the kinetics of growth of the dendrite and its branches, and a model describing how defect formation depends on the local thermal conditions. Here we describe how some of these components can be constructed and put together to estimate the tendency for defect formation. The most difficult part of the model to quantify is the description of the relation between thermal conditions and defect formation.

10:15 AM BREAK

10:35 AM INVITED PAPER
FORMATION OF SUPERSATURATED SEMICONDUCTOR ALLOYS: *J. Narayan*[1]; [1]North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695 USA

By controlling interface velocity during crystallization in liquid-phase, it is possible to far exceed equilibrium solid solubility limits in silicon via solute trapping. There is an interesting parallel in solid-phase crystallization, where by controlling the interface morphology and velocity, it is possible to achieve equally high solubility limits (supersaturation) as much as a factor of 500 or more. The emphasis in this talk will be placed on interface kinetics and instability and their correlation with formation of defects such as twins during crystallization. The impact of these fundamental studies on the formation of defect-free shallow junctions and ohmic contacts are discussed in the context of next-generation semiconductor devices.

11:05 AM
INCREMENTAL CRYSTAL GROWTH IN DROPLET DEPOSITION: *Teiichi Ando*[1]; Charles D. Tuffile[1]; [1]Northeastern University, Dept. of Mech., Industr. and Manufact. Eng., 334 Snell Eng. Center, Boston, MA 02115 USA

Molten uniform droplets of Sn - Pb alloys having the same diameter were generated by the controlled breakup of a laminar jet and deposited onto a temperature- and motion-controlled substrate at different droplet temperatures and liquid fractions. Spray deposits so produced presented different microstructures ranging from fine, equiaxed microstructures, to those of columnar or single crystals. The columnar or single-crystalline growth in spray deposition with uniform-droplets differs from conventional columnar or single-crystalline growth in that the crystals are grown incrementally by the rapid epitaxial solidification of the depositing droplets. The resultant crystals thus present features of a rapid solidification microstructure. Such modes of crystal growth are only possible with fully molten droplets deposited under controlled conditions. Spray and substrate conditions required for the different novel microstructures are discussed.

11:35 AM
DENDRITE MORPHOLOGY DURING DIRECTIONAL SOLIDIFICATION OF BINARY ALLOYS: *Y. Lu*[1]; S. N. Ojha[2]; J. Reye[1]; G. Ding[1]; *S. N. Tewari*[1]; [1]Cleveland State University, Dept. of Chem. Eng., Cleveland, OH 44135 USA; [2]B.H.U.,Dept. of Metall., Varanasi, UP India

Theoretical models of dendritic arrayed growth assume a steady-state dendrite morphology and predict dendrite tip radius, tip composition, tip temperature and primary dendrite spacing as a function of growth parameters (alloy composition, thermal gradient and growth speed) and alloy physical properties. Directional solidification of binary metallic alloys followed by a rapid quench to retain the mushy zone morphology is the only technique which would allow a simultaneous measurement of all these features in order to carry out a meaningful evaluation of these models. Since the metallic alloys are opaque serial sectioning of dendrites in the quenched mushy zone followed by a superimposition of metallographic images is required to visualize the three dimensional dendritic morphology. Results will be presented from our experiments on Pb- 5.8 wt pct Sb alloy. The experiments indicate that the dendrite tips are not axisymmetric, their tip morphology is determined by their immediate neighbors and they do not have a steady-state tip shape, i.e., the tip morphology does not remain constant, it fluctuates within a shape envelope. The cellular arrays show more extensive tip shape fluctuation as compared with the dendrites. It, however, is not certain whether these effects are due to the natural competition among neighboring dendrites or an artifact due to convection in the melt which is ever present during terrestrial directional solidification experiments. This research was supported by NASA- Microgravity Science and Applications Division.

EPD Luncheon
Time: 12:00 Noon
Room: Marina Ballroom
Location: Marriott Hotel

Tutorial Luncheon Lecture: "Dynamic Behavior of Materials"
Time: 12:00 Noon - 1:30 PM
Room: 16A
Location: San Diego Convention Center

EPD Distinguished Lecturer
Time: 1:45 PM
Room: Marina Ballroom
Location: Marriott Hotel

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

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

Tuesday PM Room: 15B
March 2, 1999 Location: Convention Center

Session Chair: J. B. Andrews, University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., Birmingham, AL 35294 USA

2:00 PM

AN EXPERIMENTAL STUDY OF THE DYNAMICS OF A HEATED DOMAIN IN SUPERCRITICAL FLUIDS IN LOW AND EARTH GRAVITY: *V. M. Emelianov*[1]; A. K. Lednev[1]; [1]Russian Academy of Sciences, Institute for Problems in Mechanics, Prospect Vernadskogo 101, Moscow 117526 Russia

The development of a heated domain induced by a point heat source in supercritical CO2 and SF6 is studied in microgravity and Earth's conditions. New results of the processing of data obtained with the Alice-1 instrument aboard MIR station in 1996 as well as the results of experiments obtained with the instrument on the Earth are presented. A comparison of the initial stage of the development of the heated domain is made for microgravity and Earth's conditions. On the Earth the rate of the rise of the head of the heated plum is found to be independent of T-Tc in a wide temperature range above the critical point but the rate essentially depends on the duration of heating. The final stage of the relaxation of the heated domain in microgravity is also studied.

2:15 PM

SOLIDIFICATION PROCESS ON INDIUM ANTIMONIDE ALLOY THROUGH SUPERCOOLED STATE UNDER ELECTROMAGNETIC LEVITATOR AND MICROGRAVITY CONDITION: *Hideki Minagawa*[1]; Masataka Sasamori[1]; Jiro Nagao[1]; [1]Hokkaido National Industrial Research Institute, Mats. Division, Agency of Indust. and Sci. Tech., Ministry of International Trade and Industry, 2-17 Tsukisamu-Higashi, Toyahira-ku, Sapporo 062-8517 Japan

Supercooled state is usually observed under molten material under containerless system. Furthermore this phenomenon is found to be appeared under microgravity condition, when the partial evaporation of the sample is occurred in ultra-high vacuum system. Because the evaporation gas forces the sample to hover, the noncontact conditions seems to be attained. Microgravity experiment has been performed by 1.3 sec drop tower facility in Hokkaido National Industrial Research Institute (HNIRI). The properties of indium antimonide (InSb), such as energy gap, optical absorbance, and Hall coefficient have been investigated. Semiconductive behavior was observed on In Sb alloy with non-stoichiometric composition near stoichiometry. Temperature dependence of carrier density were compared with that of single crystal InSb(111). Furthermore the solidification experiments through supercooling state under electromagnetic levitator system have been performed. The structure of solidification sample by E-M levitator system was polycrystal or dendrite structure, which were dependent on the supercooled degree.

2:30 PM

AN EXPERIMENT METHOD FOR PREPARING MONOTACTIC ALLOYS IN MICROGRAVITY ENVIRONMENT SIMULATED BY ELECTROMAGNETIC FORCE: *Da Daoan*[1]; Jiang Wanshun[1]; Lu Miao[1]; [1]Lanzhou Institute of Physics, P.O. Box 94, Lanzhou, 730000 PR China

In this paper, a new method is advanced, with which binary monotactic alloys have been prepared on the ground, as a result of elimination of Stockes deposition by means of the interaction between electromagnetic force and melted metal. The experimental apparatus has been established for microgravity simulation by electromagnetic force. It consists of an electromagnet, d.c. current, furnace body, temperature controller, and vacuum system. In the paper, a principle figure and main performance parameters are given. By utilizing the test apparatus, Pb-Zn, Al-Pb, Cu-Pb, Al-In, Zn-Bi, Al-Bi, and triple monotactic alloy Pb-Zn-Bi, have been prepared. The configuration analysis and performance test show that the microcrystals of these samples are uniform in distribution. The ground samples are relatively close to the space-prepared monotactic alloy samples in space in crystal dispersion uniformity. The friction wear-resistant properties of some materials are given.

2:45 PM

DESIGN AND PERFORMANCE OF THE MSL LOW GRADIENT FURNACE (LGF) AND SOLIDIFICATION AND QUENCHING FURNACE (SQF): *A. Lundstrom*[1]; P. Behrmann[1]; H. Lenski[2]; E. Barbier[3]; B. Bonduelle[4]; [1]ESA/ESTEC, P.O. Box 299, AG, Noordwijk NL-2200 The Netherlands; [2]DASA-Dornier, Friedrichshafen Germany; [3]SNECMA-SEP, Villaroche France; [4]SOTEREM, Castanet, Tolosan France

The development of the European Materials Science Laboratory for International Space Station includes two ESA furnace inserts: the Low Gradient Furnace (LGF) and the Solidification and Quenching Furnace (SQF). Both of these have for space furnaces novel design features and performance capabilities, which are discussed in the presentation. The LGF is designed for high temperature operation (1600ϒC) with restricted, well-controlled thermal gradients. It is a multi-zone furnace based on a pyrolytic boron-nitride/pyrolytic graphite heater technology with carbon-carbon diffusers. The LGF performance has been tested in a prototype furnace breadboard. A thermal stability of better than + 0.02 K has been achieved, using a sapphire optical fibre thermometer for heater control. The thermal uniformity of the furnace cavity has been characterised, giving a circumferential isothermality of + 0.5 K. Temperature gradients have been established to be controllable in the range 3-50 K/cm. Test results also include information on the accuracy of temperature sensing and component life-time. The main objective of SQF is to achieve operating temperatures of 1800ϒC and gradients of 150K/cm in a space-compatible furnace design. This challenging task is the topic of an on-going technology development. An innovative self-supported heater design based on carbon-carbon composite or pyrolytic graphite heater elements has been established. Design and manufacturing aspects will be presented; a long-term test of this technology is planned for early 1999.

3:00 PM

LATENT HEAT EFFECTS ON THERMOSOLUTAL CONVECTION IN BRIDGMAN SOLIDIFICATION: *P. Haldenwang*[1]; R. Guerin[2]; [1]Universite de Provence, IRPHE/UMR-CNRS 6594/IMt/Lajetee, Technopole de Chateau Gombert 38, Avenue Joliot-Curie, Marseille Cedex 20 F-13451 France; [2]Universite d'Aix-Marseille III, Laboratoire Matop, URA/CNRS 1530, Faculte des Sciences et Techniques de Saint-Jerome, Case 151, Marsceille Cedex 20 F-13451 France

In upward Bridgman solidification of a binary alloys, the vertical temperature profile being known as stabilising, we address the issue concerning the strength of horizontal thermal gradients that result from the latent heat released by the sample and their influence on solutal

convection. We derive a theoretical estimate which indicates that effects due to heat release increases as the fourth power of aspect ratio (i.e. sample radius reduced with the solutal length) and decreases in inverse ratio to Lewis number squared.

3:15 PM BREAK

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A LIQUID PROPELLANT GAUGING IN MICROGRAVITY ENVIRONMENT AND ITS SIMULATIVE TEST EQUIPMENT ON THE GROUND: *Da Daoan*[1]; Zhang Tianping[1]; [1]Lanzhou Institute of Physics, P.O. Box 94, Lanzhou, 730000 PR China

The effect of microgravity condition on a liquid propellant gauging is analyzed, and one method, named pressure simulation by gas injection(PSGI), was selected for the most suitable technique for gauging liquid propellants on satellites. Our theoretical research on PSGI is presented, and a set of simulative test equipment on the ground, which was developed according to PSGI method, is described in details. This system has the function to simulate liquid propellants gauging under the condition that the propulsion system is either in gear or out of gear. The first part of our experiment results obtained by this equipment has shown that it is possible to get an accuracy higher than 1.0% of the total tank volume with PSGI when the propulsion system is in gear.

3:45 PM

DIAGNOSTIC TOOLS FOR FLUID SCIENCE APPLICATIONS IN MICROGRAVITY: *K. Wozniak*[1]; G. Wozniak[2]; J. Siekmann[1]; [1]University Essen, FB 12, Mechanik, Essen D-45117 Germany; [2]TU Bergakademie Freiberg, Institut fur Fluidmechanik und Fluidenergiemaschinen, Lampadiusstr, 2, Freiberg 09596 Germany

When selecting methods suitable for microgravity fluid physics experiments a variety of diagnostic tools must be considered, because a certain method is very often not compatible with specific experiment and carrier constrains. Furthermore, the application of conventional techniques is characterized by a complex set-up, time consuming adjustments and extensive evaluation procedures. Methods for space borne applications should feature minimum payload requirements and a simple experimental procedure in view of limited operating and crew time. We describe three optical methods in some detail. A digital holographic interferometer, a differential interferometer with Wollaston-prism and the liquid crystal tracer technique exhibiting the following advantages: The experimental set-up has a rather compact design; Extensive alignment adjustment is not required; The sensitivity can be varied in a wide range; Even higher accelerations do not influence the adjustment.

4:00 PM

DOUBLE-LAYERED LIQUID MASS UNDER MICROGRAVITY: Itaru Jimbo[1]; *Masato Takahashi*[1]; [1]Tokay University, Dept. Metall. Eng., 1117 Kita-Kaname, Hiratsuka, Kanagawa 259-1292 Japan

The study of "Double-layered Liquid Mass" under microgravity is undertaken in Tokai University, Japan. The DLM, in which the first spherical liquid mass is covered with the second liquid layer or shell, may be one of the promising material refining and processing procedures, where the reaction occurs at all over the interface between the two liquid phases. This can be well applied in the container-free processes under microgravity. As a preliminary experiment, the DLM consisting of silicone oil and water was successfully produced in a plateau tank facility and the stability of the DLM in this system was carefully investigated. The fundamental factors to control the procedure will be discussed with the calculation result of the total interfacial energy for the separated liquid mass and the DLM. The effect of the application of super sonic wave on the separation of the two liquid phases will also be discussed.

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SOLIDIFICATION OF Al ALLOYS IN MICRO-GRAVITY: *Johan Dahlstrom*[1]; *Hasse Fredriksson*[1]; [1]Royal Institute of Technology, Dept. of Mats. Processing, Dr. Kristinasv. 6, Stockholm S-100 44 Sweden

Solidification of pure Al, Pb and Sn and alloys of Al-Cu, Al-Sn, Pb-Sn has been done in the same experimental set-up in micro-gravity and on earth. The micro-gravity experiments were done on two parabolic flight campaigns. The cooling rates were from 10 C/s to 150 C/s and the size of each sample was around 10 mm3. For Al 2% Cu it was found that the microstructure was finer in the samples solidified on earth. It was also found for Al-alloys that the latent heat was smaller and that the melting point was lower in earth based compared to micro-gravity experiment at similar cooling rates. This was explained by the creation of more excess vacancies during solidification on earth compared to solidification in micro-gravity. The number of vacancies and the phase diagram was calculated for excess vacancies.

4:30 PM

GROWTH OF II-VI SOLID SOLUTIONS IN THE PRESENCE OF A ROTATING MAGNETIC FIELD: *D. C. Gillies*[1]; S. Motakef[2]; M. Dudley[3]; R. Matyi[4]; H. Volz[4]; [1]NASA/Marshall Space Flight Center, Huntsville, AL 35812; [2]Cape Simulations Inc., Newton, MA 02458; [3]State University of New York at Stony Brook, Stony Brook, NY 11794; [4]University of Wisconsin-Madison, Madison, WI 53706

The application of a rotating magnetic field (RMF) in the frequency range 60-400 Hz and field strength of 2-8 mT to crystal growth has received increasing attention in recent years. To take full advantage of the control of fluid flow by the forces applied by the field, the liquid column must be electrically conducting. Also, the application of RMF to the directional solidification of a column of liquid can result in complete mixing in the resultant solid. Thus, the technique of RMF is suited to solvent zones and float zones where the composition of the liquid is more readily controlled. In the work we report on, numerical modeling has been applied to II-VI systems, particularly tellurium based traveling heater techniques (THM). Results for a spectrum of field strengths and acceleration levels will be presented. These show clearly the effects of competing buoyancy forces and electromagnetic stirring. Crystals of cadmium zinc telluride and mercury cadmium telluride have been grown terrestrially from a tellurium solvent zone. The effects of the RMF during these experiments will be demonstrated with micrographs showing etch pits, white beam x-ray synchrotron topographs and triple axis x-ray diffraction.

4:45 PM

SURFACE TENSION AND VISCOSITY MEASUREMENTS IN MICROGRAVITY: SOME RESULTS AND FLUID FLOW OBSERVATIONS DURING MSL-1: R.W. Hyers[1]; *G. Trapaga*[2]; and M.C. Flemings[2]; [1]NASA-MFSC, Mail Code EC76, Huntsville, AL 35812; [2]Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Ave. Rm. 8-101, Cambridge, MA 02139

The viscosity of a liquid metal was successfully measured for the first time by a containerless method, the oscillating drop technique. This method also provides a means to obtain a precise, non-contact measurement of the surface tension of the droplet. This technique involves exciting the surface of the molten sample and then measuring the resulting oscillations; the natural frequency of the oscillating sample is determined by its surface tension, and the damping of the oscillations by the viscosity. These measurements were performed in TEMPUS, a microgravity electromagnetic levitator (EML), on the Space Shuttle as a part of the First Microgravity Science Laboratory (MSL-1), which flew in April and July 1997 (STS-83 and STS-94). Some results of the surface tension and viscosity measurements are presented for Pd82Si18. Some observations of the fluid dynamic characteristics (dominant flow patterns, turbulent transition, cavitation, etc) of levitated droplets are presented and discussed together with magnetohydrodynamic calculations, which were performed to justify these findings.

ALUMINA AND BAUXITE: Alumina Process Developments

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

Tuesday PM Room: 6E
March 2, 1999 Location: Convention Center

Session Chair: Travis J. Galloway, Reynolds Metals, Global Bauxite/ Alumina Business Unit, Richmond, VA USA

2:00 PM

NEW WATER-CONTINUOUS RED MUD FLOCCULANTS FOR THE BAYER PROCESS: *Everett Charles Phillips*[1]; [1]Nalco Chemical Company, Alumina Process Chemicals Group, Mining and Mineral Processing Division, One Nalco Center, Naperville, IL 60563-1198 USA

A new family of water-based red mud flocculants has been developed for use in the Bayer Process. There are several key advantages of this Water-Continuous Flocculant technology compared to dry and oil-based latex red mud flocculants. These include improvements in liquor clarification performance and advantages related to the form of these products. This paper presents the developments to date, including results from plant evaluations, demonstrating the utility of this new technology for clarifying Bayer Process red mud slurries.

2:30 PM

BAYER PROCESS ZINC REMOVAL RESEARCH: Gan Guoyao[1]; *Wang Longzhang*[1]; Yang Shijie[1]; [1]Pingguo Alumina Plant, Pingguo, Guangxi 531400 China

Pingguo bauxite contains a high content of zinc. Zinc removal technology for the Pingguo plant was researched. This paper presents the results of this study. The effect of removing zinc is not good when adding as many moles of sodium sulfide as zinc. When an excess of sodium sulfide is added, the content of zinc in the pregnant liquor decreased greatly but the silica and iron increased significantly. Increased retention time and air agitation improved the process and most of the zinc was removed and settled into the red mud. The paper describes the development of this powerful process.

3:00 PM

THE CHEMISTRY OF SOLID-LIQUID CALCINATION(SLC): *Helene Boily*[1]; Flor Campa[2]; Kirsten Theisen[3]; [1]Alcan International, Ltd., P.O. Box 1250, 1055 Mellon Blvd., Jonquiere, Quebec G7S4K8 Canada; [2]ALCOAO Inespal, Apartado 71, San Ciprian, Lugo 27890 Spain; [3]F. L. Smidth & Company, Vigersley Alle 77, Valby DK2500 Denmark

Commissioning of the 120 T/D prototype SLC unit began in late summer 1998 at San Ciprian, with a feed slurry mixture containing salt cake (crystallized sodium salts from the Bayer process), bauxite slurry and concentrated spent liquor. The parameters used to control the process will be reviewed, together with the chemical and physical properties of the feedstock, the intermediate phases and end products of the process.

3:30 PM BREAK

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ALUMINA PRODUCTION FROM DIASPORIC BAUXITES: *Eric Lavalou*[1]; Bernard Bosca[2]; Odysseas Keramidas[2]; [1]Aluminium Pechiney, BP42, 13541 Gardanne, Cedex France; [2]Aluminium de Greece, Paralia Distomon, 32003, Saint Nicolas, Beotie Greece

Diasporic minerals represent only a small part of the world deposits in metallurgy grade bauxites. Nevertheless, these materials are strategic for global commercial exchanges of some countries, like Greece or People's Republic of China for example. Following an overview on geology, main locations, and characteristics of diasporic bauxites, will be discussed: chemical and technological problems during their use in Bayer process; process and technology efficiency in some industrial realizations; and current developments, and optimization prospects to break through present yield limitations that is 97% (digestion) and 90 gpl (precipitation).

4:30 PM

SETTLER THROUGHPUT INCREASE AT ALUMINIUM DE GRECE REFINERY: *X. Perrier*[1]; B. Benoit[2]; P. Dountsis[1]; [1]Aluminium Pechiney, Direction de la Technologie Alumine, BP 54, Gardanne 13541 France; [2]Aluminium Pechiney, Direction de la Rocherche et du Development, BP 54, 13541 Gardanne, Cedex France

Aluminium de Grece has five primary mud settlers, four flat bottomed and one conical bottomed. These settlers have been operated on starch based flocculants since plant start up. The maximum overflow rate has been approximately 0.6m^3/h.m^2 corresponding to 50 kg/h.m^2 mud throughput. In order to get additional washing stages and to reduce mud residence time, several synthetic flocculant strategies were investigated. The target overflow rate was 1.2 m^3/h.m^3. During industrial tests, the target was exceeded with hydroxamate both with and without starch. With polyacrylamide and starch it was difficult to simultaneously control clarity and rake torque. Hydroxamate dosage was progressively optimized and presently operation with only two settlers on line is considered.

5:00 PM

ALUMINA TRIHYDRATE PRECIPITATION PARTICLE SIZE CONTROL: *Bret Garner*[1]; Arnaud Soirat[1]; Benoit Cristol[2]; [1]Queensland Alumina, Ltd., Parsons Point, Gladstone, Queensland 4680 Australia; [2]Alumina Research & Development Centre, Aluminium Pechiney, Gardanne Cedex, France

By employing a reliable and comprehensive set of sensors and appropriate actuators according to Pechiney Particle Size Distribution Control strategy, a new methodology has been implemented to stabilize and optimise particle size distribution within Queensland Alumina's precipitation circuit. The new methodology, based on quantitative control of fines through adjustment of agglomeration set points, allows elimination of the detrimental consequences of sub-optimal seed size control, such as: reduced circuit seed surface area leading to reduced yield; increased probability of nucleation explosions; reduced pregnant liquor flows and/or seed charge due to unsustainable fines inventory build up, and swings in alumina product quality. This paper examines the impact of this sub-optimal seed size control on circuit performance and benefits derived from the improved control to date.

5:30 PM INVITED PAPER

IMPROVEMENT OF FLOWSHEET OF ALUMINA PRODUCTION IN SODA-LIME SINTERING PROCESS: *Shang-guan Zheng*[1]; Zhong-yu Yang[1]; Shen-xing Hu[2]; An Luo[2]; [1]Central South University of Technology, Dept. of Metall., Changsha, Hunan Province 410083 PR China; [2]Shandong Aluminum Corporation, Corporation Office, Zibo, Shandong Province 255052 PR China

An improved technology in the present work is proposed that pregnant liquor from sinter leaching in soda-lime sintering process is subjected to carbonization and thus obtained raw Al(OH)3 (high silica content) is processed with very simple Bayer process instead of multi-desilication process. With the new technology, sandy alumina with high quality could be produced in sintering process; the amount of sinter could be decreased by about 0.5 t/t-Al2O3; process control is easier than before; the consumption of CO2 gas for carbonization is only increased by 3.65%; the increased operation cost of inserted simple Bayer process could be completely compensated by the cost of the oringinal desilication operation which has been cancelled in the new technlogy. The production cost with the improved technology could be decreased by 82.87 yuan/t-Al2O3 and the output of alumina would be increased by 75 000 tons/year. Keywords: high silica bauxite, soda-lime sintering process, sinter leaching, purification of raw Al(OH)3

ALUMINUM REDUCTION TECHNOLOGY: Control of Reduction Lines

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, Ltd., Brisbane, Queensland 4001 Australia

Tuesday PM Room: 6F
March 2, 1999 Location: Convention Center

Session Chair: Pierre Homsi, Aluminium Pechiney, Laboratoire de Recherches des Fabrications, St. Jean De Maurienne Cedex BP114 733 France

2:00 PM
REDUCTION CELL AUTOMATION: IMPROVEMENTS OF THE ALUMINIUM SMELTER IN POLAND: E. Bugzel[2]; J. Galek[3]; A. Jozwiak; R. T. Mourão[1]; *C. M. Ritter*[1]; [1]ATAN Automation Systems, Aluminium Division, Rua Pernambuco 353, sl. 811, Funcionários, Belo Horizonte, Minas Gerais 30130-150 Brazil; [2]Huta Aluminium KONIN S A, Development and Modernization of Electrolysis Dept., ul. Hutnicza 1, Konin 62510 Poland; [3]Huta Aluminium KONIN S A, Production Dept., ul. Hutnicza 1, Konin 62510 Poland; Huta Aluminium KONIN S A, Power and Mechanical Dept., ul. Hutnicza 1, Konin 62510 Poland

This paper describes the performance and operational improvements achieved by Huta Aluminium Konin after the adoption of a new automation system and operational strategies. An historical overview of the previous control system is presented. Next, the features and the topology of the new automation system are described. The results obtained due to the new configuration and the operational interaction with the automation system are then presented. Concluding the paper, the immediate benefits and the medium and long term advantages related to these changes are discussed.

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ELIMINATING THE ANODE EFFECTS: *R. G. Haverkamp*[1]; [1]Massey University, Institute of Technology and Eng., Private Bag 11222, Palmerston North New Zealand

The anode effect in the production of aluminium by the Halt-Heroult process is a well known but poorly understood effect. In this paper the literature on the anode effect is reviewed and parallels drawn with electrode phenomena in other processes. In industrial cells the anode effect is tolerated as a check on alumina levels to reduce the possibility of sludging however it has the negative consequences of increased CF_4 emissions, cell overheating, reduced production and increased energy consumption. It is proposed that it is desirable to eliminate anode effects completely, with a possible accompanying decrease in sludging, by more careful control of dissolved alumina levels and bath temperature.

3:05 PM
POINT FEEDING FOR CONSTANT ALUMINA CONCENTRATION IN SÖDERBERG CELLS: *R. T. Mourão*[1]; a. fERNANDES[1]; E. Castro[2]; [1]ATAN Automation Systems, USC, R. Pernambuco, 353, Funcionários, Belo Horizonte, Minas Gerais 30130-150 Brazil; [2]CBA, R. Moraes 347, Aluminio, Brasil, 18125-000

This paper presents an algorithm for alumina point feeding in Söderberg cells at CBA, Companhia Brasileira de Alumínio. The developed algorithm is a variation of the "demand feeding" algorithm, where the pot is fed according to its demand of alumina. The main objective of the algorithm developed is to keep alumina concentration in the bath as constant as possible. The revised operational procedures and the improvements in the pot line overall efficiency due to the point feeding are presented and discussed.

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IMPROVED CONCEPT OF ALUMINA-FEEDING STRATEGY: *K. Hofenbitzer*[1]; [1]VAW Aluminium-Technologie GmbH, Georg-von-Boeselager-Str.25, Bonn 53117 Germany

The state-of the art Point-Feeding System recently developed by VAW Aluminium-Technologie GmbH (VAW-ATG) has already been successfully installed in all potlines at VAW's Rheinwerk Smelter. The latest Alumina-Feeding technology gives some telling advantages: compressed air consumption is significantly reduced; immersion of the breaker in the molten cryolite is reduced to an absolute minimum; possible irregularities will be indicated by alarms. The point feeders are continuously controlled via ELAS, the pot-controller. ELAS, the basic element of the Alumina-Feeding technology enables all personnel involved in aluminium reduction to control the whole modus operandi not only of crust breaking and feeding components. An up-to-date graphical user interface in conjunction with the accompanying ELAS software package allows the operator to communicate easily with his potline form anywhere in the world.

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AUTOMATIC DETERMINATION OF METAL HEIGHT IN ELECTROLYSIS CELLS: *G. A. Guðmundsson*[1]; [1]Icelandic Aluminium Company, Ltd., Eloctrolysis, Straumsvík, P.O. Box 244, Hafnarfjörður IS-222 Iceland

A reliable measurement of metal height to decide tapping amount of pots has been badly needed in the aluminium industry. In this paper a method for automatic determination of metal height is described. The method is based on calculation from insertion height of anodes and the height of the anode beam from a beam position transducer. The determination can be made more accurate by correcting for deviation from various setpoints according to known correlations. Such corrections are pot voltage, bath temperature and bath composition. At the Icelandic Aluminium Co., Ltd. this method has been used since spring 1997. The method has stabilised tapping amount, fluoride concentration and temperature. It was implemented with the existing process control system, which is based on Pot Control Units from ALESA Alusuisse Engineering, Ltd. No extra transducers had to be installed. Bath height and metal measurement is also done manually as the bath height is still needed. The tapping amount is decided by the automatic determination of the metal height but is occasionally adjusted according to the manual measurement.

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THE PECHINEY SEMI-CONTINUOUS & AUTOMATIC MEASUREMENT DEVICE (CMD), A NEW TOOL FOR AUTOMATIC MEASUREMENTS: *O. P. Bonnardel*[1]; P. Homsi[1]; [1]Aluminium Pechiney, LRF, B.P.114, 73303 Saint-Jean-De-Maurienne Cedex, Savoie France

For many years, Aluminium Pechiney has been developing advanced software tools in pot process control, which has become one of the cornerstones of Pechiney's technology. The effectiveness of this control depends on the quality of the measurements. Except for pot voltage and potline amperage, measurements are taken manually, which means high operating costs, low frequency and lack of accuracy. The purpose of this paper is to present the new Pechiney patented tool for taking automatic measurements of bath temperature and bath level. First, the inherent advantages of automatic measurements over manual measurements are summarized. We then describe the semi-Continuous & automatic Measurement Device (CMD) and its integration in the process control chain, i.e. the pot micro and the potline supervisory systems. Finally, we present quantified improvements in operating values and the overall profitability of the device.

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REAL TIME SIMULATOR TOOL FOR TRAINING AND DEVELOPMENT IN REDUCTION CELLS: *A. Meghlaoui*[1]; Y. A. Mohamed[1]; B. Jolly[1]; [1]Dubai Aluminium Company, Ltd., (DUBAL), Technology Development, P.O. Box 3627, Dubai United Arab Emirates

A computer simulator of an electrolytic cell has been constructed at Dubal. The mathematical model, already adapted using plant data, is controlled by a pot control unit (PCU). An adequate software-hardware

environment has been developed to receive the line current signal from the plant data network and to establish a reliable data exchange between the PCU and the cell simulator. This real time simulator serves as a tool for training, development of pot control strategies and testing the functioning of the whole system. In this paper several features related to the connection of the PCU to the "soft cell" and running real time simulation are presented and discussed in details to help in overcoming difficulties encountered in this integration process.

ANALYTICAL TECHNOLOGY IN THE MINERAL INDUSTRIES: Analytical Methods for Acid Rock Drainage Prediction

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 Metallurgy 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

Tuesday PM Room: 7A
March 2, 1999 Location: Convention Center

Session Chairs: Rebecca A. Miller, Minekeepers, Phoenix, AZ 85014 USA; C. Mark Wallis, Hydrometrics, Tucson, AZ 85741

2:30 PM INVITED PAPER

MODELING REACTIVE SOLUTE TRANSPORT FROM HISTORIC WASTE ROCK TO RECEIVING GROUNDWATER IN THE ROBINSON DISTRICT, ELY, NEVADA.: *Andy Davis*[1]; G. Fennemore[1]; J. Anderson[1]; S. Okeson[1]; C. Byrns[2]; [1]Geomega, Geochemistry, 2995 Baseline Rd., Suite 202, Boulder, CO 89319 USA; [2]BHP Robinson, Ely, NV 89319 USA

Groundwater flow and solute transport modeling was conducted to evaluate the environmental impacts of historic waste rock dumps and related seeps in the Robinson Mining District, Ely, Nevada. The objectives of the investigation were to: 1) understand the incremental impact of historic mining activities on groundwater; 2) identify which waste rock dumps, if any, contribute to local groundwater chemistry; 3) determine if seeps and springs affect groundwater at the local or regional scale; and 4) assess the spatial extent of mine-affected groundwater. The rate of pyrite oxidation in waste rock dumps was determined using the WROC-2D code, in conjunction with geochemical characterization data collected from waste rock dumps across the site. Waste rock dump oxidation was predicted using data from the waste rock characterization calibrated to measured oxygen and temperature profiles in the dumps. Then 4 well-benchmarked codes were coupled to determine flux from the waste rock dumps and seeps. HYDRUS_2D (unsaturated zone flow and transport), was coupled to MODFLOW (saturated zone flow), MT3D96 (saturated zone solute transport), and PHREEQC (equilibrium reaction chemistry through the vadose and saturated zones). The 60-year simulations demonstrated that sulfate, the most mobile constituent, remains close to local sources within the Robinson Mining District, including all historical source area locations where groundwater impacts are predicted by releases at the surface. The complex structural geology and metamorphism in the Robinson District and the depths to the regional aquifer have resulted in no impacts from historic mine operations to the deep carbonate aquifers within the Robinson Mining District. Laterally, groundwater flow from the mine area into the adjoining hydrogeologic provinces is recalcitrant due to numerous hydraulically impeding faults and low conductivity rocks. Solute groundwater transport simulations demonstrated that even sparingly reactive solutes, such as sulfate, have not migrated more than 1600 feet laterally from the potential historic source areas.

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ROLE OF SILICATES IN THE COMPOSITIONAL EVOLUTION AND NEUTRALIZATION OF Fe- AND Mg-SULPHATE TYPE WATERS IN WAITE-AMULET TAILINGS, CANADA: *John L. Jambor*[1]; H. Wayne Nesbitt[2]; [1]Leslie Investments, Ltd., 316 Rosehill Wynd, Tswwassen, British Columbia V4M 3L9 Canada; [2]University of Western Ontario, Dept. of Earth Sciences, London, Ontario N6A 5B7 Canada

Oxidative dissolution of pyrrhotite is largely responsible for the sulphate-rich waters of the vadose in Waite-Amulet tailings. Although the shallowest of the sulphate-rich waters are Fe-SO4 type, they evolve to Mg-SO4 type, near-neutral waters within 1 to 2 metres of the tailings surface. The mafic silicate minerals are the only Mg-bearing phases present in sufficient quantity to account for the abundance of Mg in these solutions and to neutralize the amount of acid produced. A chemical weathering methodology demonstrates that the Mg-silicates exert major influence on the evolution of these waters. Relative rates of reaction of primary minerals (quartz, feldspars, micas, pyroxenes and amphiboles) can be deduced by comparison of modal abundances in the vadose and phreatic zones of the tailings. The relative rates of sulfides, phyllosilicates, pyroboles (pyroxenes and amphiboles), feldspars and quartz weathering in the vadose zone are 10:4:2:1:1. The evolution of pore waters from Ca-Na-HCO3 type (emanating from the crushed limestone cover), to saline Fe-SO4 type and finally to Mg-SO4 type waters in the vadose zone can be summarized in three reactions. Two represent pyrrhotite and pyrite oxidative dissolution, with the former being far more important to the evolution of these waters than the latter. The major neutralizing reaction involves reaction of Mg-bearing phyllosilicates and pyroboles with the acidic waters of the tailings. Their high reactivity suggests mafic minerals have significant intermediate and long-term neutralizing capacity. Their effects should be considered when evaluating the long-term costs of mine waste treatment.

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THE EFFECT OF ARID CONDITIONS ON PYRITE OXIDATION KINETICS: *Andy Davis*[1]; C. Neller[1]; G. Fennemore[1]; [1]Geomega, Geochemistry, 2995 Baseline Rd., Suite 202, Boulder, CO 89319 USA

The primary factor influencing wall rock leachate chemistry is the propensity for sulfide minerals in the rock to oxidize and release solutes when leached. Typically, pit lake prognostications have relied upon humidity cell tests to derive possible wall rock leachate chemistry. However, these tests unrealistically simulate conditions in Nevada because moist air is employed in the oxidation step for 50% of the time, and in practice the rock material rarely dries out completely during the drying cycle. Further, the humidity cells only incorporate the <2mm fraction, while in reality there is a large range of particle sizes in the ultimate pit surface wall rock. Consequently, a more representative method was sought to determine wall rock leachate chemistry. 47 humidity cells were run on material with a wide range of NCVs (e.g., -7 to +16 for the field oxidation experiment, and -9 to +25 for the humidity cell test). Additionally, samples were crushed and sieved to 2-4 mm, 4-16 mm, and 16-64 mm to assess particle size effects. The 16 bucket leachate tests used 800 g of each of 15 rock samples collected from the pit wall that were splits of those rocks used in the humidity cell tests. Another bucket, which did not contain any rock sample, was set up as a control to collect site precipitation. Runoff from the bucket tests was collected six times following precipitation events and the volume of runoff following each event was measured to allow normalization of the field and laboratory data sets. The results demonstrate that the field oxidation tests generated lower solute concentrations than those in the analogous humidity cell tests, with the most noticeable differences observed in the acid-generating rocks where the humidity cells overestimated field leachability by up to an order of magnitude, while increasing particle size resulted in reduced leachate solute concentrations in both the field and humidity cell tests.

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4:05 PM INVITED PAPER

MANAGEMENT OF POTENTIALLY ACID FORMING OVERBURDEN AT KPC COAL MINE, INDONESIA: *Warwick Stewart*[1]; Dan Michaelsen[1]; [1]PT Kaltim Prima Coal, Mine Office, Sangatta, East Kalimantan Indonesia

PT Kaltim Prima Coal (KPC) is a large-scale truck and shovel coal mining operation situated in the tropical equatorial region of East Kalimantan, Indonesia. The operation produces 15 million tonnes of coal and over 120 million bank cubic meters of overburden annually. Approximately 35 percent of the overburden is classified as potentially acid forming and therefore there is a potential to produce large volumes of acid drainage and large areas of acid spoil. Management of acid rock drainage (ARD) is required for both overburden dumps currently under construction, and for those constructed prior to ARD being identified as an issue. A modified field net acid generation (NAG) test procedure has been developed at KPC for identification and segregation of overburden geochemical types. The development and field application of the NAG procedure is presented in a companion paper. KPC aims to prevent acid production in dumps by covering potentially acid forming spoils with materials identified as non-acid forming. The covers, built to engineering standards, minimise the flux of oxygen and water to pyritic spoils, thereby controlling the oxidation reaction to meet a design target acid sulphate generation rate of less than 10 tonnes SO4/ha/yr. Cover designs and cover material vary according to stability, materials availability, equipment availability and cost effectiveness. This paper discusses the ARD management strategies and operational procedures adopted by KPC to effectively manage potentially acid forming overburden.

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IDENTIFYING POTENTIALLY ACID FORMING OVERBURDEN TYPES USING A FIELD NAG TEST PROCEDURE AT KPC COAL MINE, INDONESIA: *Clayton L. Rumble*[1]; Stuart D. Miller[1]; [1]Environmental Geochemistry International Pty, Ltd., 81A College St., Balmain, NSW 2041 Australia

A field net acid generation (NAG) test procedure has been utilised onsite to identify, classify and map potentially acid forming overburden at the KPC (PT Kaltim Prima Coal) coal mining operation situated in the tropical equatorial region of East Kalimantan, Indonesia. The field procedure involves routine use of a calibrated, site specific NAG test (rapid oxidation test using hydrogen peroxide) which has a turn-around time of less than 24 hours and requires minimal sample preparation. It is used on geological drill-hole samples, for forward planning and overburden management; blast-hole samples, for day-to-day planning and overburden scheduling; and final dump surface samples, for verification testing. This operational monitoring program has been in use at KPC for the past four years. Calibration and implementation of the field procedure has involved validating the NAG test using conventional acid-base accounting techniques, producing an overburden geochemical classification scheme, optimising laboratory and sample preparation procedures, and ongoing quality management. This paper presents background theory and practical application in a case study format.

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USE OF THE NET ACID GENERATION PH TEST FOR ASSESSING RISK OF ACID GENERATION: *William M. Schafer*[1]; Ed Spotts[1]; Freddy Guard[1]; Michael Brewer[1]; [1]Schafer & Associates, P.O. Box 6186, Bozeman, MO 597771-6186 USA

The net acid generation (NAG) pH analysis is a method that allows rapid assessment of the acid generation risk of rock samples. This method is useful because it is simple, effective, rapid, and can be conducted at mine assay labs. The NAG pH test is particularly effective for operational testing programs used to classify, selectively handle, and route potentially acid generating waste rock. The NAG pH procedure is based on a 24-hour oxidation of a pulverized rock sample with hydrogen peroxide and subsequent measurement of the sample's pH. The 24-hour pH measurement is called the sample's "NAG pH" value. The 24-hour duration provides time for sulfuric acid to be produced through oxidation of pyrite contained in the sample, followed by dissolution and neutralization of the acid by carbonates and other rapidly-reacting neutralizing minerals present in the sample. If the NAG pH is below a critical value, determined empirically, then the sample has the potential to generate acid in the field. If the NAG pH is above this value, then it is considered a non-acid generator. The critical NAG pH value is typically within the range of 3 to 4.5. However, the exact relationship between NAG pH and potential acid generation should be determined individually for each rock type at each mine site, based on comparison with results of acid base accounting, humidity cell tests, and mineralogical analyses for each rock type. Case studies of the calibration and use of the NAG test for predicting ARD risk are described in this report. NAG pH compares favorably with results of 20-week humidity cell tests. In addition, it has been used successfully for mines with high sulfide-high carbonate mineralogy, and for low sulfide-low carbonate systems.

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ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY (EIS) TECHNIQUE FOR THE PREDICTION OF ACID MINE DRAINAGE POTENTIAL OF WASTE ROCKS: *Manoranjan Misra*[1]; *X. Su*[1]; Indira Chatterjee[3]; [1]University of Nevada, Reno, Dept. of Chem. & Metall. Eng., MS-170, Reno, NV 89557 USA; [3]University of Nevada, Reno, Dept. of Elect. Eng., MS-260, Reno, Nevada 89557 USA

The conventional methods to assess AMD potential of the waste rocks and tailings are based on wet chemistry and leachability tests. Recently, Electrochemical Impedance Spectroscopy (EIS) has been used to predict the AMD potential of the waste rocks and sulfide tailings. The EIS method is non-evasive and rapid. This method can be used for different types of reactive tailings and rocks.

AUTOMOTIVE ALLOYS III: Session IV — Applications

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

Tuesday PM Room: 3
March 2, 1999 Location: Convention Center

Session Chairs: Subodh K Das, ARCO Aluminum, Inc., Louisville, KY 40232 USA; Andrew M. Sherman, Ford Motor Company, Ford Research Laboratory, Dearborn, MI 48121-2053 USA

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DEVELOPMENT AND APPLICATION OF A MODIFIED 319 SEMI-SOLID ALUMINUM ALLOYS TO REPLACE CAST IRON AUTOMOTIVE PARTS: *S. C. Bergsma*[1]; [1]Northwest Aluminum Company, 3313 West Second St., The Dalles, OR 97058 USA

A major manufacturer of automotive seat belt components was investigating various aluminum alloys to replace a critical cast iron seat belt part. Several cast aluminum alloy parts were tested and failed but the part fabricated from 6061.T6 was approved. However, the cost of forging and machining a 6061 part was deemed prohibitive. Their first semi-solid test parts were formed from alloy 357 but a few of these parts failed. Subsequently, a modified 319 alloy (designated DF-53) was tested and approved and has been in production for over a year. The ingot used to form these parts relies on Semi Solid Thermal Transformation (SSTT) during induction heating to obtain a spherical structure suitable for semi-solid forming. Finished T6 parts from DF-53 have properties approaching 6061.T6. This report covers the ingot technology and the DOE to determine the best T5 and T6 practices for DF-53.

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MECHANISMS OF SOLDERING IN HIGH PRESSURE DIE CASTING OF ALUMINIUM ALLOYS: *Zhan Wen Chen*[1]; Mahnaz Z. Jahedi[1]; [1]CSIRO, Manuf. Sci. and Tech., Corner Raglan and Albert Sts., Preston, Victoria 3072 Australia

High pressure die casting (HPDC) is a widely used manufacturing process for mass production of near net shape automotive components of aluminium alloys. Soldering is an important die failure mode in HPDC

but the metallurgy processes of soldering are not clear. In this study, HPDC soldering experiments have been conducted using core pins as soldering targets. Due to heat loss in the process, the soldering reactions in the die cavity occurred at temperatures equal or below the liquidus temperature of the cast alloy. Local solidification time was found to be the main determining factor for the severity of soldering. Hence, in thicker sections of the casting, the pins came in contact with the solidifying alloy longer during a casting cycle than thinner sections and therefore required less cycles to solder. Soldering started with the formation of localized intermetallic particles accompanied by the build-up of solidified cast alloy. The soldered layer developed through the growth of these intermetallics via a solid state reaction together with coarsening of the phases in the cast alloy layer adjacent to the soldering intermetallics. The fracture path separating the casting and the soldered pins during casting ejection was usually in the cast alloy rather than inside the intermetallic layers. This may be related to the lower mechanical properties of the cast alloy adjacent to the intermetallic layers.

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FRICTION STIR WELDING MAGNESIUM ALLOYS: *G. Kohn*[1]; S. Antonsson[2]; A. Munitz[3]; [1]Rotem Industries, Ltd., Beer-Sheva Isreal; [2]ESAB AB, Ltd., Lexa Sweden; [3]NRCN, Beer-Sheva Israel

In the frame-work of the Israeli Consortium for the Development of Magnesium Technologies, a large variety of joining methods are being studied and their suitability for magnesium alloys is examined. Among others, a relatively new method called Friction Stir Welding (FSW) was studied. The integrity and properties of the FSW specimens was investigated using conventional destructive and non-destructive testing methods. It was found that Mg type AZ91D cast alloy can be welded using the FSW method with negligible amount of defects. A complex microstructure of the weld area was observed which was comprised of three different type of zones: I. Molten and resolidifed areas with a central heavily stirred zone and a thin molten layer at the top of the welded plates; II. Areas exhibiting evidence of severe plastic flow, that include also partially melted and recrystallized zones; III. Heat affected zones. The severe plastic deformation and rapid solidification involved in FSW resulted in fine microstructure welds with enhanced mechanical properties compared to those of the parent material.

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ROLL FORMING OF AXISYMETRIC COMPONENTS OF ALUMINUM ALLOYS II: APPLICATION TO 7000 SERIES AND CAST ALUMINUM ALLOYS: *C. K. Syn*[1]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

A unique roll forming technology that permits complex axisymmetric components, such as automotive wheels and turbine disks, to be formed in a single forming operation, has been developed by two Russian institutes, the Russian Federal Nuclear Center, Institute of Technical Physics and the Institute for Metals Superplasticity Problems. The roll-forming process offers opportunities to manufacture high strength components in continuous, economical operations. In addition, the process eliminates the need to manufacture the matching die sets that are required in conventional forging operations. This process was used to fabricate automobile wheels from a Russian AVT alloy, a 6010 aluminum alloy equivalent, as reported in the Symposium on Automotive Alloys II. In the present report, application of the process to high-strength 7000 series aluminum and cast aluminum alloys will be described. The process included steps of isothermal compression of the initial blanks, isothermal forging of the blanks into preforms, and final isothermal roll forming of preforms into wheel shapes. The microstructure and mechanical properties were evaluated for the finished wheels, preforms, and initial blanks by metallography and tensile testing at elevated temperatures.

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TENSILE AND FATIGUE PROPERTIES OF A356 AND A357 SQUEEZE AND SEMI-SOLID CASTINGS: Gautham Ramachandran[1]; *Robert M. Aikin*[1]; [1]Case Western Reserve University, Dept. Mats. Sci. & Eng., 10900 Euclid Ave., Cleveland, OH 44106 USA

The tensile and axial fatigue properties of A356 (Al-7Si-0.35Mg) and A357 (Al-7Si-0.55Mg) produced by both the squeeze and semi-solid casting processes have been examined. The samples tested were all taken from commercial automotive castings. Due to the lack of porosity and other large defects, very high tensile elongation's and very good fatigue properties were exhibited by all of the samples. The influence of microstructure on the tensile and fatigue properties of these samples will be discussed with respect to the significant differences in Si distribution produced by the squeeze and semi-solid casting processes.

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OPTIMISATION OF QUALITY OF HIGH PRESSURE DIE CAST MAGNESIUM ALLOYS: *A. K. Dahle*[1]; S. Sannes[2]; D. H. St. John[1]; H. Westengen[2]; [1]The University of Queensland, Dept. of Mining, Minerals and Mats. Eng., CRC for Alloy and Solidification Technology (CAST), Brisbane, Queensland 4072 Australia; [2]Norsk Hydro Research Centre, Porsgrunn Norway

Die cast magnesium components are finding increased use worldwide because of the excellent castability and properties that magnesium alloys can offer. Because of the excellent flow characteristics of molten magnesium, high-pressure die casting of thin-walled components is particularly suitable with magnesium. The use of magnesium die castings in automotive applications is currently increasing rapidly with new applications including components such as instrument panels, steering wheels, transmission housings, door frames and seat frames. These applications require optimisation of the quality and performance of the castings. It has been found that bands of porosity or segregation which follow contours parallel to the surface of the casting are formed under certain casting conditions in thin-walled magnesium high pressure die castings. The presence of this type of defect can have a significant effect on the mechanical properties. The present paper provides a rationale for understanding the origin of these defect s which is related to the solidification behaviour, the mushy zone rheological properties and the filling pattern of the casting with associated shearing of the mushy zone. The effect of varied casting conditions on the casting integrity and the appearance of the bands is investigated and rationalized by the theory. Methods to optimise the process parameters to control the occurrence of the banded defects and thereby optimise the quality of high pressure die cast magnesium components, are outlined.

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FATIGUE BEHAVIOR OF A356.2 CASTING ALLOYS WITH GRADIENTS IN DENDRITE ARM SPACINGS AND POROSITY: Bin Zhang[1]; David R. Poirier[1]; *Weinong Chen*[2]; Q. T. Fang[3]; [1]University of Arizona, Mats. Sci. and Eng., AME Bldg. 119, Tucson, AZ 85721 USA; [2]University of Arizona, Aerospace and Mech. Eng., AME, Tucson, AZ 85721 USA; [3]Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA

Low and high cycle fatigue lifes of A356.2-T6 cast aluminum alloy with various secondary dendrite arm spacings (DAS, 15um-55um) and porosity (10um-500um) levels in the microstructure were measured under both axial and bending loading conditions at strain/stress ratios of -1 and 0.1. SEM and optical microscopy observations revealed that high-cycle fatigue cracks initiated at near surface microstructure inhomogeneities such as porosity, oxides inclusions and eutectic silicon clusters, whereas low-cycle fatigue cracks initiated from shear slips which were also influenced by DAS and porosity. Different fatigue fracture features were related to the orientation and localized stress states of the underlying dendrite microstuctures. The effects of porosity, DAS, eutectic silicon particles on the fatigue crack propagation were also investigated.

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CREEP MECHANISMS IN DIE CAST MAGNESIUM ALLOYS AZ91D AND AE42: *Keun Yong Sohn*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Dept. of Mats. Sci. and Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Mats. Sci. Dept., Scientific Research Lab., Dearborn, MI 48121 USA

Magnesium alloys for powertrain components such as transmission case require good creep resistance at elevated temperatures. This study involves the characterization of creep of high pressure die-cast magnesium alloys, AZ91D and AE42. The effect of temperature and stress on creep behavior was investigated and correlated with cast microstructures. The creep strain rate, stress exponent and activation energy were

investigated to identify the responsible creep mechanisms for each alloy. AZ91D showed a very short range of steady state creep (better characterized as a minimum creep rate). AE42 showed a relatively long range of steady state creep, especially at lower stress levels. The stress exponent of steady state creep for AZ91D was about 5.5-6 at 150°C, while that of AE42 changed as the applied stress increased from 40 to 90 MPa. TEM investigation of crept specimens have been conducted to characterize differences in deformation mechanisms between AZ91D and AE42. Directions for the development of new creep resistant magnesium alloys will also be discussed.

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EFFECTS OF IRON PHASES ON THE MECHANICAL PROPERTIES OF A CAST ALUMINUM ALLOYS: *A. Rodriguez*[1]; [1]Faculted de Ingenieria Mecanica y Electricia Mexico

Automotive components require strict quality control to withstand the working conditions to which they are subjected to, Mechanical properties of aluminum alloys cast in permanent mold properties are affected by iron intermetallics, these hard and brittle phases develop during solidification and increase the possibility of developing cracks in the piece. A series of experimental trials were carried out in a type 319 alloy, content of iron was fixed at two levels: 0.3 and: 0.7% in weight. The melt was refined with a Ti-B master alloy and modified with strontium. Samples were poured in metallic and silica sand moulds, the solidification process was recorded with type K thermocouples inserted in the moulds. Thermal analysis was employed to obtain information related to the precipitation of iron phases, as well as the other reactions that take place during solidification. Mechanical tests were realized and the mechanical properties were correlated to the aspect and size of the iron phases.

CARBON TECHNOLOGY: Cathode Material & Corrosion

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

Tuesday PM Room: 6D
March 2, 1999 Location: Convention Center

Session Chair: Daniel Dumas, Carbone Savoie, Venissieux Cedex, France

2:00 PM SESSION CHAIRMAN INTRODUCTION

2:05 PM
PRODUCTION AND PERFORMANCE ASPECTS OF RAMMING PASTE: *Raymond C. Perruchoud*[1]; Urs Buhler[1]; Werner K. Fischer[1]; [1]R&D Carbon, Ltd., P.O. Box 362, Sierre CH-3960 Switzerland

Experiences of process optimization are reviewed which have been performed in smelters producing ramming paste. For a high and consistent paste quality, a proper selection of raw materials adapted to the process capabilities of a given plant is mandatory. Different possibilities of adjusting the binder viscosity and its impact on paste ramming or rolling as well as on paste ramming performance are addressed. The requirements for paste used in modern cells is assessed. Paste plant design and equipment requirements are evaluated.

2:30 PM
CARBONACEOUS GLUEING PASTES: *Siegfried Wilkening*[1]; [1]VAW Aluminium Technologie GmbH, P.O. Box 2468, Bonn, D-53014 Germany

A comprehensive survey as well as a substantial number of experimental results will be presented on various binders and filler materials of glueing pastes which can be carbonised and form a high-temperature bond. Examples will be given for the application of appropriate glueing pastes in the field of cathode construction and anode suspension.

2:55 PM
EVALUATION OF THE CONTACT RESISTANCE BETWEEN RODDING MIX AND COLLECTOR BAR USING THE 1/5 SCALE OF CATHODE CARBON: Shinjiro Toda[1]; Katsumi Tayama[1]; *Tsutomu Wakasa*[1]; [1]Nippon Denkyou KK., Kambara Works, Kambara 5600, Kambara-cho, Ihara-Gun, Shizuoka Pref. 421-3203 Japan

We made a study to evaluate the contact resistance between rodding mix and collector bars. As this contact resistance is one of the important factors for cathode lining drop in aluminum reduction pots, many studies of it have been made in the past. Its measurement, however, is very difficult, particularly at the lower contact pressure which is probably generated in the actual pot. We used a 1/5 scale of cathode carbon with the rodded collector bar and made a measurement of the contact resistance from the cathode voltage drop by giving it current. Locating molten metal on the upper surface of the cathode carbon, we formed an equipotential surface similar to an actual pot and calculated the contact resistance by measuring each voltage drop at the collector bar, rodding mix and cathode carbon. We also made a rodding mix comparison using this method.

3:20 PM
COMPUTER SIMULATION OF THE ANTHRACITE CALCINING FURNACE: *R. T. Bui*[1]; Jean Perron[2]; Jean-François Dessureault[3]; [1]Universite Du Quebec A Chicoutimi, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, Ltd., Jonquire, Quebec G7S 4K8 Canada; [3]Societe D'Electrlyse Et De Chimie Alcan Limitee, Jinquire, Quebec G7S4L2 Canada

Calcined anthracite is used in large quantity to manufacture the cathode blocks for use in the aluminium electrolytic cells. Calcination must be carried out properly to ensure a good quality of the anthracite especially in terms of low electric resistivity, and mechanical resistance at high temperature. Anthracite is calcined in cylindrical vertical electric furnaces in which the granular material flows down from top and is discharged at the bottom. Furnace control is based on the electric current passing through the furnace. Computer simulation is considerably complicated by the need to account for the heat treatment history of the anthracite. On one hand, the electric resistivity of anthracite measured at the high calcining temperature is not the same as that measured when the calcined anthracite has cooled down to lower temperature. On the other hand, a different cooling curve applies to each calcining temperature. A model is proposed that accounts for all above mechanisms. It simulates the furnace under its own control, which means that a control emulator is built into the furnace model. It is used to study furnace behaviour under different geometries (change in inter-electrode distance), perturbations (change in incoming anthracite properties), production schedules (increased or decreased production) or discharge schemes (discontinuous or continuous discharge). The model can be a useful tool for operating the furnace under changing production requirements all the while maintaining product quality.

3:45 PM BREAK

4:05 PM
GRAPHITIZED CATHODES WITH INCREASED ABRASION RESISTANCE: *Frank Hiltmann*[1]; [1]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, Frankfurt D-65933 Germany

Cathode wear, especially with graphitized blocks, has moved and more into the center of interest in the aluminium smelting industry since the average cell life has substantially increased during the last decades. Apart from TIB2 plasma-spray coating as protective layer cathode production-scale studies were performed in order to create a harder graphitized cathode material with increased abrasion, i.e. mechanical wear, resistance. The (non-coated) specimen showed an increase of mechanical strength by up to 35%. Abrasion tests yielded a wear reduction by up to 22%. Thermal and electrical properties remained practically unaffected making such materials promising alternatives for smelters in which cathode erosion is a major pot failure or shutdown criterion.

4:30 PM

CARBON CATHODE CORROSION BY ALUMINIUM CARBIDE FORMATION: Xianan Liao[1]; *Harald A. Oye*[1]; [1]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway

The carbon cathode corrosion by Al4C3 formation is electrochemical in nature. The corrosion in the mixture of aluminium and a cryolitic melt without an externally applied current is galvanic; little corrosion occurs in an alumina-saturated acidic melt without short-circuit between the carbon sample (cathode) and the aluminium (anode). When an external current is applied, the corrosion is electrolytic and is one of the parallel cell reactions. Solid Al4C3 is formed electrochemically and it may subsequently dissolve chemically into the melt. Addition of Al4C3 reduces the galvanic corrosion due to its suppression of carbide dissolution and the increased resistance polarisation. In the case of electrolytic corrosion, carbide addition increases corrosion at a low current density, has little influence at an intermediate current density and reduces corrosion at a high current density.

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REDUCED AND ENHANCED SODIUM EXPANSION BY CARBON BONDED COATINGS: Xianan Liao[1]; *Harald A. Oye*[1]; [1]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway

Effects of coatings consisting of fillings Al4C3, TiC, TiB2, TiO2 or their mixtures and a carbonaceous binder on sodium expansion are investigated. In the basic alumina saturated cryolitic melt and at a current density of 0.70 A/cm2, a 180% to 600% increase in expansion was observed for the Al4C3-coated carbon samples compared to the expansion of the corresponding uncoated samples. This strong expansion enhancement is attributed to diffusion hindrance by the coating resulting in an increased sodium concentration at the cathode surface. All coatings present a certain resistance to sodium penetration and slow down the sodium expansion initially for a period of time, depending mainly on the current density employed. Additives increasing the electrical resistivity of the coating increase the sodium expansion. The TiB2 coating is best wetted by molten aluminium and considerably reduces sodium expansion.

CAST SHOP TECHNOLOGY: Molten Metal Processing/Grain Refining II

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

Tuesday PM Room: 6C
March 2, 1999 Location: Convention Center

Session Chair: Dr. William C. Setzer, K.B. Alloys, Inc., Robards, KY 42452 USA

2:00 PM

AN EVALUATION OF METAL CLEANLINESS AND GRAIN REFINEMENT FOR 5182 ALUMINUM ALLOY DC CAST INGOT USING Al-3%Ti-0.15%C AND Al-3%Ti-1%B GRAIN REFINERS: *Anna J. Whitehead*[1]; Paul S. Cooper[2]; Ron W. McCarthy[3]; [1]Shieldalloy Metallurgy Corporation, Aluminum Products and Powders Division, P.O. Box 768, Newfield, NJ 08344-0768 USA; [2]London & Scandinavian Metallurgy Company, Ltd., Aluminium Division, Fullerton Rd., Rotherham, South Yorkshire S60 1DL England; [3]Reynolds Metals Company, Corporate Research and Development, 13203 N. Enon Church Rd., Chester, VA 23831-3122 USA

The Al-3%Ti-0.15%C grain refiner, which represents a new generation in grain refinement, has been used commercially for three years. Differences have been noted in melt cleanliness, ingot surface, and grain structure of the ingot cross-section when Al-3%Ti-0.15%C was substituted for Al-3%Ti-1%B. These differences have been evaluated by production scale DC ingot casting of aluminum alloy 5182 at the Reynolds Corporate R&D Cast House in Chester, VA. Ingots of aluminum alloy 5182 were produced with each grain refiner, Al-3%Ti-0.15%C and Al-3%Ti-1%B. The addition point of the grain refiner rod was varied among three locations: before the spinning degasser, between the spinning degasser and the ceramic foam filter, and after the ceramic foam filter. Molten metal cleanliness was characterized using the LiMCA II system and LAIS inclusion sampling. Comparisons were made of the grain refinement using the Aluminum Association's TP-1 Test, the ALCOA Cold Finger Test, and the etched ingot cross-sections. The effect of the grain refining nucleation particles, TiC and TiB_2, on the molten metal cleanliness and the interaction with filtration systems are described. A summary of the impact of the use of Al-3%Ti-0.15%C vs. Al-3%Ti-1%B grain refiner on commercial alloy production is discussed.

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FACTORS INFLUENCING THE EFFECTIVENESS OF THE Al-Ti-C GRAIN REFINER: *W. C. Setzer*[1]; A. Hardman[1]; G. W. Boone[1]; [1]KB Alloys, Inc., Corporate Technology, 3293 McDonald Rd., Robards, KY 42452 USA

The system Al-Ti-B has been used extensively over the past forty years for grain refining aluminum alloys and has been shown to be cost effective in providing increased casting rates without cracking and to prevent grain structure detrimental to surface appearance in a number of alloy systems. More recently, the Al-Ti-C system has been examined to determine whether it can offer specific benefits over the Al-Ti-B system. As with the Al-Ti-B system, the alloys reported on have included a number of Ti and C levels. However, there has not been an investigation which has definitively determined the relationship between grain refining effectiveness and C and Ti content for optimized processing conditions. This study was undertaken in order to examine in a systematic way the changes in grain refining effectiveness with changes in titanium content and carbon content in commercial purity aluminum containing various residual titanium levels. The discussion will include a review of these results and a literature survey, and their impact on the present view of the operative grain refining mechanism for both the Al-Ti-B and the Al-Ti-C systems.

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ALTIC GRAIN REFINERS WITH NUCLEATION POWER: *P. C. van Wiggen*[1]; J. K. Belgraver[2]; [1]KBM Master Alloys B.V., Kloosterlaan 2, TE Delfzijl 9936 The Netherlands; [2]KBM Affilips B.V., Waalkade 2, AT Oss. 5340 The Netherlands

AlTiC grain refiners form a relatively new alternative to the existing class of AlTiB type grain refiners for achieving fine equiaxed grain structures in aluminium alloys during casting and solidification. In this contribution, difference types of AlTiC master alloys are presented. Their characteristics will be evaluated and compared to the required specification criteria. The major part of the work consists of laboratory grain refinement test work and full-scale test trials essentially comparing the performance of AlTiC grain refiners relative to each other and to their AlTiB counterparts. The performance in different grain refining tests, in various alloys and using a range of test conditions will be covered. During the evaluation of the results important factors such as the Ti/C ratio in the AlTiC grain refiners and experience on the possibility of fade in the AlTiC system will be addressed.

3:15 PM

EXPERIMENTAL INVESTIGATIONS OF THE EFFECT OF VARIOUS ALLOYING ELEMENTS ON AS-CAST GRAIN SIZE OF WROUGHT Al-ALLOYS: *Hans Erik Vatne*[1]; Arild Håkonsen[1]; [1]Hydro Aluminium a.s., R&D Mats. Technology, P.O. Box 219, Sunndalsøra, Sunndal N-6600 Norway

The effect of typical alloying elements on as-cast grain size of wrought Al-alloys has been investigated. The experimental investigations included various additions of the elements Fe, Si, Mg, Mn, Cu, Cr and Ti as binary Al-alloys. The results demonstrated that Ti, Cr, Mg, Si

and Cu have a positive effect on grain size, while addition of Mn increases it. The effect of the individual elements corresponds well with the growth restriction they cause. Simple empirical relationships in terms of alloy addition and growth restriction have been developed. The relationships have been tested on multi-component alloy systems of typical commercial wrought alloys. The prediction power of such empirical relationships is good, but synergistic effects occur and the formation of phases between the various elements (above the solidification temperature) reduces the prediction power. Further, the effect of growth morphology on grain size was investigated by adding Ti and Si to several alloys. Addition of Ti is in this connection relevant for most product applications, as Ti is frequently added for purposes of grain refinement, while high addition of Si is relevant for foundry alloys. It appears that a transition in growth morphology takes place at high growth restriction factors, leading to coarse grain sizes. This transition is, however, very alloy dependent.

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SECONDARY DENDRITE ARM COURSENING DURING CHEMICAL GRAIN REFINEMENT IN SOME LIGHT METALS ALLOYS: *María Eugenia Noguez*[1]; Guillermo Salas[1]; Teresa Robert[1]; Gerardo Pacheco[1]; José G. Ramírez[1]; [1]Universidad Nacional de México, F. Química/ Dept. Ing. Metalúrgica, Reforma 321, San Miguel Xicalco, México City, D.F. 14490 Mexico

In certain conditions, chemical grain refinement does not promote a refining effect on secondary dendrite arm, on the contrary it tends to coarsen. This effect has already been pointed out for Al alloys by Mondolfo. This has practical implications in segregation, properties, cooling rate curves, and homogenization times which have not been enough discussed in the literature. A series of grain refined samples using different conditions have been cast, with a successful grain refinement, the dendrite coarsening has been found in an Al-5% Zn alloy refined with 0.1-0.5% Ti-B additions in sand molds and markedly in a Mg-5% Zn alloy using 0.03-0.12% hexacloroethane as grain refiner in metallic molds. Processing variables, structural parameters and properties are reported, related and discussed. A possible explanation regarding the absence of dendrite refinement when the grain does is included.

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CORRELATION BETWEEN GRAIN REFINING EFFICIENCY AND MICROSTRUCTURE IN Al-Ti-B MASTER ALLOYS: *Juan J. Del Campo*[1]; Mauro Martin[2]; Leopoldo Galan[2]; [1]Uuiversidad De Oviedo, Mats. Sci.& Eng. Metall., Escuela Ing. T. Industrial, Avda. Manuel Llaneza, 75, Gijon, Ppincipado De Asturias 33208 Spain; [2]Asturiana De Aleaciones,S.A. Aleastur, Quality Assurance Dept., Polígono de Maqua, Zeluan, Aviles, Pincipado De Asturias 33400 Spain

Al-Ti-B master alloys present different grain refining efficiencies depending upon the microstructure among other factors. A study was undertaken in order to establish a possible correlation between grain refining potency and microstructure in 3/1 and 5/1 rods produced in an industrial scale basis. The obtained results are comment considering the different grain refining theories proposed in the scientific literature.

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EFFECT OF GRAIN REFINERS ON INTERMETALLIC PHASES IN AA1XXX SIMULATED DC CASTINGS: *X. -G. Chen*[1]; [1]Alcan International, Ltd., Arvida Research and Development Center, Jonquiere, Quebec Canada

The effect of grain refiners on intermetallic phases in commercial purity alloys was investigated using a DC simulator, which has similar cooling conditions to the subsurface region of DC cast ingots. A commercial AA1xxx alloy was cast with different addition levels of AlTiB and AlTiC type grain refiners. The microstructure and related intermetallic phases were characterized by macro-etching, SEM and x-ray diffraction analysis. It was found that TiB2 and TiC promote the formation of the metastable AlmFe phase. The nucleation sites of intermetallic particles were observed and analyzed by using deep-etching and SEM techniques. The roles of TiB2 and TiC in promoting the fir-tree structure are discussed.

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THE GENERATION OF Al_m Fe IN DILUTE ALUMINIUM ALLOYS WITH DIFFERENT GRAIN REFINING ADDITIONS: M. W. Meredith[1]; A. L. Greer[1]; P. V. Evans[2]; R. G. Hamerton[2]; [1]University of Cambridge, Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ, UK; [2]Alcan International Limited, Banbury Laboratory, Southam Road, Banbury OX16 7SP, UK

$Al_{13}Fe_4$, Al_6Fe and Al_mFe are common intermetallics in commercial AAlXXX series Al alloys . Grain-refining additions (based on either Al-Ti-B or Al-Ti-C) are usually added to such alloys during solidification processing to aid the grain structure development. They also influence the favoured intermetallic and, hence, can affect the materials' properties. This work simulates commercial casting practices in an attempt to determine the mechanisms by which one intermetallic phase is favoured over another by the introduction of grain-refining additions. Directional solidification experiments on Al-0.3wt.%Fe-0.15wt.%Si with and without grain refiner are conducted using Bridgman apparatus. The type, amount and effectiveness of the grain-refining additions are altered and the resulting intermetallic phase selection followed. The materials are characterised using optical microscopy, scanning electron microscopy and X-ray diffraction. Al_mFe is seen to form when Al-Ti-B grain-refiner is introduced but only when the refinement is successful; reducing the effectiveness of the refiner led to Al_6Fe forming under all conditions. Al-Ti-C refiners are seen to promote Al_mFe at lower solidification velocities than when Al-Ti-B was used even though the grain structure was not as refined. These trends can be explained within existing eutectic theory, by considering growth undercooling.

CREEP BEHAVIOR OF ADVANCED MATS. FOR THE 21ST CENTURY: Engineering Aspects

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

Tuesday PM Room: 15A
March 2, 1999 Location: Convention Center

Session Chair: K. L. Murty, North Carolina State University, Raleigh, NC 27695-7909 USA; S. W. Nam, Korea Advanced Institute of Science and Technology, Chung Nam, Korea

2:00 PM INVITED PAPER

LIFE PREDICTION OF ZIRCALOY CLADDING BASED ON ANISOTROPIC THERMAL AND RADIATION CREEP: *K. L. Murty*[1]; [1]North Carolina State University, Dept. of Nuclear Eng., Hillsborough St., Raleigh, NC 27695-7909 USA

Zircaloy is used to clad UO2 fuel in water reactors and the integrity of the tubing is important to contain the radioactive species. These thin-walled cladding tubes experience complex biaxial stresses arising from external coolant pressure, internal pressure due to released fission gases, the pellet cladding interaction following radiation exposure, axial grid forces etc. The modeling efforts become complex due to the anisotropic nature of these hcp metals which arise from preferred grain orientations resulting from thermo-mechanical treatments during fabrication. We present here the thermal creep behavior of Zircaloy tubing studied using biaxial stressing through internal pressurization superimposed with axial load. The anisotropic creep behavior is described in

terms of creep loci at constant dissipation energy while the temperature and stress variations of creep-rate and strain were evaluated using the Dorn's creep formulations relevant to dislocation climb creep that is commonly observed in pure metals. The superimposed effects of radiation are considered in terms of stress-free radiation growth and radiation creep. Recent findings on the radiation exposure on thermal creep are described along with transients in creep following sudden stress changes. This work is funded by the National Sci. Foundation grant DMR-04818.

2:25 PM INVITED PAPER

DEVELOPMENT OF DIRECTIONALLY SOLIDIFIED NiAl ALLOYS FOR AIRCRAFT ENGINE APPLICATIONS: *S. V. Raj*[1]; I. E. Locci[1]; J. D. Whittenberger[1]; [1]NASA Lewis Research Center, Mats. Division, MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Binary nickel aluminide has several attractive thermophysical and oxidation resistant properties which make it a potential candidate for use as turbine blades and vanes in an aircraft gas turbine engine. However, it possesses poor high temperature creep and low temperature fracture resistance properties which have limited its use in these applications. One method for potentially improving these properties is through the use of directional solidification technology. This paper reviews current strategies being employed for improving the elevated temperature creep properties of NiAl alloys.

2:50 PM

OXIDE-DISPERSION-STRENGTHENED CHROMIUM BASED ALLOYS WITH A COMBINATION OF HIGH TEMPERATURE STRENGTH AND OXIDATION RESISTANCE: *M. Janousek*[1]; W. Köck[1]; H. P. Martinz[1]; M. Heilmaier[2]; [1]Plansee AG, Technology Centre, Reutte/Tyrol A-6600 Austria; [2]Institute of Solid State and Material Research Dresden, Institute for Metallic Mats., Dresden D-01171 Germany

New oxide dispersion strengthened chromium based alloys (chromium content > 50 weight%, trade name Ducrolloy) for structural parts in oxidising or aggressive atmospheres at high temperature have been investigated. There are two groups of chromium alloys that differ by the type of forming oxide layer: Group A, e.g. the alloy Cr-5Fe-1Y_2O_3, with chromium oxide layers and group B, e.g. Cr-44Fe-5Al-0,3Ti-0,5Y_2O_3, with alumina layers. While alloys from group A show an excellent oxidation resistance up to 1100ϒC, alloys from group B withstand temperatures of up to 1400ϒC. A high melting point between 1500 and 1900ϒC, a protective oxide layer and the particle strengthening with oxide dispersoids of typically 20-50nm diameter lead to an unique combination of high strength and oxidation resistance at temperatures far above the application temperature of conventional iron or nickel based superalloys. Hence, the extraordinary potential of these alloys is assessed with tensile and compressive creep tests under constant strain rate conditions at two different temperatures, namely 920ϒC for a comparison with the above mentioned alloy systems and 1400ϒC revealing the perspective for future applications.

3:10 PM

DISPERSION HARDENED PLATNIUM MATS. FOR EXTREME CONDITIONS: Bernd Fischer[1]; *Andreas Behrends*[1]; Dietmar Freund[1]; David F. Lupton[2]; Jürgen Merker[2]; [1]Fachhochschule Jena - University of Applied Science, Materials Technology, Tatzendpromenade 1b, Jena, Thüringen D-07745 Germany; [2]W.C.Heraeus GmbH, Heraeusstraße 12-14, Hanau D-63450 Germany

Materials to withstand extreme conditions are becoming increasingly necessary for developments in high technology fields. Because of its high melting point, good high-temperature strength and chemical stability the noble metal platinum is used in the high temperature range under the simultaneous influence of thermal, mechanical and chemical loadings. In spite of its high price platinum is well suited to being used in glass melting plants for highly stressed components as well as in space technology e.g. for rocket engines. These extreme conditions require materials with improved properties. The paper will report on examinations of the microstructure and properties of dispersion hardened platinum and platinum alloys. These materials are characterized by high levels of strength and low creep rates together with sufficient ductility in long term use at highest temperatures. Small zirconium and yttrium oxide particles incorporated in the matrix effect not only these properties but also a stable fine-grained structure. The excellent chemical stability of platinum against glass melts has been maintained. The dispersion hardened platinum materials have good weldability and even in the welded state show a significantly higher stress-rupture strength than pure platinum. These properties will be discussed on the basis of extensive stress-rupture strength tests on platinum materials in various conditions at temperatures between 1200ϒC and 1700ϒC, corrosion tests in glass melts, metallographic examinations, scanning electron microscopy (SEM) and chemical microanalysis (EDX, SIMS).

3:30 PM BREAK

3:40 PM

TEXTURE AND DEFORMATION OF SUPERPLASTIC ALUMINUM ALLOYS: O.A. Ruano[1]; M.T. Perez-Prado[1]; G. Gonzalezx-Doncel[1]; T. R. McNelly[2]; [1]Centro Nacional de Investigaciones Metalurgicas, C.S.I.C. Avda. Gregorio del Amo 8, 28040 Madrid, Spain; [2]Department of Mechanical Engineering, 700 Dyer road, Naval Postgraduate School, Monterey, CA 93943-5146

The deformation mechanisms in fine-grained materials deformed in the superplastic range of temperatures and strain rates are not fully understood. There are at least four different viewpoints on the roles of grain boundary sliding and slip: a) deformation occurs by grain boundary sliding with slip as the accommodation mechanism; b) grain boundary sliding and slip occur simultaneously and both contribute to deformation; c) slip occurs in the initial stages of deformation, with a transition to slip-accommodated grain boundary as microstructure evolves during straining; and d) slip predominates during superplastic deformation. Here, X-ray texture analysis methods have been coupled with recently developed computer-assisted electron backscatter pattern analysis techniques to assess the operative deformation mechanisms in various superplastic aluminum alloys. Superplastic aluminum materials may be classified according to the mechanism of transformation during annealing after deformation processing. In materials that transform by a continuous recrystalliztion reaction, superplastic flow occurs by grain boundary sliding accompanied by slip, wherein intra-granular slip occurs on two slip systems. When such materials are deformed outside of the superplastic regime, slip predominates but still occurs only on two slip systems. In alloys that transform by discontinous, or primary, ecrystallization, grain boundary sliding predominates during superplastic deformation, although slip may also occur as an accommodation mechanism. During deformation under non-superplastic conditions, slip takes place on five independent slip systems.

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SOLUTE-DRAG CREEP AND TENSILE DUCTILITY IN Al ALLOYS: *Eric M. Taleff*[1]; Paul E. Krajewski[2]; [1]University of Texas, Aerospace Eng. and Eng. Mech., ASE/EM, C0600, Austin, TX 78712-1085 USA; [2]General Motors Company, Global R&D Operations, Bldg. 1-6, Box 9055, 30,500 Mound Rd., Warren, MI 48090 USA

Solute-drag creep is an important deformation mechanism at warm-working temperatures in Al alloys containing Mg. The high strain-rate-sensitivity resulting from solute-drag creep, m = 0.3, can lead to enhanced tensile ductility, an important benefit in the commercial forming of complicated shapes. Mechanical testing of commercial Al alloys 5754 and 5182 has produced tensile ductilities in excess of 100% at temperatures of 300ϒC and higher and initial strain rates of 0.01/s. Tensile ductilities of less than half those found for 5754 and 5182 were measured for commercial alloy 7150 and an experimental Al alloy containing 5 wt pct Zn, Al-5Zn. These differences in ductility result from the primary solute additions in each material, Mg in 5182 and 5754 and Zn in 7150 and Al-5Zn. Mg leads to solute-drag creep and high ductility while Zn does not. The tensile ductilities of the two commercial 5xxx-series alloys studied do not reach those observed in low-impurity, binary Al-Mg materials, which have provided tensile ductilities of up to 325%. The ductility of the commercial alloys is limited by cavitation during solute drag creep arising from Fe, Si, and Mn additions.

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MODELLING THE ANISOTROPIC PRIMARY AND SECONDARY CREEP BEHAVIOR OF SINGLE CRYSTALS AND COM-

PARISON WITH EXPERIMENTALLY OBSERVED BEHAVIOR OF VARIOUS ALLOYS: *Uwe Glatzel*[1]; [1]Metallische Werkstoffe, Friedrich-Schiller-Universitaet Jena, Loebdergraben 32, Jena D-07743 Germany

A material model is proposed which describes single crystal creep behavior by evolution equations for dislocation densities on individual slip systems. An interaction matrix determines the influence from one glide system to the other. Assuming a face centered cubic crystal, allowing deformation on octahedral glide planes and cube glide planes with a Burgers vector of the type a/2 <110>, nine independent parameters of the interaction matrix can be distinguished. A parameter check of the nine independent parameters has been carried out, showing the influence of parameters on specific orientations of the load axis. If one assumes dislocation interaction of a glide system only with itself a smooth behavior is predicted with a maximum creep rate for [001] orientation, followed by [011] and [111]. This behavior has been observed for single crystal single phase Mats., such as Ni_3(AlTiTa) and a nickel solid solution crystal. If a strong interaction is assumed, the orientation dependent creep behavior is not at all smooth, instead it shows a sharp drop in creep rates mainly in symmetric positions of the standard orientation triangle. The orientations with highest creep rates are in this case those which favor single glide. Highly symmetric orientations, such as [001], [011] and [111] have strongly decreased stationary creep rates. Single crystal Nickel based superalloys show a similar behavior.

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DENSIFICATION CHARACTERISTICS OF AMORPHOUS ZrO2-Al2O3 POWDERS: *Ashutosh Suresh Gandhi*[1]; Vikram Jayaram[1]; Atul H. Chokshi[1]; [1]Indian Institute of Science, Dept. of Metall., Sir C.V. Raman Ave., Bangalore, Karnataka 560012 India

Amorphous ZrO2 Al2O3 powders produced by spray pyrolysis have been hot pressed up to ~98% relative density at temperatures of 923K or less under uniaxial pressures up to 750 MPa. The dense amorphous pellets have been crystallized to form bulk ultrafine microstructures with grain sizes in the range 10 nm to ~300 nm, with metastable and stable phases. The pronounced ability of the amorphous phases in ZrO2-Al2O3 to densify during hot pressing at very low temperatures has been investigated through hot pressing and compressive hot deformation experiments. The results of densification studies over a range of temperatures and applied pressures have been analyzed on the basis of the deformation characteristics of the fully dense amorphous compacts. The behavior of the amorphous powders has been compared with the existing models of viscous sintering.

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CREEP BEHAVIOR OF SiC/SiC CERAMIC MATRIX COMPOSITES: *Shijie Zhu*[1]; Mineo Mizuno[2]; Jianwu Cao[2]; Yutaka Kagawa[3]; [1]University of Electro-Communications, Dept. of Mech. and Control Eng., 1-5-1 Chofugaoka, Chofu, Tokyo 182-8585 Japan; [2]Japan Fine Ceramics Center, Atsuta-ku, Nagoya, Aichi 456 Japan; [3]The University of Tokyo, Institute of Industrial Sciences, Minato-ku, Tokyo 108 Japan

The tensile creep tests of SiC fiber reinforced SiC composites at 1000-1400ϒC were conducted. For the Standard SiC/SiC composite, the apparent stress exponent and activation energy for creep increase with a decrease in stress. The threshold stress approach was used to interpret the experimental data. For the Enhanced SiC/SiC composite, the apparent stress exponents for creep at high stresses are 12-14, but become 2 at low stresses. Creep of SiC matrix is attributed to control the creep rates of the SiC/SiC composites. Creep fracture mechanism and life prediction method were also discussed.

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STRENGTH AND CREEP BEHAVIOR OF Si3N4 WITH Yb2O3 AS A SINTERING AID: *Yesha Zheng*[1]; [1]University of Aveiro, Dept. of Eng.Ceramics and Glass, Aveiro 3810 Portugal

Si3N4 is one of the most promising engineering ceramic materials because of its excellent thermomechanical properties. Recently, Yb2O3 was found to be effective as a sintering aid of Si3N4 in improving the mechanical properties, especially high-temperature strength, of the material due to the formation of crystalline phases at the grain boundary. The heavier lanthanide oxides, Yb2O3, has high melting points. Microstructures typical for in situ toughened Si3N4 or self-reinforced ceramics, i.e., were observed. All materials contained thin amorphous films separating the grains. The amorphous intergranular films along grain boundaries revealed excess ytterbium and oxygen. The thickness of the intergranular films was about 1.0 and 2.5 nm for the grain boundaries and the phase boundaries, independent of additive content and heat-treatment history. Fracture toughness was dependent upon the morphology of microstructure. The retention in room-temperature strengths displayed at 1300ϒC was 80-90%, with no evidence of inelastic deformation preceding failure. The observed increase in fracture resistance was mainly attributed to crack deflection and crack bridging mechanisms. The steady-state creep rates, at 1400ϒC inflexural mode are the lowest reported for sintered Si3N4. The creep behavior was found to be strongly dependent on residual amorphous phase viscosity as well as on the oxidation behavior of these materials. The thickness and viscosity of the amorphous intergranular films are believed to play an important role in the high-temperature mechanical properties.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Electromagnetic - Emerging Technologies

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

Tuesday PM Room: 2
March 2, 1999 Location: Convention Center

Session Chairs: Rene Moreau, Institute National Polytechnique de Grenoble, Lab. EPM-MADYLAM (UPR CNRS A 9033), F-38402 Saint Martin d'Heres, Cedex France; Peter Davidson, University of Cambridge, Dept. of Eng., Cambridge, CB2 1PZ UK

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INFLUENCES OF MAGNETIC, ELECTRIC, GRAVITATION, AND TEMPERATURE FIELDS ON ONSET OF HYDRODYNAMIC FLOWS IN LIQUID METALS: *Alexander I. Raitchenko*[1]; [1]National Ukrainian Academy of Science, Institute for Problems of Mats. Sci., 142, 3, Krzhyzhanovsky St., Kiev 252142 Ukraine

Liquid metals under action of various fields (influencing simultaneously together or separately) may be in equilibrium or convective movement states. Knowledge of conditions of equilibrium and of convection onset has theoretical importance and practical value. Named conditions depend radically, in particular, on electroconductivity and on temperature coefficient of electroconductivity of liquid metals. Factors influencing equilibrium-convection onset conditions (besides the thermal expansion in the gravitational field) include the magnetic and electric fields also. If the spatial Lorentz-force originating from the crossed electric and magnetic fields' action is directed downwards its effect strengthens the gravitational action, and value of critical Rayleigh number must decrease; opposite situation (increasing of critical Rayleigh number) should be observed in case of Lorentz-force directed upwards. The extent of such an influence is determined by dimensionless complex[2] relation of spatial Lorentz- force to hydrostatic head of fluid column with unit height in the gravitational field. It is ascertained that the competition between the electromagnetic force due to non-unifor-

mity of electroconductivity on account of its temperature dependence and the buoyancy force may have the ambiguous effect: both increase or decrease of the critical Rayleigh numbers. The effect of such a balance is measured by value of corresponding dimensionless complex which may be named magnetic-electric-thermal-resistive-buoyant (METRB) criterion (number). Influences of electromagnetic-gravitational-hydrostatic relation number and METRB number can strengthen or weaken one another; in particular case they can "annihilate" mutually, and then the critical Rayleigh numbers must be remained invariable. Liquid Mg, Zn, and Cd near their melting points have positive temperature coefficient of electroconductivity values in contrast to rest metals, thus their behavior in many situation are different than for majority other liquid metals. The proposed conception is applicable to the analysis of situations in liquid metals under combinations of various fields.

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ANALYSIS AND FINITE ELEMENT SIMULATION OF MHD FLOWS WITH AN APPLICATIONS TO LIQUID METAL PROCESSING : *A. J. Meir*[1]; P. G. Schmidt[1]; [1]Auburn University, Dept. of Mathematics, Parker Hall, Auburn, AL 36849-5310 USA

We describe and analyze a finite element method for approximating the solutions of the equations of viscous, incompressible magnetohydrodynamics posed on a bounded domain with a nonideal boundary. Our method is based on a novel formulation of the underlying partial differential equations as a system of integro-differential equations. We describe the results of some recent numerical experiments relevant to metallurgy flow phenomena.

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NUMERICAL MODELING OF ELECTROMAGNETICALLY-DRIVEN TURBULENT FLOWS USING LES METHODS: F. Felten[1]; *Yves Fautrelle*[1]; Y. Du Terrail[1]; O. Metais[2]; [1]Institut National Polytechnique de Grenoble, CNRS-EPM, ENSHMG, B.P. 95, 38402 Saint Martin d'Heres Cedex France; [2]LEGI-INPG ENSHMG, P.B. 95, 38402 Saint Martin d'Heres Cedex France

Fluid flows generated by electromagnetic stirrers have been extensively investigated. Many works have been published on the subject. In most of the previous works, one-point closure models, e.g., as k-e model, have been used. Those models yield fairly good results as for the mean velocity prediction. However, they have two main drawbacks: (i) The turbulence is not predicted accurately, (ii) Such models are not well fitted for problems where the electromagnetic force are unsteady as in the low frequency magnetic stirring. We deal with the prediction of electromagnetically-driven turbulent flows by means of a Large-Eddy-Simulation method (LES). The method stems from the Smagorinsky eddy viscosity model. The model is applied in the case of a liquid metal pool submitted to a polyphase linear electromagnetic stirrer. We investigate two cases: (i) The frequency of the magnetic field is equal to 50 Hz; in that case the effects of the pulsating part of the Lorentz forces may be neglected; (ii) The frequency of the magnetic field is low (f = 1 Hz), then the oscillating part of the electromagnetic forces is taken into account. The LES predictions agree well with the mean velocity measurements, as does the standard k-e model. However, as for the turbulent kinetic energy predictions, there is a large discrepancy between the two models. When the oscillating of the Lorentz forces is taken into account, the computations show that the fluid flow is sensitive to the unsteady part of the forces. Without any fluid flow measurements, we only compare the cases with and without an oscillating part. We find that the mean velocity amplitudes are not affected by the fluctuating component of the force. As for the turbulence parameters, the turbulent kinetic energy is also weaker than in the previous case, whilst the turbulence length scale decreases.

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MATHEMATICAL MODELING OF TURBULENCE IN AN ELECTROMAGNETICALLY-LEVITATED NICKEL DROPLET: *Livia M. Racz*[1]; Shaun R. Berry[1]; Robert W. Hyers[2]; Behrouz Abedian[1]; [1]Tufts University, Dept. of Mech. Eng., Anderson Hall 204, Medford, MA 02155 USA; [2]MIT, Mats. Sci. and Eng., 77 Massachusetts Ave., Rm. 4-033, Cambridge, MA 02139 USA

Electromagnetic levitation is a containerless technique of great interest in materials processing. The presented work represents an effort to improve the understanding and prediction of turbulent flow inside electromagnetically-levitated droplets. It is shown that the flow field in a test case, a nickel droplet levitated under microgravity conditions, is a low Reynolds number turbulent flow, i.e. in the transitional regime between laminar and turbulent. Past research efforts have used laminar, enhanced viscosity, and k-e turbulence models to describe these flows. The method used in our study is the RNG algorithm. We show that an accurate description of the turbulent eddy viscosity is critical in order to obtain realistic velocity fields, and that the turbulent eddy viscosity cannot be uniform in levitated droplets. In the RNG method there are no characteristic length or time scales associated with the flow, thus allowing such anisotropic features to be captured. We perform calculations and analyses for two cases (1) small, nondeforming, spherical droplets, and (2) deforming, oscillating droplets. In addition to flow field calculations, we simulate numerically the oscillating drop method of thermophysical property measurements, and compare calculated values of viscosity and surface tension to experimentally-determined values.

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OSCILLATORY AND ROTATIONAL INSTABILITIES IN ELECTROMAGNETIC LEVITATION: J. Priede[1]; *Gunter Gerbeth*[1]; [1]Research Center Rossendorf, Inc., P.O. Box 510119, Dresden D-01314 Germany

We consider three different mechanisms which can cause spontaneous rotation or oscillation of a levitated spherical body. The first is that due to the virtual coupling between the electric current passing through the magnetic system and the variation of position of the body. This mechanism can result in unstable mass center oscillations of a levitated solid body as well as in increasing shape oscillations of a molten sample. Another type of instabilities may occur because of the coupling between the motion and the electric currents induced in the body. This effect can cause a spontaneous rotation of the body setting in as the frequency of the alternating magnetic field exceeds certain critical threshold depending on the configuration of the field. The third mechanism is due to the finite diffusion time of the magnetic field into the body which results in a delay of the induced currents with respect to the variation of body position. This may cause unstable oscillations of the mass center as well as those of the shape of liquid sample.

4:10 PM BREAK

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BEHAVIOUR OF THE SOLID ALLOY PARTICLES DISPERSED IN LIQUID TIN UNDER THE ELECTRIC CURRENT PASSAGE: *Alexander I. Raitchenko*[1]; Victor P. Popov[1]; Alexander V. Derevyanko[1]; [1]National Ukrainian Academy of Sciences, Institute for Problems of Mats. Sci., 142, 3, Krzhyzhanovsky St., Kiev 252142 Ukrainian

If an electric current flows along some electroconductive suspension in columnar form the solid particles are experienced thermal, mechanical, and electromagnetic influences. The objects were suspension Sn (liquid) - Ni-Cr-Mo-W alloy (solid particles). This is refractory alloy on the Ni base. The direct constant current (with density ~106 A/m2) flowed through the named suspension during 180-300 sec. At the beginning of experiments samples were ones containing particles suspend uniformly in volume. Although density of solid particles was ~9000 kg/m3, i.e. higher than that of liquid Sn (~6800 kg/m3), after end of this processing in samples was nonuniform distribution of particles in volume of samples such: concentration of solid particles in lower part was smaller than in upper part of samples, and simultaneously their concentration is increased near outer surface. Firstly, there was probably natural convection of Benard-Raileigh type which had carried solid particles upwards, and, secondly, in perpendicular directions was action of spatial Lorentz-forces directed to center. The knowledge of named phenomena can be useful for elaboration of technology of dispersed materials and development of analogues fields of materials science.

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ELECTROMAGNETIC CONTROL OF LIQUID METAL JETS: D. J. Short[1]; *P. A. Davidson*[1]; [1]University of Cambridge, Dept. of Eng., Trumpington St., Cambridge CB2 1PZ UK

In this paper we discuss an electromagnetic means of controlling high-temperature, liquid-metal jets of small diameter (5mm). Operating on the combined principles of partial magnetic levitation and magnetic field concentration via a segmented copper cylinder, this will provide a pollution free means of modulating, stabilising and positioning the jet. This paper describes the design and test of a laboratory prototype based on experiments performed with liquid tin and aluminium, as well as our numerical model of the process. We also discuss the potential applications of our device in the handling of superalloys, particularly in the context of sprayforming.

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THE CROSSED ELECTRIC AND MAGNETIC FIELDS IMPACT ON WAVE FLOWING OF LIQUID METAL LAYERS: *Alexander I. Raitchenko*[1]; [1]National Ukrainian Academy of Sci., Institute for Problems of Mats. Sci., 142, 3, Krzhyzhanovsky St., Kiev 252142 Ukraine

An electroconductive viscous liquid may flow in laminar or turbulent regime under Lorentz-force born by crossed electric current and magnetic field. When there is a starting flat layer between hard and free boundaries the spatial Lorentz-force can create a specific stationary regime of flowing: free surface will transform into wavy one, and movement in the depth will be not laminar and not turbulent, but this regime will be characterized by organized forms with indications of "rolling of long cylindrical drops". It was found that wavy regime is more advantageous energetically than laminar one. Mean thickness of wavy layer is becoming lesser with movement in the direction of Lorentz-force. Length of wave is determined by viscosity, density, surface tension, specific flow of liquid, and Lorentz-force. Limit of wavy regime is determined by most long of wave which is directly proportional to the critical value of specific flow and inversely proportional to the kinetic viscosity. The proper critical value of Reynolds number is determined by dimensionless combination containing surface tension, density, viscosity, and Lorentz-force. The named dimensionless combination in our case is some criterion substituting the known Kapitsa number for case of thin viscous liquid layers under gravity. It was shown that spreading of admixture by mechanism of wavy regime is more rapid than by common diffusion. Suggested conception can be a theoretical basis for elaboration of some specific liquid metals technologies.

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CONTROL OF THE POWDER DISPERSION IN INDUCTIVE PLASMAS BY USING A DOUBLE FLOW INJECTOR: P. Proulx[2]; *C. Trassy*[1]; [1]Laboratoire EPM-Madylam, ENSHMG, BP 95, 38402 Saint Martin d'Heres Cedex France; [2]Centre de Recherches en Technologie des Plasmas, Faculte de Genie, Sherbrooke, Quebec J1K 2R1 Canada

An inductive discharge presents, in the coil region, recirculation eddies due to the Lorentz forces, resulting in an axial backflow. Generally, this axial backflow disappears on the centerline when the central jet is strong enough to "pierce" the recirculation. However the external layer of the injection flow is skimmed by the remaining eddy, and particulates are carried to the discharge periphery. This results in treatment differences for the powder: particulates rejected to the plasma periphery are not treated. To avoid this disadvantage, it is common to introduce the injection pipe inside the discharge, so that its outlet is positioned after the recirculation eddy. The powders remain confined in the axial zone, to the expense of shortened residence time. In order to increase the efficiency of the particle trajectories, a double flow injector has been designed. It enables the particles to travel closer to the axis, while the outer schrouding gas is skimmed and passes through the electromagnetic eddies. This injector is made up of two concentric tubes. The powders are injected with the carrier gas in the inner tube. A schrouding gas, for instance pure argon, is injected in the annular space between the inner and the outer tubes. The powder is confined to the vicinity of the axis, resulting in a more homogeneous thermal treatment. Tungsten powders have been spheroidized with and without this device. Using the double flow injection, the spheroidization yield has been increased from 70% up to 95%. the design of such an injection device has been improved by modelling. A visualisation technique has been used for the experimental study. Injecting yttrium oxide into the discharge results in a colorful display of the 4000 K isotherm: the "cold" zone, below 4000, appearing as red, and the "hot" zone as blue. This visualization technique drastically simplifies the comparison between experimental and theoretical results. Furthermore, it is of great industrial interest since it is a very cheap and efficient way to obtain a picture of an important isotherm. The comparison between the numerical and experimental investigation will be presented.

GENERAL ABSTRACTS: Session 6 - General Metallurgy

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

Tuesday PM — Room: 12
March 2, 1999 — Location: Convention Center

Session Chairs: Benji Maruyama, WL/MLLM, Wright Lab Mats. Directorate, WPAFB, OH 45433 USA; David Senor, PNL

2:00 PM

AN OVERVIEW OF THE DOE-OIT ALUMINUM PROGRAM: *Sara A. Dillich*[1]; *Toni Grobstein Marechaux*[1]; [1]U.S. Dept. of Energy, EE-20, 1000 Independence Ave. S.W., Washington, D.C. 20585—0121 USA

The goal of the DOE-Office of Industrial Technologies (OIT) Aluminum Industry of the Future program is to work in partnership with the aluminum industry to develop and deploy energy-efficient, and pollution prevention technologies. The program addresses technology needs and priorities identified in the Aluminum Industry Technology Roadmap (May, 1997) and the supplementary Inert Anode Roadmap (February, 1998). The portfolio consists of industrially cost-shared and partnered research in primary aluminum processing, recycling and semi-fabrication processing. The project partnerships, goals and anticipated benefits will be described, as will the project solicitation and selection process. Future plans for the program, and the relevance of other federally-funded R&D to the Roadmap goals will also be discussed.

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OZONE CONTROL OF CASTHOUSE PROCESS COOLING WATER IN A PRIMARY ALUMINIUM SMELTER: *David Rees*[1]; Peter Dann[1]; Derek Scott[2]; Peter Miller[3]; [1]Tomago Aluminium, P.O. Box 405, Raymond Terrace, NSW 2324 Australia; [2]ANCO Australasia, 102 Derby St., Silverwater, Sydney, NSW 2119 Australia; [3]Ionics Watertec, 895 Pacific Highway, Pymble, NSW 2073 Australia

Tomago Aluminium, a 400,000 tpa primary aluminium smelter in Australia has installed Ozone for biological control of its process water. The system was designed in Australian by Ionics Watertec Engeering, whilst Anco Australasia monitors the water quality and provides the technical support for the project. The motivation was initially to reduce operating costs, however investigations indicated that there were substantial environmental benefits to be achieved by the elimination of a part of, or all of, the chemicals being used to control the system. Also the existing chemicals appeared to be losing their ability to control biological growth, in particular, the legionella bacteria. Ozone commenced in January 1998 and after a short transition has operated without chemical addition since. There appears to be no doubt that the biological control is effective, the clarity and purity of the water has improved and initial testing indicates that the corrosion rate is acceptable, although some chemical corrosion control may be introduced after further testing around the total circuit to guarantee protection.

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PROTECTION OF METALS AGAINST MICROBIOLOGICALLY INFLUENCED CORROSION USING MOLECULAR SELF-ASSEMBLIES: *Rajendra Uddhav Vaidya*[1]; Susan Brozik[2]; Darryl P. Butt[1]; Larry E. Hersman[3]; Alina Deshpande[3]; Kestas Laurinavichius[4]; [1]Los Alamos National Laboratory, Metallurgy Group, MST-6, Mail Stop G

755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, NMT-6, Mail Stop G 755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Life Sciences Division, LS-7, Mail Stop M 888, Los Alamos, NM 87545 USA; [4]Russian Academy of Sciences, Institute of Anaerobic Processes, Pushchino, Moscow Region 142292 Russia

The effectiveness of a self-assembled silane coating as a corrosion inhibitor for beryllium, aluminum alloy 6061, and an alumina particle reinforced aluminum 6061 matrix composite, against microbiologically influenced corrosion (MIC) was demonstrated. Tensile tests and four-point bend tests on coated and uncoated samples were conducted to test the effectiveness of these coatings. Application of these self-assembled silane coatings to the sample surfaces was found to prevent degradation of the failure strength and displacement. In contrast, the uncoated samples exhibited a severe reduction in these mechanical properties in the presence of the aerobic and anaerobic bacteria. This study demonstrates the potential for developing fast, easy, and cost-effective MIC protection for metals using self assemblies.

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METALLOGRAPHIC PREPARATION AND QUANTITATIVE IMAGE ANALYSIS OF AUTOMOTIVE PAINT SPECIMENS: *Mathias Hoffman*[1]; William R. Creech[2]; [1]Buehler, Ltd., Lake Bluff, IL USA; [2]BMW Manufacturing Corporation, Spartanburg, SC USA

This paper focuses on the metallographic preparation of automotive paint specimens. Typically, 5 different layers make up the pain on a car part. These layers are analyzed for thickness and flaws using automated image analysis equipment. The integrity of the various layers is essential for the longevity of the overall part. Automotive paints are exposed to the natural elements and potential damage from flying debris eventually causing corrosion damage. Various tests have been established in the automotive industry to simulate the exposure of a car to the elements to improve the paint quality and process. Special techniques are used to prepare these specimens metallographically to achieve gray level differences of the various layers needed to use automatic image analysis for evaluation.

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PRODUCTION OF STEEL FOAMS: *Chanman Park*[1]; Steve R. Nutt[1]; [1]University of Southern California (USC), Dept. of Mats. Sci. and Eng., University Park, Los Angeles, CA 90089-0241 USA

Steel foams have some obvious advantages over aluminum foams, including superior mechanical strength, specific stiffness, high melting temperature, corrosion resistance, and reduced thermal conductivity. Both can be used as core materials in structures where weight efficiency or energy absorption is critical, although the technology for producing steel foams is currently undeveloped. In the present work, steel foams are produced using a PM (powder metallurgy) method that involves a foaming agent. In this method, steel powder is mixed with the granular foaming agent, and the mixture is cold-pressed. The compact is heated to the melting temperature, whereby gas is evolved from the foaming agent and the material is expanded into foam. Relative densities of 1/3-1/2 are achieved in the foams, with regular pores ranging from <0.5mm to several mm. Initial results from compression tests will be presented, along with observations of associated deformation mechanisms.

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SURFACE TREATMENT OF STEEL AND CARBON WITH OZONE FOR IMPROVED BONDING TO CONCRETE: Weiming Lu[1]; *Deborah D. L. Chung*[1]; [1]State University of New York at Buffalo, Mech. and Aerospace Eng. 608 Furnas Hall, Amherst, NY 14260 USA

Surface treatment using ozone was found to be effective for steel rebar and carbon fiber for increasing the bond strength to concrete. Steel rebar surface treatment involving ozone was more effective than those involving water immersion and sand blasting, which were in turn more effective than acetone treatment. Carbon fiber surface treatment involving ozone was more effective than those involving nitric acid, NaOH, H2O2 and acetic acid. For carbon, the effectiveness of the ozone treatment is due to the increase in surface oxygen concentration and the consequent improved wettability by water. For steel, it is due to a surface oxide layer and the consequent improved wettability. The ozone treatment increased the tensile strength, modulus and ductility of short carbon fiber reinforced cement paste, in addition to increasing the degree of fiber dispersion and reducing the drying shrinkage.

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LEACHING OF ZIRCON FOR THE REMOVAL OF URANIUM, THORIUM AND IRON: *Padmakar Ramchandra Khangaonkar*[1]; Meor Yusoff[2]; Kamarudin Hussin[1]; [1]Universiti Sains Malaysia, Perak Campus, Tronoh, Perak 31750 Malaysia; [2]Malaysian Institute of Nuclear Technology, Bangi, Kajang, Selangor 52100 Malaysia

Two samples of the mineral zircon from Malaysia were examined by leaching to achieve the removal of uranium, thorium and iron from zircon. One of the samples (containing 0.16% U, 0.08% Th and 0.22% Fe) was leached in 4M HCl at 70°C, which led to the removal of 21% of the uranium present, 26.2% of thorium and 39.8% of the iron present in the mineral. Prior thermal treatment at 600°C and grinding of the material to < 45 micron size, enabled the removal of upto 72.5%, 87.5% and 80% of the uranium, thorium and iron respectively. Leaching in 20% NaOH gave a relatively better removal of uranium but a lower degree of removal of thorium andiron. With a view to assess the relation between the leaching behaviour and the presence of amorphous zircon (metamict zircon) in the sample leached, the degree of metamictization was assessed by the crystallite size and lattice strain using X-ray methods.

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AN OVERVIEW OF THE DOE-OIT ALUMINUM AND MINING PROGRAMS: Sara A. Dillich[1]; Toni Grobstein Marechaux[1]; [1]U.S. Dept. of Energy, EE-20, 1000, Independence Ave. S.W., Washington, D.C. 20585 USA

The goal of the DOE-Office of Industrial Technologies (OIT) Industries of the Future program is to work in partnership with industry to develop and deploy energy-efficient, and pollution prevention technologies. The Aluminum program addresses technology needs and priorities identified in the Aluminum Industry Technology Roadmap (May 1997) and the supplementary Inert Anode Roadmap (February 1998). The Aluminum portfolio consists of industrially cost-shared and partnered research in primary aluminumprocessing, recycling and semi-fabrication processing. The Mining program is just getting started, with the Mining Vision document (September 1998) and plans for two solicitations in 1999. The project partnerships, goals and anticipated benefits will be described, as will the project solicitation and selection processes. Future plans for the programs, and the relevance of other federally-funded R&D to the Vision and Roadmap goals will also be discussed.

GENERAL RECYCLING OF MATERIALS: Regulations and Steel Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee

Program Organizers: Ilaria Accorsi, Chrysler Corporation, Product Quality, Toledo, OH 43606 USA; Isrun Bohlinger, Technical University of Berlin, Institute of Metall. Mats., Berlin D-10623 Germany; Brajendra Mishra, Colorado School of Mines, Dept. of Metall. & Mats. Eng., Golden, CO 80401-1887 USA

Tuesday PM — Room: 1A
March 2, 1999 — Location: Convention Center

Session Chairs: Robert L. Stephens, Asarco, Inc., Technical Service, Helena, MT 52602 USA; Ilaria Accorsi, Chrysler Corporation, Toledo Machining Plant, Perrysburg, OH 43551 USA; Isrun Bohlinger, Technical University of Berlin, Institute of Metall. Mats., Berlin, D-10623 Germany

2:30 PM INTRODUCTION AND WELCOME

2:35 PM INVITED PAPER

A HYDROMETALLURGICAL WAY TO RECOVER ZINC AND LEAD FROM EAF DUST: *Carla Lupi*[1]; M. Cavallini[1]; A. Ferrone[1]; D. Pilone[1]; P. P. Milella[2]; R. Mussapi[2]; [1]Universita degli Studi di Roma "La Sapienza", Dip. ICMMPM, Via Eudossiana 18, Roma 00184 Italy; [2]ANPA, Via Vitaliano Brancati 48, Roma 00144 Italy

In 1996 the Italian Agency for Environmental Protection (ANPA) has initiated a study of the environmental impact of the industrial sector of steel produced by Electric Arc Furnace (EAF). Within the framework of this study a hydrometallurgical process to treat EAF dusts was considered and developed. In this work the lead and zinc recovery from fumes coming from carbon steel production was studied. The zinc extraction consists in acidic leaching followed by SX-EW steps. The leaching sludge containing lead sulfates was treated to obtain pure lead salt and inert solid residue. The whole process has been developed not only to obtain marketable products but also to minimize effluents by recycling the main liquid streams. The solid waste mainly containing spinels was subjected to elution tests in order to verify its compatibility with environmental regulation.Next year the proposed process will be tested in a pilot plant.

3:00 PM INVITED PAPER

CHROMIUM LOSSES DURING REMELTING OF STAINLESS STEEL SCRAP: *Saad Megahed El-Raghy*[1]; H. A. Fayed[2]; [1]Cairo University, Metall. Dept., Faculty of Engineering, Cairo Egypt; [2]Delta Steel Mills, Mostorod, Cairo Egypt

Stainless steel scrap of different grades was remelted in an induction furnace. The extent of oxidation of both carbon and chromium was followed during both the melting down and the boiling process. Decarburization was carried out utilizing either air or oxygen blowing. During the melt down, Chromium loss was dependent on the condition of the furnace and the previous melt analysis. With melts of carbon content more than 0.25%, air was as efficient as oxygen blowing for carburizing, chromium losses were minimum. Increasing temperature and lowering carbon resulted in excessive chromium losses. A general correlation between Cr and C in the melt was observed which is: log [Cr]/[C] = A - (B/T). The paper will discuss these results compared to international experience.

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THE EFFECTS OF NIOBIUM AND TITANIUM ON Tin PRECIPITATION IN Fe- ALLOYS: *Xiuqing Li*[1]; [1]University of Leeds, Dept. of Mats., Woodhouse Lane, Leeds LS2 9JT UK

The paper describes the possibility of using Sn to form intermetallic compounds with the microalloying elements Nb, Ti and Al, which are commonly present in the steelmaking process. Two series of alloys based on Fe, Sn and Al with either Nb or Ti additions were prepared by arc melting under an argon atmosphere. The microstructure of the precipitates were studied by using SEM, TEM, EDS and microprobe analysis. It was found that the level of Nb addition has an effect on the formation of Sn-rich compounds. For a small Nb additions, no Sn containing compounds were found, while in the high Nb alloy, two types of Nb-rich phases were detected: a Laves phase (Fe_2Nb, hexagonal, a=0.4830 nm and c=0.7879) containing about 1 at % Sn and a Fe_2Nb_3 phase (cubic, a=1.1261nm) which is associated with alumina and not containing tin. In contrast, no Sn containing compounds were found with Ti additions. The experimental observations were compared with thermodynamic calculations performed using MTDATA.

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RECOVERY OF TIN FROM WASTE TINPLATES: *Srecko Stopic*[1]; Ilija Blagoje Ilic[1]; [1]University of Belgrade, Faculty of Technology and Metallurgy, Dept. of Nonferrous Metall., Karnegijeva 4, P.O. Box 5303, Belgrade 11 000 Yugoslavia

Recovery of tin from tinplate and production of good-quality detinned-plate for further processing in ironworks has been the subject of interest for many years. Tinplates are mostly used for production of tin containers for food industry and consumer goods. The research carried out so far included determination of the parameters of dissolution of tin from tinplates by alkaline solutions of NaOH and electrochemical recovery of tin from the alkaline solutions. During leaching, $NaNO_3$ and methanitrobenzoic acid (MNBA) as oxidants were studied. By leaching the waste tinplate which contains 0.31% Sn with a solution of 45 g/dm^3 NaOH, 28 g/dm^3 Na_2CO_3 and 15 g/dm^3 MNBA at a temperature of 70^{o}C for 60 min, a degree of tin leaching of 97% was attained. Using MNBA acid facilitates the oxidation of Sn^{2+} into Sn^{4+}, suitable for obtaining compact but not spongy Sn cathode deposit, as it was the case earlier. Recov ery of tin by electrolysis from solutions obtained by leaching was studied to determine the effect of electrolyte composition on the quality of cathode deposit and the degree of electric current efficiency. Experimental results obtained at a laboratory level were checked on an enlarged laboratory set-up where continuous leaching of tin from waste tinplate and its electrolytic recovery from the solution obtained were carried out. The experimental set-up operated under semi-industrial conditions, and about 1500 kg of waste tinplate was processed yielding about 1160 kg of tin (99% Sn).

4:15 PM BREAK

4:30 PM INVITED PAPER

THE REGULATORY ENCOURAGEMENT OF METALS RECYCLING - VETE A OTRO PERRO CON ESE HUESO: *Larry Southwick*[1]; [1]L.M. Southwick and Associates, Process Design, Extractive Metallurgy and Chemical Engineering, 992 Marion Ave., Suite 306, Cincinnati, OH 45229 USA

Many environmental regulations, especially those related to hazardous wastes, waste minimization and pollution prevention, have as a stated goal the recovery and recycling of wastes. These include RCRA (Resource Conservation and Recovery Act for hazardous and solid wastes) promulgated in 1976, TRI (Toxic Release Inventory, or SARA Title III reporting) of 1986 and the Pollution Prevention Act of 1990. Even regulations relating to Superfund sites have on occasion appeared to encourage recovery and reuse of contaminants. However, somewhere on the road between the statues and the regulations, and especially in administering regulations, recycling has lost emphasis. In many instances, the regulations have in fact both discouraged recycling as well as hindered creation of new technologies for recovery of usable constituents. This paper will examine several examples of the above situation as it applies to recovery and recycling of metals. The activities covered include wastes generated, efforts to delist wastes from hazardous classifications, contaminated site cleanup and pollution prevention. Also addressed will be the role of the US Bureau of Mines used to play in these activities and how its demise will likely hinder rational debate and competent government evaluation of alternatives.

4:55 PM PANEL DISCUSSION

EFFECT OF REGULATIONS ON THE RECYCLING INDUSTRY: Moderator: *Larry Southwick*[1]; [1]L.M. Southwick and Associates, Process Design, Extractive Metallurgy and Chemical Engineering, 992 Marion Ave., Suite 306, Cincinnati, OH 45229 USA

This panel will involve both regulators and practitioners discussing concepts and examples of recovering and recycling metals from wastes. Included will be both hazardous and solid wastes. Topics to be discussed will cover experiences dealing with regulations, how they impact handling and logistics of the wastes, what to do with the products and with residues from processing for recycle, what regulations do the cost, feasibility and business stability of recycling, the types of technologies that can be developed in such a situation and where new developments probably cannot succeed, Covered will be how the regulations might help the recycling industry, how they might hinder it, what areas of improvement are perceived, what in fact are the objectives of the different regulations, if and where those objectives are being achieved, and what future directionsmay be, both with regulations and with technologies.

5:55 PM CLOSING REMARKS

HIGH-TEMPERATURE SUPERCONDUCTORS: SYNTHESIS, FABRICATION AND APPLICATION: Fabrication & Characterization of BSCCO Tapes

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 Laboratory, Mats. Sci. & Tech. Div., Washington, D.C. 20375-5000 USA

Tuesday PM Room: 18
March 2, 1999 Location: Convention Center

Session Chairs: A. A. Polyanskii, University of Wisconsin, Applied Superconductivity Center, Madison, WI 53705 USA; Giovanni Grasso, INFM, Unita di Genova, Genova 16146 Italy

2:00 PM INVITED PAPER
DEVELOPMENTS OF Bi(2223) MULTIFILAMENTARY TAPES WITH LOW AC LOSSES: *Yibing Huang*[1]; Frank Marti[2]; Eric Walker[2]; Gregoir Witz[1]; Réne Flükiger[2]; [1]University of Geneva, Applied Physics Group, 20, Rue l'ecole de Médecine, Geneva CH-1211 Switzerland; [2]University of Geneva, DPMC, 24, Quai E-Ansermet, Geneva CH-1211 Switzerland

A significant reduction of ac losses Bi(2223) multifilamentary tapes with Ag sheaths has been achieved by using oxide (BaZrO3 and SrZrO3) barriers between filaments and by means of twisting process. Different ways to introduce oxide barriers in tapes with 19 to 95 filaments are presented. The effect of different configurations of the oxide barrier and filaments on ac losses will be discussed in detail. The deformation and twisting processes have been studied, which lead to a remarkable improvement on the homogeneity of the filaments and oxide barrier. The critical current density in the filaments varied between 10'000 and 25'000 A/cm2. The variation of the critical current density with bending strain and twisting process is shown to be similar to that of tapes without barriers. The decoupling effect can be gauged by the frequency at which loss shows a maximum in a low amplitude ac field applied perpendicular to the tape. So far, the frequency of the loss maximum, fm, in Ag sheathed tapes has been enhanced from 5 Hz (untwisted) to 116 Hz (13 mm in twist pitch length). The fabrication of long length tapes (>50 m) with oxide barrier for a prototype transmission cable demonstration cooperating with Pirelle will be reported.

2:20 PM INVITED PAPER
DAMAGE MECHANICS OF POLYCRYSTALLINE HIGH-T_c SUPERCONDUCTORS: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Mats. Sci. Division, Bldg. 480, P.O. Box 5000, Upton, NY 11973-5000 USA

The formation and propagation of cracks in high-T_c superconducting composites such as powder-in-tube-processed (PIT) BSCCO 2212 and 2223 wires and thick-film $YBa_2Cu_3O_x$ coated conductors play a significant role in limiting the obtainable critical current density and the service life of these conductors. In this talk I will discuss the use of damage mechanics[1] as a theoretical framework to describe the accumulation of microcracks during processing and the irreversible strain limit for polycrystalline HTSC conductors. This framework provides a theoretical basis for the description of the role of microstructure, texture, and similar variables and suggests how experimental measurements of elastic moduli, ultrasonic wave propagation, and normal-state electrical resistivity can aid in the characterization of the state of damage. 1. J. Lamaitre and J.-L. Chaboche, Mechanics of Solid Mats., Cambridge University Press (1990). This research was supported by the U.S. Department of Energy, Division of Mats. Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

2:40 PM INVITED PAPER
GRAIN BOUNDARY EFFECTS IN HIGH Tc SUPERCONDUCTORS: *K. L. Zeisler-Mashl*[1]; C. S. Pande[1]; R. A. Masumura[1]; [1]Naval Research Laboratory, Mats.. Sci. & Tech. Division, 4555 Overlook Ave., SW, Washington, DC 20375-5343 USA

A high critical current capacity is one of the most important properties needed in the newly discovered high Tc superconductors for large scale applications such as electric motors. Many factors control the critical current in these Mats.. We discuss why the low population of large angle grain boundaries and the grain alignment are most critical. Specifically, the c axis texture and a-b axis alignment seem to be most important. These results are verified by texture measurements on BSCCO tapes. Bi 2212 grain texture was determined for both c axis texture using (001) pole figures from 008 reflections and a-b axis texture using (115) pole figures. Current transport properties of these tapes were also measured and correlated with texture parameters obtained from contours of the pole figures. Crystallographic arrangement of the grains was inferred from the pole figures and was found to be consistent with a model based on global alignment of the c axis and the presence of colonies of grains differing mostly in c axis twist.

3:00 PM INVITED PAPER
EFFECT OF THE PREPARATION PARAMETERS ON GRAIN CONNECTIVITY, TEXTURE, AND PINNING PROPERTIES OF Bi(2223) TAPES: *Giovanni Grasso*[1]; Antonio S. Siri[1]; Frank Marti[2]; René L. Flukiger[2]; [1]INFM, Unità di Genova, Via Dodecaneso 33, Genova 16146 Italy; [2]Université de Genève, DPMC, 24, Quai Ernest-Ansermet, Geneve 4,1211 Switzerland

In spite of the high homogeneity that can be achieved on very long samples, Ag-sheathed Bi(2223) tapes require a further improvement of their transport properties, especially in presence of large magnetic fields. We analyzed the properties of 'state of the art' Bi(2223) tapes with self field critical current density exceeding 30'000 kA/cm2 over lengths of several meters, by various transport and magnetic techniques. From a comparison between these measurements we have individuated a domain of the H-T plane where the current density is still limited by the quality of the boundaries between adjacent grains and the polycrystalline superconductor behaves as a granular one and, on the contrary, the region where it behaves like a strongly-connected body. The temperature behavior of the intrinsic Bi(2223) current density has been also evaluated. At temperatures below 30K in zero applied field, the intragrain current density is about an order of magnitude larger than the transport current density. Finally, the effect of modifications of the heat treatment process on the pinning potential of the Bi(2223) phase will be presented.

3:20 PM BREAK

3:30 PM
CONTROLLED THERMAL PROCESSING FOR Ag-Bi2223 COMPOSITE TAPE: *Hengning Wu*[1]; Su Su Wang[1]; [1]University of Houston, Texas Center for Superconductivity, 3210 Cullen Blvd., Houston, TX 77204-5932 USA

The powder-in-tube technique has been widely used to fabricate Ag-Bi2223 composite tapes. For multi-filament Ag-Bi2223 tapes with very high critical current density, a major current limiting factor is microcracks formed during processing. A controlled thermal processing is developed to minimize the formation of the cracks, in which the temperature and oxygen partial pressure are adjusted simultaneously to maintain the phase stability of Bi2223 and alleviate the induced thermal stresses due to the mismatch of thermal expansion coefficients of silver and the superconducting phase. The phase stability of Bi2223 in the Ag-Bi2223 composite tape is investigated in a range of oxygen partial pressure and temperature to determine the proper processing parameters. The critical current densities are compared for different cases of controlled thermal processing and normal slow cooling treatments.

3:50 PM INVITED PAPER

DIRECT OBSERVATION OF PHASE FORMATION IN Ag-SHEATHED Bi,Pb(2223) MONOFILAMENTARY TAPES BY NEUTRON DIFFRACTION STUDY: *Enrico Giannini*[1]; Emilio Bellingeri[1]; Reynald Passerini[1]; Rene Flükiger[1]; [1]Université of Genève, Dépt. de Physique de la Matière Condensée, 24 quai Ernest-Ansermet, Geneva CH 1211 Switzerland

High temperature neutron diffraction measurements have been performed on Bi(2223)/Ag sheathed tapes at ILL high flux reactor in Grenoble. These tapes were submitted during the measurements to exactly the same annealing condition (Ramp rates, temperatures, times, atmosphere) as used for the preparation of high performance tapes. The especially designed experimental set up with a rotating sample older, allowed us to examine with a high precision the transformation of the precursors into the Bi(2223). Indeed the neutron diffraction measurements allowed the absolute determination of the amount of 7 different crystalline phases through the whole reaction process. In particular we found strong evidence of a partial melting at a few degrees lower than the optimal annealing temperature (838ŸC in air). These results support a nucleation and growth mechanism, thus confirming earlier results. Our measurements bring to light also the fundamental role of other cuprates mainly $(Ca,Sr)_{14}Cu_{24}O_{41}$ and Bi(2201) in the process and their behaviour under different annealing conditions. An important result based on the absolute phase quantification is that no decomposition of Bi(2223) was observed during the cooling process. The observed increase of the Bi(2212) phase upon the cooling is due to crystallisation from the remaining secondary phases. The refinement of the structure revealed that the newly formed Bi(2212) phase has different crystallographic parameters.

4:10 PM INVITED PAPER

MAGNETO OPTICAL INVESTIGATION OF MULTI-FILAMENTARY Ag/BSCCO-2223 TAPES: *A. A. Polyanskii*[1]; X. Y. Cai[1]; D. C. Larbalestier[1]; Q. Li[2]; R. Parella[2]; M. W. Rupich[2]; G. N. Riley[2]; [1]University of Wisconsin, Applied Superconductivity Center, 1500 Eng. Dr., Madison, WI 53705 USA; [2]American Superconductor Corporation, Two Technology Dr., Westborough, MA 01581 USA

The magneto-optical technique (based on the Faraday rotation in a ferrimagnetic Bi-doped garnet indicator film with in-plane anisotropy) and transport measurements were used to characterize the flux behavior in both 19 and 85 multifilamentary Ag/BSCCO-2223 tapes with very high Jc values of 54-65 kA/cm2 measured at 77K and self-field. It was found by magneto-optical imaging that magnetic flux tends to enter the tapes first perpendicular to the rolling direction. The patterns of flux penetration have a specific periodic structure which depends on residual defects arising after thermomechanical treatment, which is required to densify the BSCCO filaments after partial reaction from the 2212 to the 2223 phase. The characteristic lengths of the uncracked filaments vary from 0.2- 1.5 mm for 85 filamentary tapes to the more uniform 0.25-0.35 mm for the 19 filamentary tapes. These mechanical defects are large in scale and cause significant distortion of the current trajectories. To better understand the nature of these barriers to current flow individual filaments were extracted from BSCCO tapes and were examined by magneto-optical, transport and ultrasonic experiments. The result revealed that the Jc of individual filaments ranges from 30 kA/cm2 to at least 80 kA/cm2, a variation of more than a factor of two. This means that even for the highest Jc tapes there is still a lot of room to improve current carrying capability by eliminating the barriers to current flow.

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FABRICATION OF Ag-CLAD BI-2223 TAPES FROM COPRECIPITATED PRECURSOR POWDER: *U. Balachandran*[1]; M. Lelovic[1]; V. Selvamanickam[2]; P. Haldar[2]; [1]Argonne National Laboratory, Energy Technology Division, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Intermagnetics General Corporation, 450 Old Niskayuna Rd., P.O. Box 461, Latham, NY 12110 USA

Ag-clad Bi-2223 tapes were fabricated with precursor powders coprecipitated from a solution containing a mixture of cations. Theprecursor powder was preconsolidated in the form of a rod and inserted into an Ag tube to prepare the billet. Drawing and rolling conditions were optimized to fabricate tapes with a uniform Ag/superconductor interface. The critical current (I_c) in these tapes was more than 30% higher than that in tapes made from billets prepared by packing loose powders. Heat-treatment conditions were optimized, and processing time was reduced by 25% from that required for tapes prepared with powders made by the conventional solid-state technique. I_c values of >40 A at 77K in self-field have been obtained in long-length tapes heat treated for <100 h. The effects of powder particle size, mechanical processing, and heat-treatment conditions in enhancing the I_c of Ag-clad Bi-2223 tapes will be discussed.

HIGH TEMPERATURE COATINGS III: Coatings for Steels

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 Applic., Tullahoma, TN 37388 USA

Tuesday PM Room: 19
March 2, 1999 Location: Convention Center

Session Chairs: Narendra B. Dahotre, University of Tennessee Space Institute, Dept. of Mats. Sci. and Eng., Tullahoma, TN 37388 USA; Alejandro Sanz, Danieli Research and Development, Dept. of Mats. Develop., Buttrio 33042 Italy

2:00 PM INVITED PAPER

MICROSTRUCTURAL ANALYSIS AND PERFORMANCE EVALUATION FOR LASER CLAD STAINLESS STEEL ON PLAIN CARBON STEEL SUBSTRATES: W. Guo[1]; *A. Kar*[1]; [1]University of Central Florida, CREOLL, P.O. Box 162700, 4000 Central Florida Blvd., Orlando, FL 32816-2700 USA

Laser materials processing can produce finer and newer microstructures than conventional methods due to its inherent rapid solidification and is applied widely in many industrial applications. Laser cladding provides a way to make high-performance coatings to upgrade the properties of the substrate such as oxidation and wear resistance. The application of stainless steel coatings on carbon steel is useful for solving nuclear power plant erosion-corrosion problems that affect the feedwater, condensate and steam system piping components. Stainless steel powders 304L and 316L were deposited onto a plain carbon steel substrate to form a layer with a high corrosion resistance. A diffusion model for rapid solidification is used to predict the type of microstructures. The theoretical calculation is in good agreement with the experimental observation of microstructures. The microstructures of the interface show a complete fusion between the cladding material and the substrate. The cladding defects are minimized by controlling the processing parameters. Oxidation resistance and hardness tests were conducted to compare the properties of the clad parts with those of the base metal. The results show that laser cladding of stainless steel can improve the corrosion resistance and hardness of the surface.

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EVALUATION OF A DUPLEX STAINLESS STEEL COATING DEPOSITED ON A CARBON STEEL BY THE TIG WELDING PROCESS: C. R. Xavier[1]; A. L. B. Baptista[1]; L. C. A. Vieira[1]; E. L. Luiz[1]; *P. R. F. Ribas*[1]; [1]Escola de Engenharia Industrial Metalzrgica de Volta Redonda, UFF, Av. dos Trabalhadores, 420, Volta Redonda, RJ 27260-740 Brazil

The application of special alloys on the surface of steel components is largely used in order to confer particular properties to the steel sur-

face. Such properties are usually required when the surfaces are subjected to critical working conditions. In addition, the adequate application of an alloy with the required properties by welding may lead to a component extra life thus reducing significantly the maintenance costs. In the present work, a medium carbon steel was coated with a duplex stainless steel applied by the tungsten inert gas (TIG) welding process. The mechanical properties in the deposited material were assessed after cold working. Metallographic analysis revealed a mixed austenite and ?-ferrite microstructure in the deposited layer, absence of microcracks, as well as a reduced thermally-affected region. Such characteristics give a good indication that the deposition of this special alloy by the TIG welding process leads to a high wear resistance, good mechanical properties, as well as high ductility and tenacity.

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ELEVATED TEMPERATURE OXIDATION PROTECTION OF CARBON STEELS BY COMBUSTION CHEMICAL VAPOR DEPOSITION: *M. R. Hendrick*[1]; S. Shanmugham[1]; H. Shao[1]; A. T. Hunt[1]; [1]Microcoating Technologies, 3901 Green Industrial Way, Chamblee, GA 30341 USA

Carbon steels make up a substantial portion of the steel produced in the United States. Their uses are numerous, from aircraft parts to automobile components to nuts and bolts. Generally, carbon steel is limited to a use temperature less than 500ŸC, after which considerable oxidation weight gain results. This paper introduces the application of protective coatings by a unique, flame-based thin film deposition process called combustion chemical vapor deposition (CCVD) to increase the use temperature of AISI 1010 and 1095 steels while also providing wet corrosion resistance. The innovative CCVD process is an open atmosphere technique that does not require a vacuum or reaction chamber. High quality films are deposited comparable to those produced by traditional CVD. Steel coupons used in these experiments were maintained at low enough temperatures during the application of oxide thin films so as to prohibit any substrate oxide formation. Tests included weight gain measurements, wet corrosion tests and adhesion analyses. Results are discussed.

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CHROMIUM NITRIDE COATINGS WITH PULSED DC MAGNETON SPUTTER DEPOSITION: *Ray Y. Lin*[1]; Jin Seok[1]; [1]University of Cincinnati, Dept. of Mats. Sci. and Eng., M.L. #12, Cincinnati, OH 45221-0012 USA

Coatings of Cr and chromium nitride on steels have been investigated with RF and pulsed DC magnetron sputter deposition at various temperatures with powers in the range of 2.19 and 8.77 W/cm2. Ultra pure argon (99.99%) with nitrogen was used as the sputtering gas. X-ray diffraction analysis and microhardness measurements have been used to characterize the coatings. It was observed that increasing the sputtering power increased the coating hardness. However, due to the residual compressive stress, hillock formation was observed for coatings at high power deposition. X-ray diffraction analysis and TEM (Transmission Electron Microscope) analysis indicated that all as-deposited films were microcrystalline in nature and exhibited a BCC (body centered cubic) Cr phase for nitrogen content in the sputtering gas below 5%. For higher nitrogen contents, Cr2N and CrN phases were both detected.

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HIGH TEMPERATURE COATING SYSTEMS FOR THE STEEL INDUSTRY: *Alejandro Sanze*[1]; [1]Danieli & Company SpA., Centro Research and Development, Via Nazionale 41, Buttrio 33042 Italy

There is a large number of steel making processes in which great demands are made on the surface behavior of several components that come to direct contact with steel under various conditions. The surface quality is measured in two different ways: a) the influence of the surface properties on the quality of the products being manufactured or b) the weight of the component replacement cost in the unit economical equation and on the plant productivity. materials in contact with solidifying Continuous Casting Machine (CCM) inner mold coatings have firmly established themselves as a means for improving the product quality and enhancing a longer mold life. New chrome-free protective coating systems are introduced at the mold level for granting environmental compliance and with improved properties. Wear represents, directly or in relation with other damaging mechanisms, the most frequent cause of machine damage. Rollers are particularly subjected to various types of aggressive operating conditions. It is necessary for rolls to resist to high thermal and mechanical stresses, wear, thermal fatigue cracking and chemical aggressions among others. Under high-load rolling contact, the operating life limiting factor of a coating may be the surface components in contact with steel products, coating may provide a solution against wear, melting damage, corrosion and fatigue. Coating selection must be carefully designed for required function. An overview on several high temperature coating applications in the steel mills will illustrate the service life and productivity enhancement of coated components. Coated elements last longer and allow a more flexible and more accurate maintenance planning. New advanced steel production methods and more rigorous operating conditions are opening the possibilities for new coating techniques and new coating Mats..

3:45 PM BREAK

4:00 PM INVITED PAPER

FORMATION OF HIGH CHROMIUM SURFACE ALLOYS ON 21/4Cr-1Mo AND 9Cr-1Mo STEELS USING A SINGLE STEP LASER TREATMENT FOR IMPROVING THEIR HIGH TEMPERATURE OXIDATION AND CORROSION RESISTANCE: *R. Streiff*[1]; A. S. Khanna[2]; K. Wissenbach[3]; [1]Universite de Provence, Laboratoire de Physico-Chimie des Materiaux-EA 838, Equipe de Chimie du Solide-CASE 26, Centtre Sainte-Charles-3, Place Victor Hugo, Marseille, Cedex 1331 France; [2]Indian Institute of Technology-Bombay, Corrosion Sci. and Eng., Powai, Mumbai 400 076 India; [3]Institute for Lasertechnik, Aachen Germany

Laser surface alloying is becoming an important tool in modifying the surface compositions of various relatively reactive substrates for enhancing their oxidation and corrosion resistance. In the present work, heat exchanger Mats., namely 21/4Cr-1Mo and 9Cr-1Mo Steels were modified by changing their surface chemical composition by increasing the chromium and nickel concentration. This was done by a single step laser surface alloying. A 3 kW CO_2 laser in continuous mode was used for this purpose. A 50%Ni-50%Cr powder with particle size of 22-45 μm was fed simultaneously with a feed rate of 2 gm/min, using a line focus mode with a spot size of 2 mm. Various samples were obtained by varying the scan speed from 500 mm/min to 1000 mm/min. After laser treatment the samples were characterized for surface morphology, surface composition and cross section of laser melted zone. Oxidation tests are being conducted in air at 800ŸC and aqueous corrosion tests in 1N H_2SO_4 solution and 3.5% NaCl solution. One of the aims of the work is to compare the results on the surface composition, microstructure, corrosion and oxidation with alloys of similar composition, made earlier using two step laser surface alloying.

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USE OF THE SPOTFACE TECHNIQUE TO REDUCE ASSESSING TIME OF THE PAINTED GALVANIZED STEEL UNDERFILM CORROSION: J. C. G. Carneiro[1]; A. N. C. Costa[1]; *P. R. F. Ribas*[1]; [1]Escola de Engenharia Industrial Metazrgica de Volta Redonda, UFF, Av. dos Trabalhadores, 420, Volta Redonda 27260-740 Brazil

The increasing interest of the steel industry on materials with higher corrosion resistance has led to a continuous development of the corrosion process evaluation techniques. One major goal of the new methods is to reduce the analysis time. A common technique consists in scribing the painted galvanized steel surface in order to expose steel to the corrosion environment but still demands usually long times for measurement. In this work, a new analysis technique for assessing the underfilm corrosion propagation is used with the major benefit of reducing the time of analysis, without altering the basic corrosion mechanisms. In this method, industrially-painted galvanized steel sheets have the painting and coating removed from a 13-mm diameter spot in order to increase the cathodic/anodic area ratio. This increase leads to a lower sacrificial layer protection power thus reducing significantly the total time of the corrosion test as compared to the time consumed during the scribe method test.

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CHARACTERIZATION AND TRIBOLOGICAL BEHAVIOR OF BORIDE COATING ON STEEL DEPOSITED BY LASER: Arvind Agarwal[1]; *Narendra B. Dahotre*[1]; [1]University of Tennessee Space Institute, Dept. of Mats. Sci. and Eng., Center for Laser Applications, B. H. Goethert Parkway, Tullahoma, TN 37388 USA

Titanium diboride coatings are deposited on AISI 1010 steel using a Nd:YAG laser. An ultrahard "composite" coating is produced on the steel surface comprising of titanium diboride particles and iron. The coating is adherent and metallurgy sound in nature. Microstructural characterization is performed using SEM, EDX, XRD and TEM. As a consequence of non-equilibrium synthesis by laser, formation of a novel metastable phase(s) is also indicated. Tribological characterization of the boride coatings is performed using a block-on-disc test. In addition to microhardness measurements, nanoindentation tests are also carried out for mechanical characterization of the boride coating. The work is partially supported under the subcontract from United States Air Force through Mats. Modification, Inc.

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ROLE OF RARE EARTH OXIDE COATINGS IN HIGH TEMPERATURE OXIDATION: *S. Seal*[1]; [1]University of Central Florida, AMPAC and MMAE, Eng. 381, P.O. Box 162450, 4000 University Blvd., Orlando, FL 32816 USA

High temperature material degradation or protection of Fe-Cr alloys and steels are often related to the nature of their oxide scale formation. Breakdown of passive films leads to localized corrosion. Many a times, various alloying elements are incorporated in these alloys to prevent high temperature degradation. Addition of alloying elements are cumbersome and not always cost effective. In this paper, we investigate the role of rare earth oxide coatings on high temperature corrosion prevention of both low and high Cr steel. An in-situ high temperature oxidation set up has been built to study the oxidation kinetics of both coated and uncoated alloys under ambient pressure and dry air. The oxide films are characterized by SEM, XPS, AES to understand the scale morphology, chemistry and structure. This work relates some of these data to explain the linear, para-linear and parabolic growth kinetics observed in both low and high Cr steels.

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A NEW DIFFUSION COATING RESISTIVE TO HOT SULPHURIC ACID: *Karol Jozwiak*[1]; Andrzej Mlynarczak[2]; Piotr Grzesiak[3]; Jan Jakubowski[2]; Teresa Gapinska[3]; Gerard Mesmacque[1]; [1]Universite de Lille 1, IUT A, Rue de la Recherche, Villeneuve d'Ascq, Nord Pas de Calais 59653 France; [2]Politechnika Poznanska, Instytut Inzynierii Materialowej, Pl.M.Sklodowskiej-Curie 5, Poznan, 60-965 Poland; [3]Instytut Chemii Nieorganicznej, ul. Miczurina, Poznan, 61-653 Poland

Different special steels used in sulphuric acid plants, mainly for acid coolers have been reviewed with special attention to their corrosion-resistance at elevated temperatures. Our investigations were oriented towards formation of a coating with better corrosion-resistance to hot (150-300°C) concentrated sulphuric acid. Two methods were employed: PVD by plasma jet and CVD using a mixture of powders. The coatings in the systems: C-Cr; Si-Cr; Si; B; Ni-B; Si-B; Ni-Cr were obtained on plain constructional steels with carbon content in the range of 0.1-0.5 wt%. The coatings have been characterized by optical microscopy, microhardness, SEM and EPMA and X-ray diffractometry. The corrosion tests have been performed on a laboratory scale in the temperature range of 150-250°C during 500-2000 h. The coatings are composed of intermetallic compounds and solid solutions. The outer layer is always porous so it can be concluded that the inner solid solution layer is responsible for corrosion-resistance. A coating of the type Si-B formed by CVD powder method revealed the best corrosion-resistance with corrosion rate of 0.1 mm/year compared to the Sandvik SX steel 0.6 mm/y and a type 18-9 stainless steel 1.5 mm/y.

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

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

Tuesday PM — Room: 14A
March 2, 1999 — Location: Convention Center

Session Chairs: Gary J. Shiflet, University of Virginia, Dept. of Mats. Sci., Charlottesville, VA 22901 USA; M. Enomoto, Ibaraki University, Dept. of Mats. Sci., Hitachi, Japan 316-8511

2:00 PM INVITED PAPER

DIGM IN BICRYSTALS AND POLYCRYSTALS: *Alexander H. King*[1]; [1]State University of New York, Dept. of Mats. Sci. and Eng., Nicolls Rd., Stony Brook, NY 11794-2275 USA

Diffusion-induced grain boundary migration (DIGM) has been studied widely and a broad range of experimental tests have confirmed Hillert's theory that it originates from the coherency-strain effect. However, a number of complications still exist. In particular, there are some rather interesting differences between DIGM in polycrystals and bicrystals, including the rather puzzling observation that DIGM is almost always uni-directional in bicrystal experiments, but frequently bi-directional in polycrystals. In this talk, we consider what significant differences exist between the two cases, and we focus especially upon the effects caused by triple junctions, to see if any explanations for the observed differences can be suggested. Acknowledgment: this work is supported by the NSF, grant number DMR9530314.

2:40 PM INVITED PAPER

DRIVING FORCE FOR DISCONTINUOUS PRECIPITATION AND CHEMICALLY INDUCED INTERFACE MIGRATION: *Duk Yong Yoon*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. and Eng., 373-1 Kusungdong, Yusong-Gu, Taejon 305-701 Republic of Korea

In discontinuous precipitation and dissolution (of precipitates), solute atoms diffuse along the moving grain boundaries to and from the precipitates of usually lamellar shape. The regions either enriched with or depleted of the solute atoms are formed behind the advancing grain boundaries. Liquation can also occur discontinuously. In chemically induced interface migration, the grain boundaries and intergranular liquid films migrate, forming behind them regions either enriched with or depleted of the solute atoms which diffuse along the moving grain boundaries or liquid films either from the source or to the sink. Hillert formulated theories for the driving force of these processes based on coherency strain energy in the solute diffusion zone ahead of the moving interfaces. Critical experiments have been performed by systematically varying the coherency strain in ternary alloys and the results confirm the validity of Hillert's theory. The effects of external stress on discontinuous precipitation and of the interface curvature on the boundary migration are also consistent with the coherency strain theory. The migration direction is also determined by the asymmetry of the coherency strain energy across the boundary. The grain boundary faceting can cause zigzag boundary migration and also influence the shape of the slowly moving boundaries.

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INTERFACE MIGRATION IN NATURAL AND ARTIFICIAL LAYERED STRUCTURES: A PARALLEL BETWEEN DISCONTINUOUS AND INTERFACE MEDIATED INSTABILITIES IN MULTILAYERS: *Yves Brechet*[1]; Leonid Kinger[1]; Gary Purdy[1]; [1]ENSEEG/INPG-Domaine Universitaire, Laboratoire de Thermodynamique et Physico-Chemie Metallurgiques, 1130 Rue de la Piscine, Saint Martin D'Heres BP 75, 38402 France

Interface migration coupled with a diffusion process at the interface occurs in many situations in physical metallurgy: eutectoid reactions, discontinuous precipitation, liquid film migration, DIGM. In this class of problems, the contribution of Mats Hillert has become classic. The present contribution will focus on discontinuous precipitation as an example of a natural layered structure, and on multilayer instability as an example of an artificial one. For both examples, we will investigate the central questions open for modelling: the macroscopic kinetics of the phenomenon, and the morphological instabilities associated with interface migration. The difference between interphases and grain boundaries will be shown to have important consequences on the pattern selection process. For discontinuous precipitation we will revisit the question of spacing selection. For multilayer instability, we will investigate the continuous front and the fingering solutions for both full miscibility of the elements (discontinuous homogeneisation) and reactive systems (discontinuous peritectic reaction).

4:10 PM INVITED PAPER

MASSIVE TRANSFORMATION GROWTH KINETICS: *J. H. Perepezko*[1]; G. Purdy[2]; [1]University of Wisconsin-Madison, Dept. of Mat. Sci. & Eng, 1509 University Ave., Madison, WI 53706 USA; [2]McMaster University, Dept. of Mat. Sci. & Eng., 1280 Main Str. W., Hamilton, Ontario L8S 4L7 Canada

During the massive transformation a parent phase crystal structure is converted into different single or dual phase product structures without any change in composition. The reaction interface is driven by the free energy change due to the crystal structure change and controlled by thermally activated boundary diffusion. Once the rate limiting nucleation step is accomplished, the high driving free energy for massive growth is reflected by a rapid transformation. Interface migration rates can exceed 1 cm/sec and can yield a departure from local interfacial equilibrium due to solute trapping. For typical massive reactions involving bcc, fcc and hcp structures, the growth kinetics can be modeled by a continuous growth mechanism, but other effects involving interface conditions and step mediated growth may be important at low driving free energies. Under the limiting growth conditions at the transition into lattice shear transformations or solute partitioning, the reaction yields product structures of metastable supersaturated solid solutions.

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Structure-Property Relationship and Reliability I

Sponsored by: Electronic, Magnetic & Photonic Materials Division,
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

Tuesday PM — Room: 17A
March 2, 1999 — Location: Convention Center

Session Chairs: M. McCormack, Fujitsu Computer Packaging Technologies; Z. Mei, Hewlett Packard Company

2:00 PM INVITED PAPER

MICROSTRUCTURAL EVOLUTION AND RELIABILITY IN FINE-PITCH SOLDER JOINTS: *J. W. Morris*[1]; C. Gonzales[1]; M. Barney[1]; [1]University of California at Berkeley, Dept. of Mats. Sci. and Eng., Center for Advanced Mats., Lawrence Berkeley Laboratory, Berkeley, CA 94720 USA

An important active trend in microelectronics packaging is the trend toward increasingly small joint sizes. As size decreases, two potential problem intrude: unusual mechanical behavior due to a relatively coarse microstructure, and bond-line brittleness due to fine voids at the solder-substrate interface. The microstructure ordinarily very fine in the initial state because of the rapid solidification rate, but coarsens during service, changing the mechanical behavior. The void structure is also very fine in the as-solidified condition, causing brittleness at the interface. Voids coarsen during subsequent processing and service, which actually improves mechanical behavior. For both reasons, the evolution of the microstructure must be taken into account to understand and control the behavior of solder joints.

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STRUCTURE-PROPERTY RELATIONSHIP FOR SOLDER INTERFACES: *Jian Ku Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., College Of Eng., 1304 West Green St., Urbana, IL 61801 USA

Bonding between solder alloys and metallizations is often achieved by developing metallurgy reactions at the interface. Consequently, the reliability of a solder bond strongly depends on the microstructure of the solder interface. In this presentation, recent work on microstructures and fatigue resistance of solder interfaces will be reviewed. Relationship between interfacial microstructure and property will be examined on solder interfaces prepared by systematically controlling processing conditions and by additions of select alloying elements. Fatigue resistance of the newly designed interfaces was found to depend on the type of the phase(s) present at the interface as well as the morphology of the interfacial phase. Theoretical models are presented to explain the dependence of the interfacial crack resistance on interfacial microstructure.

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EFFECT OF THERMAL CYCLES ON THE MECHANICAL STRENGTH OF QUAD FLAT PACK LEADS/Sn-3.5Ag-X(X=Bi, Cu) SOLDER JOINTS: *Yoshiharu Kariya*[1]; Yasunori Hirata[1]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Mats. Sci. and Eng., Shibaura 3-9, Tokyo, Minato-ku 108-8548 Japan

Tin-silver eutectic is an attractive candidate alloy to meet requirement for demanding high temperature service environment such as automotive under-hood. The alloy has, however, a melting point con-

siderably higher than that for Sn-Pb eutectic. The melting temperature of a new Sn-Ag based solder alloy could be brought close to Sn-Pb eutectic temperature by adding a third element. It is, however, not clear to what extent the additional elements affect on mechanical reliability (i.e. fatigue properties) of eutectic Sn-Ag solder alloy. In our previous study, we clarified the effect of such third element as bismuth, copper and indium on the fatigue life of bulk Sn-3.5%Ag binary alloy. In this study, Quad Flat Pack (QFP) Leads/Sn-3.5Ag-X(X=Bi and Cu) joint were thermally cycled between 243K and 403K with a ramp rate of 1.78K/min to evaluate the effect of third element on thermal fatigue damage. Both metallographic examination and mechanical pull test were performed to evaluate thermal fatigue damage of that joint. The pull strength of QFP/Sn-3.5Ag-Bi solder joints was drastically degraded due to thermal cycles. On the other hand, the pull strength of QFP/Sn-3.5Ag-Cu solder joints slightly decreased with increasing number of cycles, though its magnitude still remains higher in comparison to bismuth containing solder joint. The behavior observed here is similar to that in bulk solder material or solder/copper joint specimen.

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DAMAGE EVOLUTION GOVERNED BY MICROCRACK NUCLEATION WITH APPLICATION TO THE FATIGUE OF 63Sn-37Pb SOLDER: *V. Stolkarts*[1]; L. M. Keer[1]; M. E. Fine[2]; B. Moran[1]; [1]Northwestern University, Dept. of Civil Eng., 2145 Sheridan Rd., Evanston, IL 60208-3109 USA; [2]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

This paper uses methods of statistical physics, micromechanics and applied mathematics to model damage governed by nucleation of microcracks such as often occurs in eutectic tin-lead solder. This approach derives from the understanding that the nature of such damage is stochastic due to randomness of both defect geometry and material microstructure. When the damage evolution is dominated by the nucleation of new microcracks rather than expansion of the existing ones, application of percolation theory to an array of cracks gives an estimation of the percolation threshold which is related to the microcrack density. This leads to a microstructural based model for a failure criterion and for failure prediction. The model also takes into account size effect. The model has application to cases such as Sn-Pb eutectic solder where microcracks originate at interfaces between phases and between grains and remain microcracks because of microstructural obstacles to growth.

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INFLUENCE OF REFLOW PROCEDURE ON THE MICROSTRUCTURE AND STRENGTH OF 62Sn36Pb2Ag SOLDER JOINTS: Y. Fahmy[1]; Di Yang[1]; *H. Conrad*[1]; [1]North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695-7907 USA

The influence of burn-off time at 100°C and reflow duration at 195°C on the microstructure and strength in shear of 62Sn36Pb2Ag/Cu solder joints prepared from solder paste were determined. Regarding burn-off time, it was found that the amount of porosity in a joint and its variation from specimen-to-specimen decreased with increase in burn-off time from 0 to 5 min. Associated therewith was a decrease in the scatter and an increase in the magnitude of the maximum load in the shear test. The influence of the porosity on the mechanical response was mainly to reduce the load-bearing cross sectional area. Increase in the reflow time from 1 to 37 min gave a 20% decrease in the true maximum stress and an increase in scatter from specimen-to-specimen. EDS analysis of cross sections of the solder joints revealed a significant difference in the chemical composition from the bottom to the top elevation during reflow, Sn, Pb and Ag being higher at the top compared to bottom. Furthermore, Sn and Pb were higher in the middle compared to either the top or bottom, while the reverse was the case for Ag. The compositional differences were relatively uninfluenced by the reflow duration time. The differences in composition between the top and bottom are attributed to convection.

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EFFECTS OF THICKNESS OF Ni LAYER OVER Cu PLATE ON INTERFACIAL REACTION AND RELIABILITY: *Won Kyoung Choi*[1]; Hyuck Mo Lee[1]; [1]Korea Advanced Institute of Sci. and Technology, Dept. of Mats. Sci. and Eng., Kusong-Dong 373-1, Yusong-Gu, Taejon, Choongchungnam-Do 305-701 Korea

Sn3.5Ag and Sn3.5Ag8.5In were soldered on the various substrates of bare Cu, Ni(2§≤)/Cu, Ni(4§≤)/Cu, and bare Ni plates. The morphology and composition of the intermetallic compounds at the interface were examined using SEM and XRD. With varying the Ni thickness, various intermetallic compounds were formed, those were Cu_6Sn_5 on bare Cu, $NiSn_3$ on Ni(2§ ≤)/Cu, Ni_3Sn_2 on Ni(4§≤)/Cu and Ni_3Sn on bare Ni, and wetting behavior was different. Such phenomena seemed to be caused by the type of intermetallic compounds differently formed on each substrate. Therefore the thickness of the Ni layer which reacted with solder directly have to be considered in the prediction of interfacial reaction. After annealing for 400 hours, the growth behavior and mechanical test result of each intermetallic compound were different. It was found that the intermetallic compounds formed differently with varying the Ni layer thickness have an effect on the solder reliability.

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CREEP DEFORMATION BEHAVIOR IN Sn-3.5Ag SOLDER JOINTS USING A NOVEL MAPPING TECHNIQUE: *James P. Lucas*[1]; Alan Gibson[1]; Tom Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Mats. Sci. and Mech., A304 Eng. Bldg., East Lansing, MI 48824 USA

Bulk solder and solder joints readily deform by creeping under the influence of both static, cyclic, and thermo-mechanical loading conditions. Deformation under creep conditions is of particular interest and importance since solder Mats., in general, function at rather high homologous temperatures. Expressed in degrees absolute, even room temperature corresponds to a temperature that is 61% of the melting temperature of Sn-3.5Ag solder. Therefore, creep conditions will always persist. To investigate creep deformation and creep mechanism of thin solder joints and bulk solder Mats., a novel mapping of deformation test technique has been developed. This technique enables quantitative measurement and qualitative assessment of micro-scale deformation in small-volume, 100-micron-thick solder joints. Deformation behavior can be assessed in highly localized regions spanning the entire solder joint. Creep deformation data at the substrate/solder interface is obtainable by the proposed technique. In this investigation, creep deformation behavior is presented for both composite (Sn-3.5Ag with in situ Cu_6Sn_5 particles) and non-composite (Sn-3.5Ag) bulk solder and solder joints. Lifetime prediction for solder joints is possible using this novel technique.

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ANALYSIS OF A CATASTROPHIC FIELD FAILURE DUE TO CONDUCTIVE ANODIC FILAMENT (CAF) FORMATION: *W. Jud Ready*[1]; B. A. Smith[1]; L. J. Turbini[1]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

Conductive anodic filament (CAF) formation was first reported in 1976. This electrochemical failure mode of printed wiring boards (PWB) involves the growth of a copper-containing filament substrate along the epoxy-glass interface within the PWB. The filament is observed to grow from anode to cathode, and may form an electrical short within the PWB over time. CAF formation was ascribed to moisture up-take by the PWB substrate followed by an electrochemical reaction that created mobile copper ions. Despite the proposed reduced lifetime in electronic substrates due to CAF, field failures were not identified prior to the 1990s. The failure phenomena known as CAF poses serious long-term reliability concerns in PWBs exposed to adverse and hostile environments, especially those with closely spaced conductors. In this work, a catastrophic field failure due to CAF was analyzed. The failure occurred in a high T_g PWB used in a mixed technology product. The CAF failure occurred on an inner layer of a multi-layer board (MLB) between a via and ground plane where a potential difference of 320 V existed with 0.015 nominal spacing. The nature of the CAF was analyzed using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). Ion chromatography (IC) and high performance liquid chromatography (HPLC) were used to identify residue extracted from the failed boards and relate those to the hot air sodler leveling (HASL)

fluid used. Recommendations are made to aid in the prevention of field failures due to CAF formation.

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Deformation Twinning

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

Tuesday PM Room: 17B
March 2, 1999 Location: Convention Center

Session Chairs: S. Mahajan, Arizona State University, Dept. of Chem., Tempe, AZ 85287-6006 USA; Aristos Christou, University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742-2115 USA

2:00 PM INVITED PAPER

FORMATION OF DEFORMATION TWINS IN METALLIC CRYSTALS: *S. Mahajan*[1]; [1]Arizona State University, Dept. of Chem., Bio and Mats. Eng., P.O. Box 876006, Tempe, AZ 85287-6006 USA

A consensus has emerged that dislocations are involved in the formation of deformation twins in metallic crystals. Several models have been proposed to rationalize the role of dislocations, and they can be broadly classified into two groups: (i) "pole mechanisms" and "ratchet" or "cross-slip" sources and (ii) conversion of slip bands into microtwins. Experimental evidence on the early stages of twinning in BCC and FCC crystals will be presented. We will argue that these observations are difficult to explain in terms of the pole models and their derivatives, but are consistent with the slip band conversion hypothesis. In addition, the influence of various metallurgy variables on twinning can be accounted for using the latter model.

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DEFORMATION TWINNING: FROM ATOMIC MODELING TO SHOCK WAVE LOADING: *Ronald W. Armstrong*[1]; Frank J. Zerilli[2]; [1]University of Maryland, Mech. Eng., Bldg. 088, Rm. 2180, Eng. Classroom Bldg., College Park, MD 20742-5035 USA; [2]Naval Surface Warfare Center, Indian Head Division, 101 Strauss Ave., Indian Head, MD 20640-5035 USA

Connection is made between deformation twinning results obtained over a wide range of testing conditions on magnesium, 1010 steel, titanium, silicon-iron and Armco iron materials. In low temperature and/or dynamic tests, micro-slip pile-up stress concentrations provide a strong, nearly athermal, Hall-Petch (H-P) stress versus inverse square root of grain diameter dependence. The high H-P microstructural stress intensity (slope value) accounts for the weak thermal dependence of the twinning stress. Twinning is followed in compression by general viscoplastic yielding by slip in the "new" twin-hardened material. In computations of cylinder impact (Taylor) test results, the hardening is attributed to effective grain size refinement that also includes a strain-accommodation-type grain volume strengthening component.

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DEFORMATION TWINNING AND SHOCK STRENGTHENING IN Cu AND Cu-Al ALLOYS: *Aashish Rohatgi*[1]; Kenneth S. Vecchio[1]; [1]University of California, San Diego, Mats. Sci. Group, Dept. of AMES, 9500 Gilman Dr., La Jolla, CA 92093 USA

FCC materials have been known to undergo deformation twinning under shock loading conditions. The propensity of deformation twinning varies inversely with the stacking fault energy of the material and varies directly with the shock pressure. The deformation twins are formed within the grains and pre-existing annealing twins and contribute to the post-shock strength of the material. The strength contribution from the deformation twins can be attributed to the apparent reduction of the grain size with the deformation twin boundaries acting as "grain boundaries" restricting the dislocation motion. In the present work, pure Cu and a series of Cu-Al alloys were shock deformed at 10 and 35 GPa. The shock-deformed materials were characterized by optical metallography, TEM, ultrasonic attenuation, microhardness and quasi-static compression tests. Each of the individual strengthening mechanisms (such as dislocations, solid solution alloying, grain boundaries, etc.) present in the materials were modeled and quantified, enabling the contribution due to deformation twin density to be evaluated.

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INFLUENCE OF STACKING FAULT ENERGY, GRAIN SIZE, AND STRESS STATE ON DEFORMATION TWINNING IN FCC POLYCRYSTALS: *Surya R. Kalidindi*[1]; [1]Drexel University, Dept. of Mats. Eng., 3141 Chesnut St., Philadelphia, PA 19104 USA

This paper investigates the microstructural variables influencing the stress required to produce deformation twins in polycrystalline face-centered cubic (FCC) metals. Classical studies on FCC single crystals have concluded that the deformation twinning stress has a parabolic dependence on the stacking fault energy of the metal. In the paper, new data is presented indicating that stacking fault energy has only an indirect effect on the twinning stress. The results indicate that the dislocation density and the homogeneous slip-length are the most relevant microstructural variables that influence directly the twinnning stress in the polycrystal. The role of the stacking fault energy was observed to be critical in building the necessary dislocation density while maintaining relatively large homogeneous slip-lengths. It was also observed that the stress state (e.g. simple compression, plane strain compression, simple shear) has an important influence on both the strain hardening rates and the microstructure evolution in the low SFE polycrystals. These results will be presented and discussed.

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TWINNING IN SINGLE CRYSTALS OF HADFIELD STEEL AND AUSTENITIC STAINLESS STEEL WITH NITROGEN: *Huseyin Sehitoglu*[1]; Yuriy I. Chumlyakov[2]; Ibrahim Karaman[1]; Ken Gall[1]; Irina V. Kireeva[2]; Elena I. Litvinova[2]; Elena G. Zaharova[2]; Natali V. Luzginova[2]; [1]University of Illinois, Mech. and Indust. Eng., 1206 W.Green St., Urbana, IL 61801 USA; [2]Siberian Physico-Technical Institute, Revolution Sq. 1, Tomsk 634050 Russia

The present study has considered the deformation of single crystals of austenitic stainless steel (Fe-18wt%Cr-16wt%Ni-10wt%Mn- and 0-0.5wt%N) and Hadfield steel (Fe-13wt%Mn-1.3wt%C). An investigation into the orientation dependence and asymmetry of the critical resolved shear stresses (CRSS), hardening modulus, and fracture behavior has been carried out. The combination of strong friction forces, due to solid solution hardening with nitrogen and carbon atoms, with a low stacking fault energy leads to the appearance of non-Schmid effects. In contrast to Schmids law, the single crystals of both materials demonstrate an orientation dependence and tension-compression asymmetry of the CRSS. The [111] crystals under tension and [001] crystals under compression are characterized as "soft". Their deformation is primarily controlled by deformation twinning. In "hard" orientations ([001] at tension and [111] at compression) deformation takes place by slip, and twinning is not observed. As the test temperature is decreased, non-Schmid effects become stronger due to the favoring of twinning at low temperatures. An activation of a few of twin systems (tension [111], compression [001]) leads to high values of the work hardening rate that is conditioned by twin-twin and twin-slip interactions. During twinning, the interstitial atomic positions transform from octahedral sites to tetrahedral sites after the displacement of the leading Shocky partial (a/6<211>). Consequently, the twins act as even stronger obstacles to dislocation motion compared to twins in a pure metal. The results of this investigation provide an experimental base for the current micromechanical models of twin-twin and twin-slip interactions. These quali-

tative understandings are necessary to derive constitutive models for the deformation of polycrystalline metals that deform by combined twinning and slip.

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DEFORMATION TWINNING OF SAPPHIRE (a-Al2O3): *K. Peter D. Lagerlöf*[1]; Anquin He[1]; Arthur H. Heuer[1]; [1]Case Western Reserve University, Dept. of Mats. Sci. and Eng., 109000 Euclid Ave., White Bldg., Cleveland, OH 44106-7204 USA

Deformation twinning is an important mode of deformation of sapphire (alpha-Al2O3) below its brittle to ductile transition temperature. Two twinning systems have been identified in sapphire; basal twinning (K1=(0001), eta1=<10-10), K2={10-11}, eta2=<-1012>, s=0.635) and rhombohedral twinning (K1={0-112}, eta1=<0-11>, K2={0-114}, eta2=<02-21>, s=0.202), respectively. A new simple model relating dislocation slip and deformation twinning in sapphire was recently proposed by the authors and collaborators. Newly obtained experimental evidence supporting this twinning model will be presented. In addition, a brief discussion of how the twinning model can be applied to other materials (metals, alloys and inorganic compounds) will be carried out.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Alloying/Processing

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

Tuesday PM Room: 8
March 2, 1999 Location: Convention Center

Session Chairs: Dennis M. Dimiduk, Air Force Research, Wright-Patterson AFB, WL/MD, WPAFB, OH 45433 USA; K. A. Stevens, Air Force Research Laboratory, Mats. & Manuf. Directorate, Wright-Patterson AFB, OH 45434-7817 USA

2:00 PM INVITED PAPER

ATOM-PROBE INVESTIGATIONS OF FINE-SCALE FEATURES IN TiAl ALLOYS: *Alain Menand*[1]; [1]University of Rouen, Groupe de Métallurgie Physique - UMR CNRS 6634, Faculte des Sciences, Mont Saint Aignan, Seine Maritime 76821 France

This paper reviews the APFIM results obtained on the distribution of interstitial elements, phase transformations and the solute partitioning of additional element in TiAl-based alloys. The solubility of oxygen and carbon in the gamma phase was determined by measuring interstitial concentrations in (alpha2+gamma) two-phase alloys as well as on gamma single phase TiAl alloys. The preferential location of interstitial elements (O, C, N) in the a2 phase and their very low solubilities in gamma are explained through the existence of interstitial vacancies in a titanium rich environment. Large but fine-scale compositional variations and metastable phases observed in the lamellar structure of non equilibrium state Ti52Al48 alloys are analyzed. The partition coefficients of Cr and Nb between alpha2 and gamma phases and the influence of these elements on the phase volume fraction have been studied and the results will be analyzed in terms of the possible shape of the solubility lobes in isothermal sections of ternary phase diagrams. Tomographic Atom Probe 3D images exhibiting chromium segregation both at alpha2/gamma and gamma /gamma interfaces will be presented for a GE type alloy heat treated at 1000ϒC.

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THEORETICAL AND EXPERIMENTAL STUDY OF THE SOLUBILITY OF OXYGEN IN GAMMA-TiAl AND RELATED PHASES: *Gilles Hug*[1]; Evelyne Fries[1]; [1]ONERA-CNRS, LEM, UMR 104, BP72, Chatillon 92322 France

Oxygen and other light elements (C, N) exhibit a very low solubility in the γ-TiAl phase whereas considerable amount of them can be solved in the α_2-Ti_3Al phase. From ab-initio total energy calculations we explain such difference in solubility by the chemical environment of the octahedral cavities in which oxygen sits. In short, oxygen is found to have the highest solubility in the octaedra with eight titanium atoms at summits which are present in α_2-Ti_3Al and do not exist in gamma-TiAl. This result is in contrast with the highest affinity of oxygen with aluminium than with titanium and will be discussed together with experimental results. It is also shown that oxygen can be stabilized in cavities with 3 titanium and 3 aluminium atoms in the H-phase. Alloys with different amount of oxygen have been prepared and studied by transmission electron microscopy and electron energy loss spectroscopy to assess the local atomic environement of oxygen.

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ISOTHERMAL AND CONTINUOUS COOLING DECOMPOSITION OF ALPHA AND BETA PHASES IN GAMMA TITANIUM ALUMINIDES: *Dennis M. Dimiduk*[1]; Vijay K. Vasudevan[2]; [1]Air Force Research Laboratory, Mats. & Manufact. Directorate, AFRL/MLLM Bldg. 655, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA; [2]University of Cincinnati, Dept. of Mats. Sci. & Eng., Cincinnati, OH 45221-0012 USA

The last decade led to significant engineering advances and component demonstrations for gamma-alloy products. The variety of alloy chemistries and process variations available for these products results in a spectrum of microstructures and property variation for the alloys. However, the quantitative aspects of solid-state reactions and microstructural evolution kinetics, as well as the influences of alloy composition upon these, are relatively unstudied. This presentation succinctly describes the isothermal and continuous-cooling transformations for gamma alloys across a range of compositions from binary and ternary bases, to complex multicomponent chemistries of commercial interest. Time-temperature-transformation curves are compared, and the effects of Al concentration and selected alloying additions are highlighted. Aspects of producing fine-grained fully-lamellar microstructures, having controlled lamellar characteristics, in wrought mill products are discussed. Both B2-phase forming elements (Cr, Mo & W) and boron in the alloys are examined as grain-size controlling agents. When such agents are used, the lamellar transformation kinetics may be significantly altered relative to other gamma alloys, thus changing the thermal process path and affecting the perfection of the lamellar microstructures. These lead to concomitant changes in alloy properties. The prospects for attaining such structures and properties in large product scales are discussed.

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ON THE RELATION BETWEEN COOLING RATE AND SOLIDIFICATION MICROSTRUCTURE IN AS-CAST TITANIUM ALUMINIDES: *Marc De Graef*[1]; Nicholas E. Biery[1]; Linda Rishel[1]; Tresa M. Pollock[1]; Alan Cramb[1]; [1]Carnegie Mellon, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

The texture of near-gamma titanium-aluminum alloys depends in a rather sensitive way on the details of the solidification process. It has been reported that for aluminum levels below about 48 at%, the alpha phase predominantly grows with a c-axis [00.1] primary dendrite direction. For higher aluminum levels, the basal plane [10.0] type directions are the primary growth directions. For a given aluminum level, faster cooling rates correspond to c-axis growth, and slow rates are consistent with [10.0] growth. The alpha phase solidification texture is retained during subsequent phase transformations, and has a significant influence on the mechanical properties and microstructure of the as-cast material. We will report on detailed orientation imaging microscopy experiments on binary alloys with varying aluminum level, and on 48-2-2 type alloys, all in the as-cast condition. The solidification texture will be

correlated to the aluminum level and the cooling rate. This research was funded by AFOSR grant # F49620-95-1-0359.

3:40 PM INVITED PAPER

PROCESSING OF GAMMA-TiAl BASED ALLOYS ON AN INDUSTRIAL SCALE: *Helmut Clemens*[1]; Nico Eberhardt[2]; Heinrich Kestler[3]; [1]Universität Stuttgart, Institut für Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany; [2]Montanuniversität Leoben, Institut für Metallkunde und Werkstoffprüfung, Franz-Josef-Strasse 18, Leoben, Styria A-8700 Austria; [3]Plansee AG, Technology Center, Planseestrasse, Reutte, Tyrol A-6600 Austria

This paper describes our present status in thermomechanical and near-net shape processing of gamma-TiAl based alloys on an industrial scale. The progress achieved in forging of large ingots, rolling of sheets from forged ingot as well as HIPed powders, single and multistep extrusion of ingots and HIPing of prealloyed powders to near-net shapes parts will be presented. The impact of the different prematerial routes on process economy and quality of semi-finished products will be discussed. The mechanical properties of sheets, extruded rods and HIPed powder compacts in as-processed condition and after subsequent heat-treatments will be compared. The problems which arise when heat-treatments are transferred from small lab furnaces to large industrial furnaces will be outlined. For further manufacture of semi-finished products to final TiAl components secondary processing steps are required. Examples for forming, maching and joining will be given. Finally, gamma-TiAl components based on sheet, rod or HIPed powder are shown.

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MICROSTRUCTURAL STUDIES ON DIRECT LASER FABRICATED TiAl: *Dinesh Srivastava*[1]; Issac T. H. Chang[1]; *Mike H. Loretto*[1]; [1]The University of Birmingham, IRC in Mats. for High Performance Applications, Edgbaston, Birmingham, West Midlands B15 2TT UK

Samples of Ti48Al2Mn2Nb alloy material have been atomised using the cold wall melting facility in the IRC and these powder has been used as feedstock for laser fabrication of samples. This laser treated material has been examined using optical, analytical scanning and transmission electron microscopy, both immediately after laser fabrication and after a range of heat treatments. The microstructural observations will be presented for a number of experimental conditions including powder sizes used, laser power, laser scan rate, powder feed rate and other processing parameters. The microstructure of laser treated material has been compared with the microstructures of conventionally processed material. The stability of the microstructure has been examined after different annealing treatment.

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PROCESSING OF GAMMA-TiAl BASED INGOTS AND THEIR CHARACTERIZATION: *Volker Güther*[1]; Andreas Otto[1]; Heinrich Kestler[2]; Nico Eberhardt[3]; Helmut Clemens[4]; [1]GfE Metalle und Materialien GmbH, R&D, Höfener Strasse 45, Nürnberg D-90431 Germany; [2]Plansee AG, Technology Center, Plansee Strasse, Reutte, Tyrol A-6600 Austria; [3]Montanuniversität, Institut für Metallkunde und Werkstoffprüfung, Franz-Josef-Strasse 18, Leoben, Styria A-8700 Austria; [4]Universität Stuttgart, Institut für Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany

As a consequence of the progress in processing of gamma-TiAl based alloys to semi-finished products the availability of industrial-scale ingots with a defined homogeneous element distribution is strongly required. This paper describes the processing of ingots by means of vacuum arc melting. Due to the use of optimized master alloys segregation effects can be prevented and, therefore, a deviation of the Al-content of only +/- 0.5 atomic percent is achieved over the entire ingot. This high homogeneous distribution also applies for other alloying elements, e.g. Cr, Nb, Ta, Mo, Si, B, etc. The reliability of the process is demonstrated for a Ti-47Al-4(Cr,Nb,Mo,B) alloy. The characterization comprises the local distribution of chemical composition and microstructural investigations by light optical- and scanning electron microscopy. From the obtained results it is expected that cost-intensive annealing treatments prior to subsequent processing can be omitted.

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LASER FORMING OF GAMMA TITANIUM ALUMINIDE: *John H. Moll*[1]; Eric J. Whitney[2]; C. Fred Yolton[1]; Ulrike Habel[1]; [1]Crucible Materials Corporation, Crucible Research, 6003 Campsbell Run Rd., Pittsburgh, PA 15205-1022 USA; [2]The Pennsylvania State University, Applied Research Laboratory, P.O. Box 30, State College, PA 16804-0030 USA

Laser forming is a process for producing a part directly from a three dimensional computer model. The attraction of the process is that it requires no hard tooling. As a result, there is the possibility for saving considerable time and cost in prototyping or manufacturing a metal article. In the process, a laser is used to melt input powder one layer at a time; the part is built up layer upon layer. The computer model is used to index the laser and/or the part. Development of the process for conventional titanium, such as Ti-6Al-4V, is well underway with at least one commercial facility in operation. The properties of laser formed conventional titanium alloys are equal to HIP castings. Application of laser processing to titanium aluminide should be beneficial in reducing segregation, refining the microstructure and reducing lead time for hardware. This paper will describe the results of initial studies to evaluate the feasibility of applying laser forming to the manufacture of gamma titanium aluminide hardware. The process will be described in detail and the results of microstructrural and mechanical property evaluations will be presented. Comparisons will be made with cast, ingot metallurgy and powder metallurgy processes.

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MICROSTRUCTURE, POROSITY AND MECHANICAL PROPERTIES OF SPRAY FORMED GAMMA TITANIUM ALUMINIDES: *Rainer Gerling*[1]; Kaiwen Liu[1]; Peter Schimansky[1]; [1]GKSS-Forschungszentrum, Max-Planck-Strasse, 21502 Geesthacht, Schleswig-Holstein Germany

Spray forming is a powder metallurgy technology which combines the powder production and compaction process. The ideal result of spray forming is a deposit with a fine microstructure, a high degree of chemical homogeneity and a low porosity, which can be used for further processing as extrusion, forging or rolling. Using the atomization technology EIGA (Electrode Induction Melting Gas Atomization) and a specially designed collector system, spray forming experiments have been conducted. For different - binary and advanced - gamma TiAl-alloys variations of process parameters as melt flow rate, atomization gas pressure and collector drive resulted in a number of deposits of cylindrical shape. For the different alloys the results from tensile tests at room temperature are correlated to the respective porosity levels and microstructures.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Poster Session II – 6:00 to 10:00 PM

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

Tuesday PM
March 2, 1999

Room: San Diego Ballroom A&B
Location: Marriott, North Tower

Session Chairs: Sun-Keun Hwang, Inha University, Inchon 160 Korea; Kevin J. Hemker, Dept. of Mechanical Engineering, Johns Hopkins University, 3400 N. Charles St., Baltimore, MD 21218-2686 USA; Ian J. Perrin, ALSTOM, Mech. Eng. Center, Whetstone, Leicester LE8 6LH UK

A DESCRIPTION OF THE DISLOCATION POPULATION BEHAVIOUR IN TITANIUM ALUMINIDES WITH AN ACCOUNT OF THE DISLOCATION SOURCES OPERATION: *Bella Aleksandrovna Greenberg*[1]; Michael Alekseevich Ivanov[2]; [1]Institute of Metal Physics, Ural Branch of Russian Academy of Sciences, 18 S.Kovalevskaya St., Ekaterinburg GSP-170, Sverdlovsk Region 620219 Russia; [2]Institute of Metal Physics, National Academy of Sciences, 36 Vernadsky Av., Kiev Region 252142 Ukraine

A new approach to the description of the plastic deformation processes has been developed. The dislocation ensemble is examined as a certain population, an evolution of which is defined by a multiplication of dislocations as well as their transformations, such processes occuring on the background of elastic stress fields created by the dislocation ensemble itself. The non-linear equation for the change of dislocation density with time due to the operation of sources is proposed. It is analogous to the well-known equation for the population growth, but it contains specific values, such as critical stress and characteristic time required to switch-on the dislocation sources. It is with this last value that is connected the possibility of a fast or slow mutual adjustment of the dislocation density and external stress. On this basis, an explanation was proposed for the non-monotonous stress-strain dependence observed in certain cases at the transition from elastic to plastic deformation. An analysis of the defor mation curves at various values of the systems parameters was performed with an account of the dislocation transformations in titanium aluminides. The peculiarities of plastic deformation for TiAl, Ti3Al and their lamellar structure alloys are described.

PROPERTY/STRUCTURE RELATIONSHIPS IN EXTRUDED HIGH STRENGTH TiAl-BASED ALLOYS: *D. N. Horspool*[1]; T. T. Cheng[1]; P. A. Blenkinsop[1]; M. H. Loretto[1]; [1]The University of Bimingham, IRC in Materials for high Performance Applications, Edgbaston B15 2TT UK

Current g-TiAl alloys are based on Ti-44/48 Al (at.%) with additions of a few percent of various elements. Recent work has shown that a higher percentage of alloying additions, particularly of Nb, Zr and Hf, produces alloys with superior properties. A dispersion of TiB2 particles has been shown to have a beneficial effect on the as-cast grain size of current alloys, and extrusion at temperatures around the alpha transus has been shown to develop microstructures which are on a much finer scale than those formed by forging. Three alloys containing >3 at.% of refractory alloying additions and a few percent of B and Si were plasma melted to produce 50kg ingots, which were then extruded in full section at 1200ϒC. This paper describes a preliminary assessment of the microstructures and mechanical behaviour of these three alloys, in the as-extruded and heat-treated state. A comparison is also made of the properties of these alloys after they have undergone different processing routes.

ON THE BRITTLE-DUCTILE TRANSITION OF POLYSYNTHETICALLY TWINNED CRYSTALS OF TiAl: *Dongliang Lin*[1]; [1]Shanghai Jiao Tong University, Institue of Matls. Sci. & Eng., 1954 Huashan Rd., Shanghai 200030 P.R. China

Both single-phase and two-phase TiAl alloys have the brittle-ductile transition (BDT) behavior. For polysynthetically twinned (PST) crystals of TiAl, the transition temperature is in the range of 400-600°C. However, this behavior has not been fully understood yet. In this paper, the dislocation configurations in the PST crystals deformed at room temperature and at elevated temperature have been investigated by transmission electron microscope. It was found that 1/2<112] superlattice dislocations, as an independent slip vector, can be activated easily and played an important role in the deformation. Its core structure underwent a noticeable change from sessile to gilssile in the BDT temperature range. Taking into account the behavior of the other types of dislocations, it is reasonable to ascribe the main cause for the BDT phenomenon of PST TiAl crystals to the behaviour of 1/2<112] superlattice dislocations at elevated temperatures.

THE EFFECT OF TERNARY ELEMENTS, SURFACE TREATMENT AND COATINGS OF OXIDATION OF TiAl BASED ALLOYS: *Fuhui Wang*[1]; Zhaolin Tang[1]; [1]Chinese Academy of Sciences, Institute of Corrosion and Protection of Metals, State Key Lab for Corrosion and Protection, 62 Wencui Rd., Shenyang 110015 China

The status of intermetallics as structural materials was reviewed at First International Symposium on Structural Intermetallics(ISSI-1) held at Seven Mountain Resort, Champion, Pennsylvania, 1993. Since then, many research papers have been published about the following five classes of intermetallics: a2-Ti3Al, g-TiAl, Ti2AlNb(Orthorhombic), g*-Ni3Al and b-NiAl. However, as Williams[1] pointed out in his Keynote Address at ISSI-2, 1997, There are two systems that appear to be the front runners: g-TiAl and g*-Ni3Al. g-TiAl is attractive because of its low density, which leads to good specific stiffness and strength. In the last decade, g-TiAl based alloys have been attempted to use as compressor vanes for aero-engine[2] and turbochargers for automobile[3,4]. However, the oxidation resistance of g-TiAl based alloys could mot meet the demand of the real applications, since the Al2O3 and TiO2 mixed oxide scales formed on the alloy surfaces, but not the pure Al2O3[5-7]. In the last few years, as a result of this, o xidation and protection of g-TiAl alloys have become a world-wide subject of high temperature field. In this paper, the oxidation mechanisms of g-TiAl alloys and the effects of third element additions, surface treatment and coatings on oxidation resistance of g-TiAl were reviewed. A proper coating system and applicable surface treatment technique for the protection of g-TiAl alloys were proposed,

BONDING MECHANISMS IN TERNARY Γ-TiAl+X (X = Cr, Mn, Nb): A COMPARISON BETWEEN ABSORPTION NEAR EDGE FINE STRUCTURES AND THEORETICAL PREDICTIONS: Thierry Sikora[1]; Michel Jaouen[2]; *Gilles Hug*[1]; [1]ONERA-CNRS, Lem, UMR 104, BP72, Chatillon 92320 France; [2]University of Poitiers, LMP, UMR 6630 du CNRS, Bd 3 - Téléport 2 - BP 179 - SP2MI, Futuroscope 86960 France

The electronic structure of titanium aluminides seems to play a critical role on their plastic properties by influencing the core structure of dislocations, the planar faults energies, or the Peierls relief. The addition of small quantities of a third element (Cr, Mn, Nb) modify the interatomic bonds and enhances ductility in some cases. In this work, the local atomic order and the electronic structure of γ-$(Ti_{46}Al_{54})_{97}X_3$ (X=Mn,Nb,Cr) alloys have been studied by Electron Energy Loss and X-ray absorption spectroscopies. The local crystallographic structure and the substitution sites of addition elements have been determined by EXAFS in the framework of a multiple scattering formalism. It is shown that a strong elastic relaxation exists around the solute atom. A good match between experimental near-edge structures and theoretical unoccupied states has confirmed the existence of strong Ti-d~Ti-d and Ti-

d~Al-sp hybridizations in TiAl. From a band structure formalism, the influence of the ternary solute atom on charge density distribution has been studied and has shown that they improve the intensity of the d-d bond within the (001) planes.

HOW STABLE IS THE W PHASE IN TiAl-X ALLOYS: *Guosheng Shao*[1]; Panos Tsakiropoulos[1]; [1]University of Surrey, School of Mech. and Mats .Eng., Guildford, Surrey GU2 5XH UK

This work contributes to the stability of the w phase in TiAl-X alloys by combining electron microscopy and theoretical calculations of lattice stability. Starting from experimental observations, a hypothesis on the effects of the interaction between Al and transition metal elements on the w phase stability was proposed. While the hypothesis was supported by calculated heats of formation of the w phase using the total energy LMTO method, the LMTO calculations have also given predictions of w phase stability beyond the hypothesis. The interaction between experimental work and theoretical modelling has allowed us to produce a master figure for the prediction of w stability in various TiAl-X systems. Excellent agreement exists between our prediction and experimental findings both by us and others.

TENSILE AND FATIGUE PROPERTIES OF HIPED GAMMA-TiAl PREALLOYED POWDERS AND EXTRUDED GAMMA-TiAl INGOTS. : *Hartmut Baur*[1]; Rainer Joos[1]; Nico Eberhardt[2]; Alexander Lorich[2]; Heinrich Kestler[2]; Helmut Clemens[3]; [1]Daimler-Benz AG, Research and Technology/Metals, Wilhelm-Runge Strasse 11, Ulm, Baden-Wuerttemberg 89013 Germany; [2]Plansee AG, Technology Center, Reutte, Tyrol A-6600 Austria; [3]Universitaet Stuttgart, Institut fuer Metallkunde, Seestrasse 71, Stuttgart, Baden-Wuerttemberg 70174 Germany

Abstract: Gamma-TiAl alloys are considered as potential materials for automotive components, e.g. valves. Two processing routes for valve production are under investigation, namely near-net shape processing of prealloyed powders via HIPing and single/multistep extrusion of cast ingots to rods which are subsequently hot-formed to final dimensions. In this paper the mechanical properties of rod materials are presented. The tensile properties of extruded material are superior to those of HIPed Material. The fatigue behavior was investigated at RT and 650ϒC, which corresponds to the brittle-to-ductile transition temperature. Due to their higher strength levels extruded material shows higher fatigue limits than the HIPed one. Also the scatter of the data is small in comparison to that of the HIPed material. This behavior is explained by the low portion of nonmetallic inclusions and microstructural inhomogeneities within the extruded material. Such imperfection are frequently observed in HIPed material, where they act as preferred sites for crack initiation.

MICROSTRUCTURE CONTROL AND LAMELLAR STABILITY IN A CAST TiAl AS A FUNCTION OF Mo AND C: *Ho-Nyun Lee*[1]; David Ray Johnson[1]; Haruyuki Inui[1]; Myung-Hoon Oh[2]; Dang-Moon Wee[3]; Masaharu Yamaguchi[1]; [1]Kyoto University, Dept. of Mat. Sci. & Eng., Yoshida Honmachi, Sakyo-ku, Kyoto 606-8501 Japan; [2]Kumoh National University of Technology, Dept. of Mat. Sci. & Eng., Shinpyung-Dong 188, Kumi, Kyungbuk 703-701 Korea; [3]KAIST, Dept. of Mat. Sci. & Eng., Gusong-Dong, Yusong-Ku, Taejon 305-701 Korea

The lamellar stability in a cast TiAl alloys containing small amounts (less than 1.5 at.%) of Mo and C were investigated in order to determine the processing window where the orientation of the lamellar microstructure could be controlled by directional solidification using a seeding technique. A partial liquidus surface near gamma TiAl was constructed for Ti-Al-Mo-C system. The lamellar stability was determined by quickly heating the material into the single phase alpha region, holding, and then cooling to room temperature. By maximizing the volume fraction of the alpha2 phase present at room temperature while ensuring that the alpha phase can form directly from the liquid, compositions for the seeding experiments were chosen such that original orientation of the lamellar microstructure was restored upon heating to and cooling from the melting temperature. Two prospective compositions were found in Ti-Al-Mo and Ti-Al-Mo-C systems and directional solidification of these alloys will be discussed.

PHASE STABILITY AND HIGH TEMPERATURE BEHAVIOR OF MOLYBDENUM AND BORON CONTAINING NEAR-GAMMA TITANIUM ALUMINIDES: *Julie Ann Christodoulou*[1]; Harvey M. Flower[1]; [1]Imperial College of Science Technology and Medicine, Dept. of Mats., Prince Consort Rd., London SW7 2BP UK

Both molybdenum and titanium diboride are of interest in the development of near-gamma titanium aluminide alloys. For example the diboride refines grain size while molybdenum introduces the ordered cubic B2 phase (Ti2AlMo) which may enhance ductility while providing high temperature capability. The effects are expected to be interactive as molybdenum can alter the boride chemistry and morphology and the overall phase equilibria. In the present work both these additions have been evaluated, principally in two multiphase alloys: Ti-50Al-5Mo+8vol%TiB2-particulates and Ti-44Al-8Mo+11(Ti,Mo)B whiskers. The combined influences of the phase stability, volume fraction and morphology on mechanical behavior at temperatures between 750ϒC and 900ϒC will be described.

DESIGNING CAST XD TiAl ALLOYS: PROS AND CONS: *Ji Zhang*[1]; [1]Central Iron and Steel Reserach Institute, Dept. of Superalloys, Beijing 100081 China

One of the present desires for cast TiAl alloys is now placed on producing refined cast lamellar microstructure, resulting in the development of the XD alloys containing certain amount of Boron. However, the positive and negative effects of the monolithic TiB2 particles in current XD alloys are rather difficult to be modified. In this paper, the compound additions of Boron and rare-earth element Nd are employed to generate hetergeneic refractory particles for grain refinement. The metallurgy and casting conditions will be taken into account as well. The prospect of designing the cast XD TiAl alloys to pursue the optimum grain refinement effectiveness without a negative effect on the ambient ductility will be discussed

ELECTROSLAG REMELTING OF GAMMA TiAl INGOTS: *Lev B. Medovar*[1]; Boris I. Medovar[1]; Boris B Fedorovskii[1]; Alexander V. Chernets[1]; [1]ELMET-ROLL, P.O. Box 259, Kyiv 252150 Ukraine

There ae several "ingot" technology for manufacturing of intermetallic compounds ingots: already tested and in use Vacuum Arc Remelting and Electron Beam Melting. ESR known as "ingot technology" with more flexibility than VAR or EBM and intendent for use like smelting method even before use like refining method. Were tested ESR smelting process with heat generation in slag pool by non-consumable electrode: so known current conductive mould. The results of process computer simulation and laboratory scale meltings of the TiAl (mainly 50%:50%) ingots will be discussed as well as some suggestion for the ESR using for industrial manufacturing of TiAl ingots.

A NOVEL PROCESS TO FABRICATE Ti/Ti ALUMINIDE LAMINATE COMPOSITE: *N. L. Richards*[2]; Mahesh C. Chaturvedi[1]; Weijie Wang[3]; Qiang Xu[1]; [1]University of Manitoba, Dept. of Mech. and Indust. Eng., 364 Eng. Bldg., Winnipeg, Manitoba R3T 2N2 Canada; [2]Bristol Aerospace Ltd., Materials/Processes, 660 Berry Street, Winnipeg, Manitoba R3C 2S4 Canada; [3]Lanzhou University, Department of Materials Science, Lanzhou, Gansu China

This paper will present our current research into fabrication technology of a Ti/Ti aluminide laminate composite using elemental Ti and Al foils. The fabrication process consisted of two steps: (1) a reaction sequence; (2) a post-reaction heating process. Influence of the first-step processing parameters including heating temperature, holding time, applied pressure and thickness of the starting Ti and Al foils on reaction between the Ti and Al foils was systematically investigated and thickness of the resultant Ti aluminide as a function of these parameters was established. The second-step process was also developed to remove residual porosity and close cracks that formed during the first step process. Post-processing heat treatment was employed to modify the microstructure in the Ti aluminide layer and the microstructural constituents in the Ti aluminide layer were characterised with TEM in detail. The results successfully demonstrated the potential of this technique for producing Ti/Ti aluminide composites.

EFFECT OF HOT-EXTRUSION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A TWO-PHASE TIAL ALLOY: *Michael Oehring*[1]; Uwe Lorenz[1]; Roland Niefanger[1]; Fritz Appel[1]; Richard Wagner[1]; Helmut Clemens[2]; Nico Eberhardt[3]; [1]GKSS Research

Center, Institut for Material Research, Max-Planck-Strasse, Geesthacht D-21502 Germany; [2]Universitaet Stuttgart, Institut fuer Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany; [3]Plansee AG, Technology Center, Reutte A-6600 Austria

Cast ingots of a Ti-47at.% Al-(Nb, Cr, Mn, Si, B) alloy were hot-extruded in order to investigate the influence of processing conditions on microstructural development and the resulting mechanical properties. Hot-extrusion was performed over the temperature range 1250 - 1380ϒC on differently heat treated billets. Depending on extrusion temperature either fine equiaxed or refined lamellar microstructures were obtained. After hot-extrusion, compression and fracture mechanical tests were conducted with specimens parallel and perpendicular to the extrusion direction so that the effect of the texture as well as of an anisotropy in microstructure on the mechanical properties could be examined. It is found that the specimen orientation has no or only a relatively slight effect on the flow stress for alloys with equiaxed or refined lamellar microstructures, respectively. In contrast, the fracture toughness significantly depends on the specimen orientation for both types of microstructures.

PHASE EQUILIBRIA IN THE Al-RICH PART OF THE Al-Ti SYSTEM: *Martin Palm*[1]; Frank Stein[1]; [1]MPI fuer Eisenforschung GmbH, Phys. Metall., Max-Planck-Str. 1, Duesseldorf, NRW D-40237 Germany

According to the latest assessment of phase equilibria in the Al-Ti system, phase stability and phase equilibria at Al-contents above 55 at% Al are still not well established. This holds for the Al-rich phase boundary of g-TiAl as well as for the adjoining phases up to Al3Ti. It is known that between 65 at% and 75 at% Al a number of long-periodic superstructures exists. However, the exact number of compounds and their stability in dependence on composition and temperature has not been established. In order to clarify which phases do exist on the Al-rich side of the Al-Ti system and to establish the phase relations among these compounds, an experimental investigation was carried out. From diffusion couple experiments and investigation of equilibrated bulk alloys by metallography, X-ray diffraction, differential thermoanalysis and electron microprobe analysis the phase equilibria in the Al-rich part of the Al-Ti system were determined. The results are presented and discussed with respect to their significance for future alloy developments.

THE INFLUENCE OF NIOBIUM ON THE OXIDATION BEHAVIOUR OF TiAl-BASED INTERMETALLICS: *Marinus Frederik Stroosnijder*[1]; Jan Sunderkötter[1]; Vincent Haanappel[1]; [1]European Commission, Institute for Advanced Materials, Via Fermi 1, Ispra, VA 21020 Italy

The high temperature applications of gamma-TiAl based intermetallics are still limited by their corrosion resistance. Improvement of the corrosion resistance can be obtained by alloying. Particularly, small additions of niobium may exhibit beneficial effects on the oxidation resistance. A general overview of the effects of niobium additions on the oxidation behaviour of TiAl-based alloys will be given in the presentation. Experimental results using alloying and ion implantation in combination with advanced techniques, such as isotope tracer techniques will be presented. The mechanisms proposed in the literature to explain the niobium effects are critically discussed in view of the presented experimental results. Suggestions for clarification of the underlying mechanisms are given.

VARIATIONS IN THE MECHANICAL BEHAVIOR OF SINGLE CRYSTALLINE GAMMA-TiAl AS A FUNCTION OF ALUMINUM CONTENT: *Marc Zupan*[1]; K. J. Hemker[1]; [1]The Johns Hopkins University, Mech. Eng., 122 Latrobe Hall, 3400 North Charles St., Baltimore, MD 21218 USA

Dislocation activity in two-phase commercial TiAl alloys occurs most readily in the γ-TiAl phase, and measurements of the CRSS of γ-TiAl are needed to provide a solid foundation for modeling the mechanical performance of these alloys. Single crystal studies of γ-TiAl with greater than 55%Al have been forth coming, but differences between the results of these works and studies involving polycrystalline and two-phase alloys suggest that the underlying deformation mechanisms vary with Al content. Single crystals of γ-TiAl cannot be grown near the stoichiometric composition, but microsample tensile specimens have been machined from within very large single grains of overaged polycrystalline Ti-52%Al. The dependence of the CRSS on crystallographic orientation, temperature and Al content, as measured with single crystalline microsamples, will be discussed. TEM observations are being used to identify the controlling deformation mechanisms for both alloys, and comparisons will be made with current dislocation theories for γ-TiAl.

STUDY OF FRACTURE ORIGINS IN GAMMA-TiAl ALLOYS USING DISPLACEMENT MAPPING AND MICROSTRUCTURAL CHARACTERIZATION: *Nicholas E. Biery*[1]; Rafael Raban[1]; Tresa M. Pollock[1]; Marc De Graef[1]; [1]Carnegie Mellon, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Gamma-TiAl alloys are potential replacements for nickel-based alloys in engine applications. However, the modest ductility of these alloys is of concern, particularly in the vicinity of stress concentrators. An understanding of the events leading to fracture may help address these concerns by identifying desirable and undesirable features, which can be taken into account during alloy and component design. The strain mapping technique measures the surface components of the displacement of arrays of markers deposited on notched samples (250 micron notch radius). The markers are deposited by evaporation of gold or platinum through a commercially available 1500 mesh Ni grid. A pattern recognition algorithm is then used to locate the individual markers with sub-pixel resolution. Strain components are computed from the displacement components. Superposition of strain contours on scanning electron micrographs and maps obtained by orientation imaging microscopy then facilitates the correlation of microstructural features with strain localization and fracture. Strain mapping experiments reveal highly localized strains in large, favorably oriented gamma grains near the notch root. Subsequent transgranular fracture was commonly observed to initiate in these grains. This research was funded by AFOSR grant # F49620-95-1-0359.

HOT DEFORMATION OF GAMMA TiAl ALLOYS: FUNDAMENTALS AND PRACTICE.: *Renat Imayev*[1]; Gennady Salishchev[1]; Marat Shagiev[1]; Andrey Kuznetsov[1]; Fritz Appel[2]; Fritz Appel[2]; Fritz Appel[2]; Michael Oehring[2]; Valery Imayev[2]; [1]Institute for Metals Superplasticity Problems, Russian Academy of Sciences, Khalturina Str., 39, Ufa, Bashkortostan 450001 Russia; [2]Institute for Materials Research, GKSS Research Centre, Max-Planck-Str., Geesthacht, D-21502 Germany; [2]Institute for Materials Research, GKSS Research Centre, Max-Planck-Str., Geesthacht, Germany D-21502 Germany; [2]Institute for Materials Research, GKSS Research Centre, Max-Planck-Str., Geesthacht, Germany D-21502 Germany

Microstructural and chemical homogeneities are determinant for obtaining the high-quality parts of gamma TiAl alloys. One of the effective ways of homogeneity improvement is hot deformation including the grain refinement due to the dynamic recrystallization occurrence and the considerable improvement of microstructural and chemical homogeneities due to superplastic processing. In this talk, the following questions will be discussed: Effects of the material preparing techniques (cast, powder metallurgy) and the preliminary heat treatment on the dynamic recrystallization kinetics and chemical (phase distribution)homogeneity; Effects of Al content, alloying, grain size and lamellae orientation on the deformation mechanism and kinetics of dynamic recrystallization; The reasons leading to the striped microstructure formation and the possible ways of its avoidance; Formation of submicro- and microcristalline (d~0.1 and 1 mkm, respectively) structure in largescaled billets by means of multiple isothermal forging and isothermal equal channel angular extrusion under superplastic conditions.

TITANIUM ALUMINIDE POWDER PRODUCTION VIA REACTION BETWEEN TiCl4 AND Al: *Stephen J. Gerdemann*[1]; David E. Alman[1]; [1]Albany Research Center, Dept. of Energy, 1450 Queen Ave. SW, Albany, OR 97321 USA

TiAl is emerging as a candidate material for applications at elevated temperatures and in aggressive environment. This is being driven by the low specific properties and superior oxidation resistance of the compound compared to titanium alloys. For utilization of this material beyond a few highly specialized applications (e.g., aerospace/defense), new cost effective methods for producing TiAl components are re-

quired. This includes powder metallurgical and powder production unit operations. The present paper discusses the feasibility of producing TiAl by reacting TiCl4 and Al. This is similar to the well known Kroll process to produce Ti sponge from TiCl4 and Mg. Thermodynamic calculations show that above 600ϒC it is possible to produce TiAl from TiCl4 and aluminum. At lower temperatures AlTi3 is favored and TiCl4 is only reduced to TiCl2. Above 650ϒC TiCl4 utilization is improved but aluminum melts at 660ϒC. The results of the thermodynamic analysis were used to design experiments and the results of the experiments are compared with the thermodynamic analysis. Some ideas on the possibility of making inexpensive (i.e., <$3.00/lb) TiAl powder by this route are evaluated.

HIGH TEMPERATURE OXIDATION OF GAMMA TiAl ALLOYS: INFLUENCE OF REFRACTORY AND RARE EARTHS ELEMENT ADDITIONS: Stefano Gialanella[1]; Mohamed Nazmy[2]; Marc Staubli[2]; *Andrea Tomasi*[3]; [1]Università di Trento, Dipartimento di Ingegneria dei Materiali, Mesiano, Trento 38100 Italy; [2]ABB Power Generation, Gas Turbine Development, Baden 5401 Switzerland; [3]Istituto di Ricerca Scientifica e Tecnologica, Via Sommarive, Povo, Trento 38050 Italy

A number of alloy compositions and tempers have been considered in the research efforts carried out to get better gamma TiAl base alloys. Mechanical properties have been considered so far as main guidelines for alloy development. However, the increasing temperature capabilities achieved with the latest formulations of TiAl alloys have made of surface durability and, particularly, oxidation resistance a main issue for a valuable and reliable application of these materials. In the present study several alloys have been considered with respect to the isothermal oxidation behaviour in air over the 600-800°C temperature range. The effect of such alloying elements as W, Ta and Zr has been considered when added to base compositions containing Cr, Si and Y. The study, carried out using conventional thermoanalytical and microscopic investigation techniques, provides preliminary indications about the role the different elements play in improving the oxidation resistance of these promising alloys.

ROLE OF TITANIUM SILICIDES ON CREEP STRENGTH OF Si BEARING TiAl ALLOYS: Seung Eon Kim[1]; [1]Korea Institute of Machiney and Materials, Mats. Eng., 66 Sangnam, Changwon, Kyungnam 641-010 Korea

Prominent creep strength of Si bearing TiAl base alloys has been well known. Si addition to TiAl alloys inevitably yields Ti5Si3 type silicides. Precipitation hardening effect by the titanium silicides has been generally recognized in improving creep resistance. Unlike this, recently, solid solution hardening effect by Si itself has been also asserted to be dominant rather than precipitation strengthening. Another interesting report showed that strain assisted silicides are produced during creep test and enhance creep strength. So far, the creep strengthening mechanism by Si addition seems controversial. In this study, a systematic data for the effect of Si content on creep properties of Ti52Al48 base alloys will be depicted. From the results, role of titanium silicides on creep properties will be discussed and strengthening mechanism will be clarified.

INFLUENCE OF GASEOUS SPECIES ON THE OXIDATION BEHAVIOUR OF TiAl AT HIGH TEMPERATURES: *S. Taniguchi*[1]; [1]Osaka University, Dept. of Mats. Sci. and Proc., Graduate School of Engineering, 2-1 Yumadaoka, Suita, Osaka 565 Japan

TiAl coupon specimens were oxidized in various gas mixtures, O2-H2O, O2-CO2 and O2-N2, and in the constituent single gases under atmospheric pressure at 1100 and 200 K for up to 100 ks. Conventional metallographic examinations were performed for characterizing the oxidation products, using X-ray diffractometry, scanning electron microscopy combined with energy dispersive X-ray spectroscopy and so on. All the gases added to oxygen significantly enhance the oxidation, with the influence decreasing in the following order, water vapour, carbon dioxide and nitrogen. The addition of water vapor and carbon dioxide disturbs the sintering of oxide grains resulting in a fine porous structure in the inner scale layer. When carbon dioxide was added, TiC was found near the scale/substrate interface in the scale and rutile crystals in the outer scale layer show characteristically stepped structure implying the directional mass transport through them.

EFFECT OF LONG TERM STATIC AND CYCLIC THERMAL EXPOSURE ON THE MICROSTRUCTURAL STABILITY OF Ti-44Al-11Nb ALLOY: Carlos Hernandez[1]; Erica Corral[1]; Alvaro Chan[1]; Rabindra Mahapatra[2]; *Shailendra K. Varma*[1]; [1]The University of Texas at El Paso, ept. of Metall. and Mats. Eng., El Paso, TX 79968-0520 USA; [2]Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670 USA

Polycrystalline and aligned crystals of Ti-44Al-11Nb alloy have been subjected to oxidation environment in air at 900, 950 and 1000oC for a period of about one week. One cycle is defined as 55 minutes of heating and then 5 minutes cooling in air and has been continued for up to seven days or longer. Static mode consists of continuous heating of the sample in the furnace without interruption for similar periods of time. Cyclic mode appears to indicate lower rate of oxidation due to the necessity of recreating the diffusive paths after every cycle. Oxidation penetration in to the alloy follows a crystallographic direction parallel to the alternate lamellar directions of a2 and g phases in the microstructure. Large number of dislocations have been found to be generated during cyclic heating and semicoherent O-phase has been observed at 1000oC.

FRACTOMETRY OF TWO DIFFERENT TiAl-MICROSTRUCTURES: *Thomas Hebesberger*[1]; Christopher Semprimoschnig[1]; Reinhard Pippan[1]; Otmar Kolednik[1]; Helmut Clemens[2]; [1]Austrian Academy of Sciences, Erich-Schmid-Institut for Material Science, Jahnstr. 12, Leoben, Styria 8700 Austria; [2]Univ. of Stuttgart, Inst. f. Metallkunde, Seestrasse 71, Stuttgart 70174 Germany

In this study the fracture behaviour of a Ti - 46.5Al - 4(Cr,Nb,Ta,B) alloy is investigated in the temperature range between -196ϒC and 700ϒC. Two different microstructures are tested: a coarse-grained fully lamellar $\gamma + \alpha_2$ microstructure (FL) and a fine-grained near γ microstructure (NG). For both microstructures impact bending tests are performed, as well as conventional fracture mechanics tests. Conventional fractographic studies revealed that specimens with the NG microstructure change their fracture mode with increasing temperature from transcrystalline to intercrystalline fracture. Specimens with the FL microstructure, however, fail predominantly in a translamellar mode. For a more thorough inspection of the fracture surfaces the crystallographic fractometry is applied. This is a new tool for the analyses of cleavage fracture surfaces, which is based on the combination of crystal orientation measurements with electron backscatter diffraction (EBSD) and the automatic reconstruction of fracture surfaces from stereo-image pairs [1]. The technique allows the measurement of the spatial orientation of cleavage facets and their crystallographic indication. It is found that for the FL microstructure different cleavage planes are activated in the two different fracture tests: In the broken fracture mechanics specimens the predominant cleavage facets lie in the α_2 phase having a {0001} orientation. Contrarily, in the impact bending specimens cleavage planes with {111} orientation inside the γ-phase are activated. For the NG microstructure no such difference was observed in the fracture behaviour between the impact bending and the fracture mechanics tests. The material was kindly provided by the PLANSEE AG, Austria. Semprimoschnig C.O.A. et. al: "A new powerful tool for surveying cleavage fracture surfaces", Fatigue & Fracture of Engineering Materials & Structures, Vol. 20 No. 11, 1997, pp. 1541-1550.

CHEMICAL COMPOSITION EFFECTS UPON CREEP STRENGTH OF GAMMA-BASE TITANIUM ALUMINIDES ALLOYED WITH VANADIUM: *Tohru Takahashi*[1]; Toshiaki Sujino[2]; Yoshihiro Abe[2]; Tadashi Hasegawa[1]; [1]Tokyo University of Agriculture and Technology, Dept. of Mech. Syst. Eng., Naka-cho 2-24-16, Koganei, Tokyo 184-8588 Japan; [2]Tokyo University of Agriculture and Technology, Graduate School, Naka-cho 2-24-16, Koganei, Tokyo 184-8588 Japan

Creep strength is one of critical parameters for materials applied in heat resisting structures. In the present study, creep characteristics have been investigated in gamma-base titanium aluminides of aluminum-titanium-vanadium ternary compositions. Aluminum content varied from 50 to 60 at.%, and vanadium was added up to 25 at.%, and the remainder was titanium. A small amount of beta phase was present in materials with higher vanadium content. In order to characterize the effect of vanadium content upon creep strength, compressive creep tests were carried out in recrystallized polycrystals with various compositions.

Minimal value in minimum creep rates was found at 5 at.% V and 10 at.% V in 50 at.% Al and 55 at.% Al materials, respectively. Creep strength decreased in the materials containing higher vanadium, probably due to the presence of beta phase which seemed to be weaker than the gamma phase at high temperatures.

GAMMA TITANIUM ALUMINIDE DEVELOPED BY INTERDIFFUSION OF MULTI-LAMINATED COMPOSITES OF TITANIUM AND ALUMINUM: *Jian-Guo Luo*[1]; Viola L. Acoff[1]; [1]The University of Alabama, Metall. & Mats., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

Multi-laminated sheets of pure titanium and aluminum were prepared by cold-bonding and annealing at various temperatures and times. The technique of developing gamma titanium aluminide from the interdiffusion process between titanium and aluminum were examined by scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy, x-ray diffraction and light microscopy. Based upon these examinations, the active energy and mechanism of interdiffusion between titanium and aluminum, the interdiffusion coefficients and the reaction kinetics were determined for different annealing temperatures and imes were determinated. The mechanical properties of the resulting gamma titanium aluminide layers were determined using microhardness and nanoindentation testing. Results of this study show that TiAl3 forms during the developing process for gamma titaniu aluminides. A two-stage annealing process, in which one is at higher temperature and longer time, is needed in order to develop higher level gamma titanium aluminides. The research also confirmed the interdiffusion of titanium and aluminum is a parabolic growth process controlled by volume diffusion.

CREEP BEHAVIOR OF A CAST TiAL-BASE ALLOY FOR GAS TURBINE BLADE APPLICATION: *Valentino Lupinc*[1]; Wei-Min Yin[2]; Maurizio Maldini[1]; [1]CNR-TEMPE, Via Cozzi 53, Milan 20125 Italy; [2]Institute of Metal Reserach, 72 Wenhua Road, Shenyang 110015 PR China

Creep behavior of various batches of the Ti-47Al-2W-0.5Si cast alloy having small composition variations has been examined. The microstructure, that strongly influences the creep resistance, was found to vary with composition, solidification path and heat treatment region in the phase diagram. In particular the creep behavior of the most creep resistant duplex microstructure material is described in detail, trying to define some physical parameters controlling the minimum creep rate, and interpolated within the experimentally explored range. The duplex microstructure consists mainly of lamellar gamma/alpha2 colonies, a smaller amount of globular gamma grains, and also some secondary beta and silicide phases, and provides a good compromise of mechanical properties when solidification occurs through the b phase. Stress-rupture and creep resistances of this cast alloy are compared to the properties of some other TiAl base alloys and to the density corrected IN738LC stress-rupture behavior.

HIGH TEMPERATURE LOW CYCLE FATIGUE PROPERTIES OF Ti-45Al-2Nb-2Mn+0.8vol.% TiB2 AND Ti-47Al-2Nb-2Mn+0.8vol.% TiB2 IN SITU GAMMA TITANIUM ALUMINIDE COMPOSITES: *Viktor Recina*[1]; [1]Volvo Aero Corporation, Mats. R&D, Maloga, Trollhattan, Vastergotland 461 81 Sweden

Low cycle fatigue testing of Ti-45Al-2Nb-2Mn+0.8vol.% TiB2 and Ti-47Al-2Nb-2Mn+0.8vol.% TiB2 in situ gamma titanium aluminide composites is going to be performed at 600ϒC in fully reversed push-pull mode (R=-1) with a triangular wave form. The microstructures of the two alloys are going to be characterized through optical and scanning electron microscopy. The fracture surfaces will be investigated to elucidate the fracture initiation and growth behavior. Both the fracture behavior and the fatigue performance will be correlated to the microstructure. The fatigue properties and behavior will be compared to previous low cycle fatigue testing of monolithic gamma titanium aluminide alloys tested at similar conditions. The low cycle fatigue testing is planned to be finished in the end of May, 1998, the evaluation will be finished not later than August, 1998.

THE INFLUENCE OF VARIOUS ALLOYING ADDITIONS ON Z-PHASE STABILISATION AND THEIR ROLE IN THE OXIDATION BEHAVIOUR OF GAMMA-TiAl BASED ALLOYS.: *Vladimir Shemet*[1]; Lorenzo Singheiser[1]; *Willem Joe Quadakkers*[1]; *Willem Joe Quadakkers*[1]; [1]Forschungszentrum Juelich, Institute for Materials in Energy Systems, Juelich D-52425 Germany

The high temperature oxidation behaviour of gamma-TiAl intermetallics containing various alloying additions has been studied in air and oxygen at 800-900ϒC. It was found that alloying elements can have a beneficial effect on the oxidation resistance of gamma-TiAl intermetallics in two ways: I) reducing the growth rate of the mixed TiO2/ Al2O3 scale; II) promoting protective Al2O3 formation. The beneficial effect of Nb, Mo, W, Sc and La additions are related to modification of the properties of the mixed scale (mechanism I). The alloying elements (Ag, Cr, Zr and Hf) promote formation of a protective alumina based scale. This is due to stabilisation of the ternary Z-phase (Ti5Al3O2) beneath the scale. The best long term oxidation resistance for temperature up to 900ϒC in Ar/O2 was obtained for gamma-TiAl alloys containing small additions of both Zr and Nb. For interpreting the beneficial or detrimental effect of alloying addition on gamma-TiAl alloys oxidation, the significant of the composition of the test atmosphere (oxygen or air) has to be considered. It was found that presence of nitrogen in the oxidation atmosphere is in some cases detrimental, in others beneficial. For gamma-TiAl based alloys, which tend to form alumina based scale, nitrogen always seems to be detrimental because it retards the Z-phase formation in the Al-depletion layer. As a result it promotes the formation of more rapidly growing, mixed Al2O3/TiO2 scales. A beneficial effect of nitrogen was found for gamma-TiAl based alloys which tend to show internal oxidation of aluminium. The nitride layer which is forming at the scale/alloy interface prevents this process. The presents results indicate, that long term protective alumina formation can be obtained by alloying elements which stabilize the Z-phase as well as prevent the formation of Ti-rich nitrides beneath the alumina scale.

MICROSTRUCTURAL MODIFICATION OF INVESTMENT CAST Ti-47Al-2Nb-1Mn-0.5Mo-0.5W-0.2Si ALLOY BY HEAT TREATMENT: *Weijie R. Chen*[1]; Jonathan Beddoes[1]; Linruo Zhao[2]; [1]Carleton University, Dept. of Mech. & Aero. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]National Research Council of Canada, Structures, Materials & Propulsion Lab., Institute For Aerospace Research, Montreal Road, M-13, Ottawa, Ontario K1A 0R6 Canada

The modification of the columnar cast duplex structure of a complex investment cast gamma alloy via heat treatment is presented. An innovative primary heat treatment process is applied to cast bars to develop an equiaxed fully lamellar structure while preventing minor phase precipitation and the formation of massively transformed g. The resulting fully lamellar structure has relatively fine lamellar interface spacing that is dependent on the homogeneity of the cast structure, and well interlocked lamellae along lamellar grain boundaries. It is believed that these features will enhance elevated temper-ature properties. An advantage of the primary heat treatment profile utilized is the ability to control the formation of minor phases precipitated during a secondary heat treatment process. Minor phase precipitates are enriched in Nb and Si, and precipitate primarily along lamellar interfaces. The formation of the precipitates is also strongly dependent on the compositional segregation of the cast material.

DEFORMATION BEHAVIOUR OF GAMMA TITANIUM ALUMINIDES - MICROMECHANICAL MODELLING OF SINGLE PST CRYSTALS AND POLYCRYSTALLINE MATERIALS: *Wilfried Thomas Marketz*[1]; Franz Dieter Fischer[1]; *Helmut Clemens*[2]; [1]Montanuniversitaet Leoben, Institut fuer Mechanik, Franz-Josef-Strasse 18, Leoben A-8700 Austria; [2]Universitaet Stuttgart, Institut fuer Metallkunde, Seestrasse 71, Stuttgart D-70174 Germany

The mechanical properties of γ-TiAl based two phase alloys consisting of the γ-TiAl phase and a small volume fraction of the α2-Ti3Al phase are determined by the microstructure. The deformation characteristics are simulated by a three dimensional model based on the unit cell technique using the finite element method [1]. This model considers crystallographic slip and deformation twinning as the deformation mechanisms in addition to the elastic behaviour. The microstructure and the crystallography are incorporated by the framework of crystal plasticity. Initially, this micromechanical concept was applied to single polysynthetically twinned (PST) crystals of TiAl. The results of the

simulation reflect the anisotropic plastic behaviour of the lamellar microstructure which was experimentally observed, too. Since it has been shown that the chosen micromechanical model reproduces the deformation mechanisms in a proper way, the concept was now extended to polycrystalline material. In order to start with a rather simple case, a so called near gamma microstructure was chosen. Furthermore it was demonstrated that only a fully three-dimensional modelling delivers reasonable predictions.[1] S.M. Schloegl: Micromechanical modelling of the deformation behaviour of gamma titanium aluminides, Fortschritts-Berichte VDI, Reihe 18: Nr. 220,1997.

FATIGUE INITIATION IN NEAR-LAMELLAR 3-95 (Ti-46Al-2Nb-2Cr-1Mo-0.2B): *W. John Porter*[1]; Kezhong Li[1]; David C. Maxwell[1]; Andrew Rosenberger[2]; James M. Larsen[2]; [1]University of Dayton Research Institute, Structural Integrity, 300 College Park, Dayton, OH 45469-0128 USA; [2]United States Air Force, Air Force Research Labs, 2230 Tenth Street, Ste. 1, WPAFB, OH 45433-7817 USA

Gamma titanium aluminides are being considered for application as rotating components in aerospace turbine engines. To increase the likelihood of gamma application, a thorough understanding of the relationship between microstructure and fatigue initiation is required. This study investigated the fatigue performace of a wrought, near-lamellar gamma alloy, 3-95 (Ti-46Al-2Nb-2Cr-1Mo-0.2B), at RT, 540 and 700ºC tested at stress ratios of 0.1 and 0.6. Up to five samples per stress level were tested in order to determine the causes for data scatter. Detailed fractography was perfomed to determine the mechanisms leading to initiation. A comparison of the results from this alloy are made to other current gamma alloys tested under similar conditions.

THE FUTURE USE OF GAMMA TITANIUM ALUMINIDES BY ROLLS-ROYCE: *Wayne E. Voice*[1]; [1]Rolls-Royce Plc, Aero-Engine/ Materials, Elton Rd., (ELT-38), P.O. Box 31, Derby, Derbyshire DE24 8BJ UK

Gamma TiAl is essential for meeting military and civil engine performance targets in the future and potentially it could be used throughout the engine from the compressor to turbine. The current Rolls-Royce alloy is the established Ti-45-2-2-XD and this is competing for lower temperature applications such as stators and structural components which take advantage of the lower costs arising from the casting route. Rigorous design criteria are required to compensate for the risks in using these relatively new materials in components and this requires investigation into the effects of manufactured surface conditions, of microstructures local to load bearing regions and of compositional variations. For the future, Rolls-Royce has patented a number of next generation cast titanium aluminides resulting from alloy development programmes undertaken by Birmingham University. These aim to optimise castability with strength and creep resistance and their potential for commercial use within the aero-engine will be discussed.

MICROSTRUCTURAL CHARACTERIZATION OF LASER-DEPOSITED TiAl ALLOYS: *Xiao-Dong Zhang*[1]; Richard Grylls[1]; Dan Evans[2]; Hamish Fraser[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Wright Patterson AFB, Wright Laboratory, WL/MLLM, Dayton, OH 45433 USA

Several TiAl alloys have been laser deposited using Laser Engineered Net Shaping (LENS) technology. LENS is a laser direct metal deposition process that combines laser cladding technologies with advanced rapid prototyping methods with capability to directly manufacture complex three-dimensional components. Different microstructure can be obtained depended on the processing parameters (laser scan speed, powder feed rate and pressure, subtract materials and post heat treatment). A number of high temperature metastable phases, such as a and b phases, have been retained at room temperature. By careful control of post heat treatment, we can optimize the microstructure and mechanical properties. Significance of the LENS technique will be discussed in terms of near net-shape manufacture, microstructural control and potential applications.

THE FRACTURE MECHANISM STUDY OF PST CRYSTAL THROUGH IN-SITU OBSERVATION: Yonggang Zhang[1]; [1]Beijing University of Aeronautics and Astronautics, Dept. of Mats. Sci. and Eng., 37 Xueyuan Rd., Haidian, Beijing 100083 China

The fracture mechanisms of PST crystals of Ti-49at.%Al in different orientations have been investigated through in-situ observations by both SEM and TEM. The results showed that the fracture behavior and mechanisms are strongly dependent on the angle of loading axis to the lamellae and controlled by ability of dislocation emission ahead of crack tip. When the loading axis is parallel to the lamellae, the crack propagated transversally by nucleation, growth and linkage of microcracks, and the main crack could be blunted by interface sliding and emission of ordinary dislocations and twinning. When the loading axis is perpendicular to the lamellae, the crack propagated along the interfaces and the tip kept sharpness, and no dislocations were emitted ahead of the crack tip. The results were analyzed micromechanically with the particular attention on stress distributions and toughness mechanism.

MICROCRACKING IN POLYSYNTHETICALLY TWINNED (PST) TiAl CRYSTALS UNDER COMPRESSION:: *Zhe Jin*[1]; Carl M. Cady[1]; George T. Gray[1]; Masaharu Yamaguchi[2]; Masaharu Yamaguchi[2]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech., MST-8, MS G755, Los Alamos, NM 87545 USA

Microcracking behavior of polysynthetically twinned (PST) TiAl crystals under a compressive loading condition was studied at different strain rates and temperatures. The microcracking was observed to start at very small strains at room temperature for all strain rates studied, ranging from 0.001/s to 3000/s. The interlamellar microcracks were formed primarily due to a mismatch shear stress across lamellar interfaces, which depended on the deformation of individual domains on these interfaces. The translamellar microcracks were observed to be a mode-I type crack. As the testing temperature increases, the formation of microcracks was observed to be delayed in terms of strain.

LEACHING THEORY PROCESS DEVELOPMENT & INDUSTRIAL PRACTICE: Pressure 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

Tuesday PM — Room: 1B
March 2, 1999 — Location: Convention Center

Session Chairs: Dr. D. Dreisinger, UBC; C. Fleming, Lakefield Research

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THE BEHAVIOR OF ORPIMENT DURING PRESSURE OXIDATION: PART 1, CHEMISTRY: *Hu Long*[1]; David G. Dixon[1]; [1]University of British Columbia, Dept. of Metals and Mats. Eng., 309-6350 Stores Rd., Vancouver B.C. V6T 1Z4 Canada

In this, the first systematic study of the oxidation of orpiment (As_2S_3) in sulfuric acid solution under oxygen pressure at temperatures above 170ºC, the efforts of temperature, particle size oxygen parietal pressure, the concentration of sulfuric acid and pulp density were evaluated The effect of ferric ion addition was also examined. The products of oxidation are As (III), As (V), SO_4^{2-} and elemental sulfur. At temperatures ranging from 170 to 230ºC, most of the arsenic was found to in the trivalent state, and oxidation to As (V) with oxygen was very slow. This was confirmed by attempting to oxidize a synthetic arsenious solution. However, the rate of oxidation of As (III) to As (V) is rapid in the

presence of ferric ion. Elemental sulfur was found at all temperature investigated except at 230ϒC.

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PRESSURE LEACHING OF NIOBIUM AND TANTALUM FROM COLUMBO-TANTALITES: *M. Ruiz*[1]; C. Rodriquez[1]; M. Gonzalez[1]; J. Rivarola[1]; [1]Universidad Nacional de San Luis-CONICET, Instituto de Investigaciones en Tecnologia Quimica (INTEQUI), Casilla De Correo 290, San Luis Argentina

The extraction of niobium and tantalum with hydrofluoric acid from a columbo-tantalite of San Luis (Argentina) using a pressure Parr reactor of 450 ml, made of monel, is being studied. The reagents, products and residues have been characterized by techniques such as SEM, FRX, ICP-AES, BET and particle size analysis. The effect of the following variables on recovery has been studied: temperature, reaction time, acid concentration, pulp stirring rate, particle size and solid-liquid ratio. Results indicate that Nb and Ta extractions increase with increasing reaction time at 75ϒC. For higher temperatures the extraction grows until 90 minutes and for longer times the extraction values decreases They are not markedly affected by the stirring rate in the interval studied. Decreasing particle size, from +80 to -325 mesh, does not considerably improve extraction levels. Increasing solid concentration causes a decrease of the amounts of metals recovery. The ICP-AES analysis of the reaction products show that there is strong dissolution of niobium, tantalum, iron and manganese contained in the mineral. These results might indicate that Nb and Ta extraction is a consequence of a dissolution process of all the particles in the mineral. The XRD diagrams of the residues did not show formation of new crystalline structures but the minerals accompanying de columbo-tantalite (quarz, muscovite and feldspar) almost totally disappeared. FRX analysis of the residues showed that 1) for the least energetic working conditions, the amounts of Nb, Ta, Fe and Mn remaining in the residues correspond to the mineral stoichiometry, which would indicate a uniform attack on all the particle; 2) the residues treated at higher temperatures and longer reaction times show a slight enrichment in Ta.

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DESIGN OF PRESSURE HYDROMETALLURGICAL PROCESS PLANTS: *F. Campbell*[1]; L. Trytten[1]; W. D. Vardill[1]; [1]Dynatec Corporation, Metall. Tech. Div., P.O. Box 17, 10103-114 St., Fort Saskatchewan, Alberta T8L 2WI Canada

More than 45 years of experience in the scale up of pressure hydrometallurgical processes are reviewed from pioneering collaboration between Sherrit and Chemical Construction Company to process development in the present by their successor Dymatec Corporation. The evolution of testwork is discussed, from traditional pilot plant operations using semi-commercial equipment to small scale or minipiloting with equipment several thousand times smaller than commercial units. Nickel, uranium, zinc acid gold processes have been developed and successfully implemented in world scale operations, treating a variety offered materials including concentrates, ores and mattes. Historical data on testwork duration and ramp up of several commercial plants are presented.

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SULPHURIC ACID PRESSURE LEACHING OF LATERITES - METAL SOLUBILITIES AND SPECIATION ANALYSIS: *D. H. Rubisov*[1]; V. G. Papangelakis[1]; [1]University of Toronto, Dept. of Chem. Eng. and Applied Chem. 200 College St., Toronto, Ontario M5S 3E5 Canada

Sulphuric acid pressure leaching of nickeliferous laterites has attracted an attention from the nickel industry. This process allows for recovering nickel and cobalt by rejecting iron and aluminum in the same time. The process is especially advantageous for linmonitic laterites that mostly contain ferric oxo-hydroxide because iron precipitates releasing acid and thus rendering low acid consumption. It is also applicable to mixtures of linmonites and saprolites. Effective process design requires the solubility of metals that may precipitate during the process to be known. In the present work, determination of metal solubilities is based oil a simple specification program that assumes the presence of only a dominant complex for each metal. The thermodynamic data for the precipitation reactions are extracted from high-temperature experiments with monometallic systems published previously. The validity of the approach is then tested against mixed bimetallic systems, and finally applied to calculate the solubility of aluminium, iron and magnesium is laterite leaching effluents at temperature. In both cases of limonitic feed and lirnonitic/saprolitic blends, the prediction closely follows metal solubilities measured experimentally at temperatures from 230 to 270ϒC and at terminal free acidities ranging from 0.1 to 0.7 mol/1.

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PRESSURE LEACHING OF LAS CRUCES COPPER ORE IN THE DYNATEC MINIPLANT: *T. Xue*[1]; M. Collins[1]; M. Makwana[1]; J. MacLean[2]; I. Barton-Jones[2]; M. Southgate[2]; [1]Dynatec Corporation, Metall. Tech. Div., P.O. Box 17, 10103-114 St., Fort Saskatchewan, Alberta T8L 2W1 Canada; [2]Rio Tinto Technical Services, Castlemead, Lower Castle St., Bristol BS99 7YR UK

A hydrometalluical process has been developed for treating ore of the Las Cruces massive sulphide deposit located near Seville, Spain. A two-stage countercurrent leach process, consisting of an atmospheric leach and a pressure leach, has been developed to effectively leach copper from the copper-bearing minerals and to generate a solution suitable for the subsequent solvent extraction and copper electrowinning operation. The results of batch and continuous miniplant tests are presented.

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PRESSURE HYDROMETALLURGY, NO LONGER REGARDED WITH TREPIDATION FOR THE TREATMENT OF GOLD AND BASE METAL ORES AND CONCENTRATES: *Peter G. Mason*[1]; Jim W. Gulyas[2]; [1]Highlands Pacific, Ltd., Brisbane Queensland; [2]H. A. Simons, Ltd., Suite #400, 111 Durnsmuir St., Vancouver, British Columbia Canada

Over the last 15 years numerous Pressure Oxidation and Pressure Leach plants have been installed throughout the world. The technology is no longer regarded with awe and trepidation as plants designed to treat a wide range of feedstocks continue to be successfully brought on line. Important aspects of the design of such plants include feed preparation, slurry pumping, heating and heat recovery, pressure control and let-down, vessel design and provision of associated services.

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STOICHIOMETRIC AND KINETICS EFFECTS ON THE PRESSURE LEACHING OF ZINC CONCENTRATES: G. P. Demopoulos[1]; S. A. Baldwin[2]; [1]McGill University, Department of Mining and Metallurgical Engineering, 3610 University Street, Montreal, QC H3A 2B2, Canada; [2]University of British Columbia, Department of Chemical & Bioresource Engineering, Vancouver, B.C., Canada

Zinc pressure leaching is a very complex reaction system with the main reactions being oxygen mass transfer, oxidation of ferrous to ferric iron, ferric ion leaching of marmatite, and iron precipitation; the oxidation of other sulphide minerals present in the concentrate is a further complicating factor as is the small (but critical) conversion-oxidation of sulphur to sulphate. In literature the individual kinetics of all main reactions of this system have been published following investigations involving in general dilute mineral slurries in small bench scale batch reactors. A certain degree of uncertainty is inherent with the laboratory experimental measurements and the derivation of the appropriate stoichiometrical and kinetic relationships. In this paper, we examine the effect of these uncertainties have on the operation of zinc pressure leach autoclaves by performing a series of simulations with the aid of a comprehensive reactor model we have previously developed and described in literature. In particular in this study, we consider the sensitivity of the autoclave operation (attainment of steady state temperature, level of zinc recovery and final acid concentration) on (i) the stoichiometry of the marmatite oxidation reaction, (with emphasis given on the impact of the iron content of marmatite and the fraction of sulphide oxidized to sulphate; (ii) the activation energy of the marmatite oxidation reaction; (iii) the constant kinetic parameter for ferrous to ferric oxidation; and (iv) the apparent equilibrium constant for ferric ion precipitation.

LIGHT WEIGHT ALLOYS FOR AEROSPACE APPLICATIONS V: Titanium 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 Advanced Aerospace Materials, Pohang 790-330 Korea

Tuesday PM Room: 9
March 2, 1999 Location: Convention Center

Session Chair: James Fragomeni, Ohio University , Dept. of Mech. Eng., Athens, OH USA

2:00 PM

DEVELOPMENT OF A NEW BETA TITANIUM ALLOY (BETA-636): *Dongjian Li*[1]; Kevin J. Doherty[2]; S. J. Poon[1]; Gary J. Shiflet[2]; [1]University of Virginia, Dept. of Phys., 205 McCormick Rd., Charlosville, VA 22903 USA; [2]University of Virginia, Dept. of Mats. Sci. & Eng., Mats. Sci. Bldg., Charlottesville, VA 22903 USA

A new class of beta titanium-based alloys has been developed that possesses the desirable combination of properties of a high yield strength of up to 1650 MPa and an elongation of 10% in the aged state. After casting or solution annealing, its matrix is almost exclusively beta phase with trace amount of omega phase. No alpha phase is observed. Nanometer-sized alpha particles precipitate out from the beta matrix during aging, and grow very slowly. This, along with the homogeneous distribution of precipitates contribute to the excellent properties observed in this alloy. Further experiments are being conducted to produce ingots suitable for the industrial application of this alloy.

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OXIDATION BEHAVIOR OF TERNARY TiAl-Nb ALLOY: *Yang Li*[1]; *Ramana G. Reddy*[1]; [1]University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487 USA

The oxidation behavior of ternary TiAl-Nb alloy has been studied in pure oxygen over the temperature range of 750ϒC to 1100ϒC. The experiments were carried out using TGA. The oxidation products were analyzed using X-ray diffraction, SEM and EDS. Parabolic rate constants were calculated. An effective activation energy of 295 kJ/mol was deduced. The oxidation products were mainly a mixture of TiO2(rutile) and Al2O3 (alumina). For oxidation scale at 1000ϒC and 1100ϒC, it exhibited a layered scale of alternate rutile and alumina. The present results were also compared with that of binary TiAl.

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ENVIRONMENTAL EFFECT ON MECHANICAL PROPERTIES OF ALUMINIDE MATRIX COMPOSITES: *Masahiro Inoue*[1]; Katsuaki Suganuma[1]; Koichi Niihara[1]; [1]Osaka University, ISIR, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

Mechanical properties of nickel and iron aluminides and their matrix composites with ceramic particles and fibers were investigated in several types of environments. Ni3Al and FeAl alloys exhibits significant environmental embrittlement in air. Although the fracture properties of their matrix composites predominantly depends on the ductility at a crack tip, those are also strongly influenced by the environmental effect. The moisture induced embrittlement is a serious problem at ambient temperatures for the fracture resistance of these composites. The embrittlement phenomenon is found to be accelerated dynamically during the loading. The effect of stress applied to the specimens on the environmental embrittlement will be discussed in detail.

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EFFECT OF PROCESS VARIABLES ON THE HOT FORGE-ABILITY OF Ti-10V-2Fe-3Al ALLOY: *C. S. Lee*[1]; K S Choi[1]; D H Shin[2]; [1]Pohang University of Sci. and Technology, Center for Advanced Aerospace Mats., San 31, Hyoja-dong, Pohang 7909-784 Korea; [2]Hanyang University, Metall. and Mats. Sci., Ansan, Kyunngi-Do 425-791 Korea

Forgeability and mechanical properties of final forged products are largely dependent on various process routes. However, the detailed information on how the formability of Ti-10V-2Fe-3Al alloy is related to the initial microstructure and process variables such as temperature, strain rate etc. is not well understood. In this study it is aimed to investigate the flow characteristics of Ti1023 alloy over a range of temperature (540-840°C) and strain rate of 0.2 to 20 per s during hot compression tests and establish more reliable constitutive equation for stimulating the forging process. Constitutive equation based on the power law creep has been determined and incorporated into the commercial DEFORM program. Then the results of computer simulation have been compared with those of actual forged parts. To investigate the microstructural influence on the forgeability, microstructures containing thin and thick grain boundary alpha layers have been prepared and forged. Hot forging has been carried out above and belo w the beta transus. The optimum conditions for forging in both microstructures and process variables are discussed on the basis of present results.

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HIGH TEMPERATURE PROPERTIES OF TiB2/Ti SURFACE -ALLOYED MATERIAL FABRICATED BY HIGH ENERGY E-BEAM IRRADIATION: *Seong Hun Choo*[1]; J C Oh[1]; K Euh[1]; Sunghak Lee[1]; Yangmo Koo[1]; Nack J Kim[1]; [1]Pohang University of Science and Technology, Center for Advanced Aerospace Mats., Pohang 790-784 Korea

This study aims at improving the high temperature properties of TiB2/Ti surface alloyed materials fabricated by a high energy e-beam irradiation technique. The mixture of TiB2 powders and flux were deposited on a pure Ti substarte and a Ti10V2Fe3Al alloy substrate and then ebeam was irraditaed on these mixtures. Cracks and pores were found in the melted region of the sample processed without flux because of inhomogeneous thermal transfer. In the samples processed with a flux mixing ratio of 50% the melted region of about 1.5 mm thickness was homogeneously formed without defects and was composed of primary and eutectic TiB in the matrix. This microstructural modification including TiB greatly improved hardness especially high temperature hardness upto 450°C. These findings suggested that surface alloying using high energy e-beam irradiation was economical and usefull to the development of new advanced material with improved high temperature properties.

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OXIDATION OF ORTHORHOMBIC TITANIUM ALUMINIDE Ti22Al-25Nb IN Ai BETWEEN 650 AND 1000°C: *C. Leyens*[1]; [1]Oakridge National Lab, P.O.Box 2008, Oak Ridge, TN 37381 USA

Orthorhombic titanium aluminides are currently under investigation for aeroengine applications up to about 700°C. In some cases, orthorhombic alloys have proven superior to g-TiAl and Ti3Al based alloys with respect to fracture toughness, ductility and specific yield. Due to their low coefficient of thermal expansion and their low reactivity with SiC, orthorhombic titanium alloys are also considered as matrix material for fiber reinforced composites. The oxidation resistance of Ti22Al-25Nb, a typical orthorhombic alloy, is found to be comparable to conventional titanium alloys and Ti3Al based aluminide alloys between 650 and 800°C in air, AT 900 and 1000°C in air, the parabolic rate constant is initially on the order of that of g-TiAl based alloys, however, transition to linear growth kinetics is observed after several ten hours, resulting in poor oxidation resistance. The oxide scale contains TiO2, Al2O3 and AlNbO4 with rutile as the dominant oxide phase. Underneath the oxide scale, a nitride layer is formed. The nitride layer grows with exposure time, indicating that nitrogen diffuses through the outer oxide scale. Oxygen is enriched below the oxide scale forming an embrittled layer as wide as 300mm after 500h exposure at 900° C. The peak hardness at the outermost metal zone is up to five times higher than the bulk hardness of Ti-22Al-25Nb.

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EFFECT OF PROCESSING VARIABLES ON THE DENSIFICATION OF PIMED TiAl PARTS: *Yongchan Kim*[1]; Nack Kim[1]; Taesik Yoon[2]; S Ahn[2]; [1]Post Tech, Center For Advanced Aerospace Mats., San 31, Pohang 790-784 Korea; [2]Research Institute of Industrial Science and Technology, San 31, Pohang Korea

Very fine size of powders under 10 micron is required to achieve a near full density (>95%) of PIM(Powder Injection Molding) parts. Since the remaining pores distribute evenly throughout the PIMed part, HIPping can remove the pores completely without distorting the part shape. Therefore, PIM process can be used to manufacture a hard-to-fabricate part with a stringent material property requirement such as TiAl. In the case of the P/M processing of the alloys containing elements with a high oxygen affinity such as Ti and Al, extreme care should be taken to minimize the oxygen content in the powders and processing environment. The oxides, surface or internal, work as a diffusion barrier and cause premature halt of densification. During the thermal degradation process of organic binder, the metal powders may undergo oxidative or reducing environment. Debinding can also result in excess carbon residue depending on the process. These interstitial elements should have a significant influence on the sintering behavior of PIMed TiAl. In this study, the effect of PIM processing variables in debinding and sintering on the densification of TiAl, especially the role of interstitial elements, was investigated.

MATERIALS PROCESSING FUNDAMENTALS: Spray Forming & Thin Films

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

Program Organizers: W.D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Huimin Liu, UES, Inc., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Laboratory, P.O. Box 2008, Bldg. 4508, Oak Ridge, TN 37831-6083 USA

Tuesday PM Room: 5A
March 2, 1999 Location: Convention Center

Session Chairs: Ralph H. Zee, Auburn University, Auburn, AL 36849 USA; Huimin Liu, UES Software, Annapolis, MD 21401 USA

2:30 PM INVITED PAPER

PARTICLE MASS FLUX IN THE SPRAY CONE OF A FREE FALL ATOMIZER: *Volker Uhlenwinkel*[1]; Marko Buchholz[1]; Klaus Bauckhage[1]; [1]University of Bremen, Chem. Eng., Badgasteiner Str. 1, Bremen D-28359 Germany

The design of a free fall atomizer as well as the process parameters affect the particle mass flux distribution in the spray cone The mass flux distribution is important in spray forming because of its influence on the deposit shape and enthalpy input into the deposit. In this article the effect of different process parameters on the mass flux distribution is shown. Also different melts were atomized with the same atomizer. An empirical relation will be introduced to predict the mass flux in the spray cone. This relation will be compared with the experimental results. Finally, results of a scanning atomizer will be presented and the effect of the frequency will be discussed.

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PDA-BASED EXTENDED MODELS DESCRIBING THE MASS AND ENTHALPY FLUXES TO THE DEPOSIT AND THE PHENOMENS WITHIN THE DEPOSIT: *Klaus Bauckhage*[1]; [1]University Bremen, Chem. Eng., Badgasteiner Strabe 3, Bremen D-28359 Germany

Spray forming in some areas has become a successful alternative to conventional materials, fabrication techniques like powder metallurgy. It opens the opportunity to combine microstructural refinement (in absence of macrosegregations) with the wide flexibility in alloying and mixing as well as - under some conditions - neat net shape manufacturing with the option to produce large voluminous preforms. Spray forming means the subsequent combination of stream atomization into a wide size distribution of droplets and particle deposition on to a substrate and (furtheron) on each other, thus generating growing deposits and finally forming round billets, disks, tubes, rolls, or flat sheets without any macro- and micro-segregation. Since on the one hand spray forming has proved a good investment even under rough production conditions on the other hand growing experience under practical applications show that superheating or undercooling of the melt, mass-flow differences of the melt stream or variations of the gas pres sure May cause severe material problems of the pre-product, like coarse grain, pores of hot cracks. Thus the necessity of process control in order to avoid faults or to analyze errors in undesirable. There have been improved not only process description but also process control models and devices in order to avoid such faults of the material properties within the preform. Experimental data have been taken from representative measuring positions from inside the spray cone, helping - in comparison with the simulated date of different process modes - to describe the multiphase flow situations in the spray cone and the heat transfer conditions for the rapid cooling and partly solidifying of melt droplets before impact onto the substrate/deposit. The radial distribution of the local vertical mass flux of melt particles within the spray come for different distances from the atomizer can be described by a Gaurdian type of profile. These radial distributions differ from those profiles describing the local size distributions of the particles or the gas becomes responsible for the local enthalpy flux to the deposit. Due to the necessity of measuring and controlling the particle bound mass, momentium and enthalpy fluxes to the deposit the Phase Doppler Anemometry (PDA) has been adapted and modified in order to receive on-line and in-line data from the dynamics of the multiphase flow conditions within the spray chamber.

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DETERMINATION OF A SPLASHING THRESHOLD FOR IMPACTING METAL DROPLETS USING HIGH SPEED VIDEO IMAGING: Joachim Ulrich[1]; M. Berg[1]; *K. Bauckhage*[1]; [1]Universitat Bremem, Chem. Eng., Badgasteiner Str. 1, Bremen D-28359 Germany

Metal drops, whenever impacting with energies beyond a certain limit, create splashing. This splashing generates secondary drops - the disintergration of droplets into secondary droplets in many cases of application of spray processes is not wanted. In spray deposition processes leads to an increased overspray which reduces the yield of the process. The question is how to adjust the process parameters (e.g. drop size, drop velocity, drop temperature, target temperature in relation to the physical properties of the Mats. at those temperatures) - if possible to achieve atomization conditions leading to drop impacts with hardly any splashing. For tin, lead, copper, aluminium and steel drops experimental observations have been made using high-speed video imaging (up to 18.000 frame/sec.). The results are presented by means of non-dimensional numbers describing the fluid dynamic and heat transfer of the deposition process. An empirial found splashing threshold - described by a log-linear equation- will be presented in a double logarithmic diagram.

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EFFECT OF DEPOSITION AND POST HEAT TREATMENT ON NiTi SHAPE MEMORY ALLOY THIN FILMS: *Chen Zhang*[1]; Paul E. Thoma[2]; Ralph H Zee[1]; [1]Auburn University, 202 Ross Hall, Auburn, AL 36849 USA; [2]Johnson Controls, Inc., 1701 West Civic Dr., A37, Milwaukee, WI 53209 USA

Polycrystalline Ti-rich NiTi thin films were deposited from a single NiTi target using DC magnetron sputtering system. Free standing films were obtained by using silicon substrate. The thickness of the films was around 10-15 microns. In this investigation, the effect of different substrate temperatures during sputtering on the thin films' microstructure and transformation temperatures was examined. The influence of post heat treatment at different temperatures on the thin films' properties was also investigated. Transformation temperatures of the thin

films were measured using differential scanning calorimetry (DSC). The surface microstructure and cross sectional microstructure of the thin films were studied using a scanning electron microscope (SEM), and the crystallinity of the films was determined by X-ray diffractometry. Results show that films deposited on a hot substrate are crystalline even when the substrate temperature is as low as 300ϒC, while the normal crystallization temperature for an amorphous thin film is above 500ϒC. The microstructure of the above film has very fine grain size. The grain size increases with increasing post heat treatment temperature and increasing substrate temperature. The transformation characteristics of the films are correlated with the deposition conditions and post deposition treatment.

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ELECTRON-BEAM PHYSICAL VAPOR DEPOSITION OF MICROLAMINATE COMPOSITES: *H. L. Ludtke*[1]; G. E. Lucas[1]; C. G. Levi[2]; G. L. Bujanda[3]; J. T. Matzen[1]; [1]University of California at Santa Barbara, Dept. of Chem. Eng., Santa Barbara, CA 93106-5080 USA; [2]University of California at Santa Barbara, Mats. Dept., Santa Barbara, CA 93106 USA; [3]University of Texas at El Paso, Mats. Dept., El Paso, TX USA

Microlaminate composites were fabricated by electron-beam physical vapor deposition (EB-PVD). The laminates consisted of five 2 micron layers and were composed of either pure metal layers (Nb-Cu or Fe-Cu), or alternated between metal and intermetallic layers (Nb(Al)-Nb3Al). The metal-intermetallic microlaminates (MIMs) were synthesized in-situ by co-depositing pure Nb and pure Al, and varying the Al deposition rate to generate the layered structure. Microlaminates were examined after deposition by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy dispersive x-ray spectroscopy (EDS). The effects of deposition rate, substrate heater temperature, and substrate material on the quality of these composites will be described.

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FABRICATION OF TIN(IV) OXIDE THIN FILM BY SOL-GEL METHOD: *Seung-Chul Lee*[1]; Jae-Ho Lee[1]; [1]Hong Ik University, Dept. of Metall. Eng. and Mats. Sci., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 Korea

Transparent conducting tin (IV) oxide thin films have studies and developed for the electrode materials of solar cell substrate. Fabrication of tin oxide thin films by sol-gel method is process development of lower cost photovoltaic solar cell system. The research is focused on the establishment of process condition and development of precursor. The precursor solution was made of tin isopropoxide dissolved in isopropyl alcohol. The hydrolysis rate was controlled by addition of triethanolamine. Dip and spin coating technique were applied to coat tin oxide on borosilicate glass. The resistivity of the thin film was lower than 0.10-cm and the transmittance is higher than 90% in a visible range.

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REDUCTION OF CRACK PROBABILITY DURING SPRAY FORMING OF BILLETS: *Volker Uhlenwinkel*[1]; Joern Fischer[1]; Roland Schroder[2]; Stephan Hansmann[3]; Hilmar Muller[1]; [1]University Bremen, Chem. Eng., Badgasteiner Str. 1, Bremen D-28359 Germany; [2]University Bremen, Medical Eng., Badgast Str.1, Bremen D-28359 Germany; [3]Wieland Werke AG, Pruf-und Forschurgsanstat, Graf Arco Strasse, Ulm, Bayem D-89079 Germany

Cracks were observed at the top of spray formed billets for some alloys. In this paper the effect of process parameters on crack probability is discussed. After the spray rim the thermal conditions at the top of the billet change rapidly. A hot spot with melt residual can occur inside the billet and shrinkage will be suppressed due to the solidified surface of the billet. In this situation hot cracks can be initialized. The temperature history of the billet during and after the spray run is calculated. The temperature difference between core and outside diameter of the billet is taken as indicator for hot crack probability. The effect of different process parameter on the hot crack probability is discussed. The results lead to a better understanding of the process. The calculations are then as a tool to find process parameters in order to reduce or avoid hot cracks in spray formed billets.

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AVERAGING THERMAL CONDITIONS IN MOLTEN METAL SPRAYS: Dirk Bergmann[1]; Udo Fritsching[1]; *Klaus Bauckhage*[1]; [1]University Bremen, Chemical Eng., Badgasteiner Str. 1, Bremen D-28359 Germany

During the spray forming process, a continuous molten metal stream is atomized by impinging high speed inert gas jets. In the generated spray cone, the resulting metal droplets are rapidly cooled by the huge temperature difference to the surrounding gas phase and thereby partly solidified. After a certain flight and residence time inside the spray cone, the droplets impinge on the substrate and form the product (deposit). The material properties of this product depend on several process parameters and especially on the thermal state of the deposited droplets impingement. Smaller droplets cool very fast and may impinge onto the product in a completely solidified state as solid metal powder particles. Lager droplets contain a higher amount of thermal energy and impact during the state of phase change or completely liquid. It is obvious, that a certain amount of liquid content In the droplets is needed for forming the deposit. Therefore, the thermal history of metal droplets during flight in the spray cone is of great importance. To investigate this phenomena a CFD-program in combination with a solidification model, is used to describe the thermal history of individual droplets depending on their size and flight path inside the spray cone. in this solidification model the different stages like undercooling, recalescence and segregated solidification are taken into account. To describe the effects of the impinging droplet mass on the formation and heat balance of the deposit, the thermal conditions of the droplets have to be averaged at different locations. This averaging is achieved in different ways, giving differing results. The first method is the average over the droplets' total enthalpy. Which consists of the heat content and the remaining heat of fusion. The second method is the average over the heat content and the fraction solid separately. Results of these two methods show some deviations, especially at lager radial distances from the spray cone centreline, which will be discussed in terms of consolidation and energy conservation of the deposit.

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

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
Tuesday PM Room: 14B
March 2, 1999 Location: Convention Center

Session Chairs: I.-Wei Chen, University of Pennsylvania, Dept. of Mats. Sci. & Eng., Philadelphia, PA 19104-6272 USA; Anthony G. Evans, Harvard University, Div. of Applied Sci., Cambridge, MA 02138 USA

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CELLULAR METALS: *A. G. Evans*[1]; J. W. Hutchinson[1]; M. F. Ashby[2]; [1]Harvard University, Division of Applied Sciences, Cambridge, MA 02138 USA; [2]Cambridge University, Eng. Dept., Trumpington St., Cambridge CB2 11P2 UK

The property profile exhibited by cellular metals identifies several applications, especially in technologies requiring multifunctionality. Their specific property attributes suggest implementation as: ultralight panels/shells, energy absorbing structures and heat dissipation media as well as for vibration control. Connections between the properties that govern these performance benefits and the cellular architecture, cell morphology and density have been made. Such structural relations facilitate choices of optimum cell characteristics fordefined multifunctional application.

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MEASUREMENT OF RESIDUAL STRESSES BY INSTRUMENTED SHARP INDENTATION: *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Room 13-5056, Cambridge, MA 02139 USA

A general methodology is proposed for the determination of residual stresses using instrumented sharp indentation. Invoking the invariance of contact pressure in the presence of residual stresses, or the variation of the instantaneous yield strength in the presence of a residual plastic strain in a strain-hardening material, a step-by-step method is outlined to determine pre-existing residual fields for the following three cases. (1) The elastic residual stresses are uniform over a depth beneath the indented surface which is several times larger than the indentation contact area. (2) The elastic residual stresses vary with depth beneath the indented surface. (3) Residual plastic strains of uniform values exist over a depth several times larger than the indentation contact diameter. The method proposed here can be used to estimate residual stresses and strains in such applications as thin films, coatings, or engineered surfaces whose properties are altered by such properties as ion implantation, case hardening, shot peening, laser shock peening, or machining. The predictions of the proposed method are shown to compare favorably with finite element simulations and available experimental results.

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MECHANICAL BEHAVIOR OF SOLIDS WITH LIQUID GRAIN BOUNDARIES: *Gregory J. Rodin*[1]; [1]The University of Texas at Austin, Center for Mech. of Solids Structures and Mats., 112 WRW UT, Austin, TX 78712 USA

Many engineering and geophysical materials can be described as solids with liquid grain boundaries. The mechanical behavior of such solids is quite complex and it involves many open scientific issues. Among those issues are the following: (a) The overall creep behavior and its dependence on the grain diameter. (b) Stress distribution at the grain level that controls the flow. (c) Stress distribution at the grain boundary level that controls cavitation. We will consider these issues using asymptotic estimates valid for both newtonian and non-newtonian fluids.

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THE EFFECT OF AGING ON CRITICAL TRANSFORMATION STRESS LEVELS IN SINGLE CRYSTAL Ti-50.8at%Ni DEFORMED UNDER BOTH TENSION AND COMPRESSION: Kenneth Gall[1]; *Huseyin Sehitoglu*[1]; Yuriy I. Chumlyakov[2]; Irina V. Kireeva[2]; [1]University of Illinois, Dept. of Mech. and Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Siberian Physical and Technical Institute, Physics of Plasticity and Strength of Mats. Laboratory, Tomsk 634050 Russia

Polycrystalline NiTi that is commonly used in industrial and medical applications deforms quite differently under an applied tensile versus a compressive stress. The present research utilizes single crystal NiTi to determine the origin of tension/compression asymmetry in polycrystalline NiTi. Two different heat treatments (peak aged and over aged) and three different crystallographic orientations ([111], [110], and [100]) are utilized for the experimental study. In the over aged specimens, the asymmetry of the critical stress required to induce the transformation in tension versus compression obeys Schmid Law. For peak aged specimens, the asymmetry of the critical stress required to induce the transformation in tension versus compression is decreased, and the Schmid Law is not obeyed. The deviation from Schmid Law is accounted for by modeling the local coherent stress fields outside the precipitates in peak aged NiTi. It is also shown that polycrystalline NiTi has a strong texture of the <<111>{110} type, and the resulting deformation of polycrystalline NiTi is closely related to single crystals oriented in the [111] direction.

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TWO-PHASE CERAMIC LAMINATES FROM WET DEFORMATION PROCESSING: *I-Wei Chen*[1]; [1]University of Pennsylvania, Mats. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Laminates of structural ceramics are usually obtained by stacking up green tapes followed by hot pressing. An alternative method is by co-rolling and folding high density slurries. The exponential reduction of thickness and the exponential multiplication of layers make this process highly efficient. In addition, the different deformation resistance of the two starting slurries can be exploited to generate laminate and cellular microstructures of various spatial extent of phase connectivity. Once the long-range connectivity is lost, the sintering constraint is also relieved making pressureless firing possible. Meanwhile, a sufficiently long coherent length is still maintained to allow an extensive fracture process zone. Alumina/zirconia and other ceramic composites of very high strength and toughness have been created using this method.

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THERMAL STRESSES AND DEPOSITION PATTERNS IN LAYERED MANUFACTURING: *Alexander H. Nickel*[1]; David M. Barnett[1]; Friedrich B. Prinz[1]; [1]Stanford University, Mats. Sci. and Eng., Bldg. 550 Room 554F, Stanford, CA 94305 USA

In Layered Manufacturing objects are constructed by sequential deposition of material layers. When the deposition process involves temperature gradients, thermal stresses develop. This paper examines the effect of deposition patterns on the resulting stresses and deflections in fabricated objects. A finite element model was developed to predict the stresses and warpage for laser deposited metal parts. The resulting deflections and stresses showed a significant dependence on the deposition pattern. Experiments performed using these same deposition patterns yielded sample deflections which were in reasonable agreement with the finite element modeling predictions.

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THE MIGRATION OF GRAIN BOUNDARIES IN ALUMINUM-BICRYSTALS UNDER THE INFLUENCE OF AN EXTERNAL MECHANICAL STRESS: *Günter Gottstein*[1]; Miriam Winning[1]; [1]Aachen University of Technology (RWTH), Institute of Physical Metallurgy and Metal Physics, Kopernikusstr. 14, Aachen D-52056 Germany

The motion of grain boundaries is the key phenomenon of recrystallization and grain growth and dominates the evolution of texture and microstructure, and eventually the macroscopic physical and mechanical properties of a material. Grain boundaries are usually considered not to couple with mechanical stress owing to the lack of a long range intrinsic stress field of a grain boundary. This is certainly not true for small angle boundaries. The current study is aimed at probing the effect of a mechanical stress field on small and large angle grain boundaries. For this purpose bicrystals with <112> tilt grain boundaries and misorientation angles in a range from 4° to 21° were investigated under the influence of an external mechanical stress. The external stress used in the experiments ranged from 10-1 MPa to 10-4 MPa. In-situ measurements of grain boundary motion were conducted with an X-ray continuous tracking device. The method employed X-ray diffraction to determine the grain boundary position and, therefore, did not interfere with the grain boundary migration process itself. The mobility of the grain boundaries was measured over a wide range of temperature (250°C - 600°C) and the activation enthalpy could be determined from these measurements. It is shown, that there is an influence of a mechanical stress on grain boundary motion far beyond the commonly accepted transition between small and large angle grain boundaries. This transition from low angle to high angle grain boundaries could be identified by a conspicuous step in the activation enthalpy.

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CREEP BEHAVIOR OF Fe-C AND OTHER IRON-BASED ALLOYS AT HIGH TEMPERATURES AND HIGH STRAIN RATES: *Donald Ritchie Lesuer*[1]; Chol Kyle Syn[1]; J. D. Wittenberger[2]; O. A. Ruano[3]; M. Carsi[3]; O. D. Sherby[4]; [1]Lawrence Livermore National Lab,

Manuf. and Mats. Eng. Division, L-342, P.O. Box 808, Livermore, CA 94551 USA; [2]NASA-Lewis Research Center, Cleveland, OH 04135 USA; [3] CENIM, CSIC AV, Dept. Phys. Metall., Gregorio del Amo 8, Madrid S28040 Spain; [4]Stanford University, Dept. of Mats. Sci. and Eng., Stanford, CA 94305 USA

It is generally recognized that the time dependent plastic flow of pure metals and alloys at elevated temperature is determined by the diffusively, elastic modulus and stacking fault energy (SFE). In this paper we explore the influence of these material characteristics on the creep behavior of several steels at high strain rates. The materials studied include ultrahigh carbon steels (containing 1.2 - 1.8 %C), HSLA steel, 316 stainless steel and pure iron. A constitutive equation has been developed to describe the results with deformation involving power-law as well as power-law-breakdown behavior. Carbon was found to influence the creep rate of the steels through its influence on the diffusion coefficient and had no effect on the SFE in these materials. The substitutional solid solution alloy additions in these materials were found to produce large variations in SFE with significant influence on the resulting creep rate. The influence of these variations in diffusivity and SFE on the dominant deformation resistance will be discussed within the context of a deformation mechanism map.

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MOLECULARLY BASED NUMERICAL EVALUATION OF FREE-VOLUME IN AMORPHOUS POLYMERS: *Santosh K. Putta*[1]; Sia Nemat-Nasser[1]; [1]University of California, San Diego, Center of Excellence for Advanced Mats., 9500 Gilman Drive, La Jolla, CA 92093-0416 USA

The concept of free volume can be utilized both to study relaxation phenomenon as well as diffusion processes in polymers. Despite the lack of a clear understanding of the structure of free volume, the concept can be used as a linking factor between diffusional processes and deformation processes, which most often are coupled in polymers. A simple geometric definition of free volume, relevant for diffusion of small molecules in polymers is given, based on the molecular structure. An initial molecular structure is obtained by using a modified RIS (rotational isomeric state) approach. Then, with the aid of molecular mechanics based on minimization procedure, computationally generated molecular structures for several polymers are used to estimate free volume based on the proposed geometric definition. Numerically evaluated free-volume distributions are then compared for different types of amorphous polymers; e.g. polycarbonates and polysulfones, stressing on their relevance to the diffusion properties.

MILTON BLANDER INTERNATIONAL SYMPOSIUM ON "THERMODYNAMIC PREDICTIONS AND APPLICATIONS": Chemical Bonding and Kinetics

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. Eng., Tuscaloosa, AL 35487 USA; Dr. A. D. Pelton, Montreal, Quebec H3C3A7 Canada

Tuesday PM Room: 4
March 2, 1999 Location: Convention Center

Session Chair: John Morral, University of Connecticut, Dept. of Metall., Storrs, CT 06269-3136 USA

2:30 PM INVITED PAPER

PREDICTION OF PROPERTIES OF INTERMETALLICS USING A CHEMICAL BONDING MODEL: *Leo Brewer*[1]; [1]University of California, Berkeley, Dept. of Chem., Berkeley, CA 94720 USA

Materials with novel properties are needed for new technology developments. It is important to be able to predict which of the millions of multi-component intermetallics might provide the desired properties. A single chemical bonding model has provided reliable thermodynamic data for the metallic elements in a variety of crystal structures. (1) The model can be extended to intermetallics. An illustration of the calculations is available (2) for Al or Mg with transition metals forming intermetallics with binary CsCl structures. A number of publications will be available for similar calculations for a variety of crystal structures. The calculations will be extended to a variety of compositions and multi-component systems. The present paper will discuss the procedures that can be used to simplify the calculations and yet maintain reliable accuracy. (1) "Calculations of Thermodynamic Properties of Metastable Phases of the Elements", by John Kouvetakis and Leo Brewer. J. Phase Equilibria 14, 663-71 (1993) (2) "Calculations of the Thermodynamic Effect of the Brewer-Engel Generallized Acid-Base Reactions of 1:1 Intermetallics for Non-Transition Metals Al and Mg with Transition Metals", by Wu Hui-Fen and Leo Brewer. J. Alloys and Cmpds. 247, 1-8 (1997)

3:10 PM

THERMODYNAMIC STUDY OF THE ELECTRON TRANSFER IN LIQUID ALLOYS: *J. P. Bros*[1]; M. Gaune-Escard[1]; E. Hayer[2]; [1]Université de Provence I, IUSTI-CNRS UMR 6595, 5 rue Enrico Fermi, Marseille, Cedex 20 13453 France; [2]Institut fur Anorganische Chemie der Universitat Wien, Wahringerstrasse 42, Wien A-1090 Austria

By the evaluation of the results of our calorimetric measurements of many liquid binary alloys composed of a sp-metal and a transition metal, and due to the introduction of the Fermi enthalpy function with some assumptions, the concept of a transfer of electrons on alloying has been established. However, the number of electrons transferred from the sp-metal to the transition metal has been found limited for most of the alloys investigated (e.g., 2 electrons with Ni-Ga alloys for X(Ni) lower than 0.33). More and more measurements (by XPS, Cp at low temperature, resistivity) of the physical properties were recently performed on solid alloys of the mentioned type and are explained in terms of a transfer of electrons, too. The present article gives a comparison of physical and thermodynamic measurements. Summarizing, the concept of the metallic bond is discussed for these alloys.

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SIMPLE INORGANIC LIQUID GLASSFORMERS AND THE RELATION BETWEEN THEIR THERMODYNAMIC AND TRANSPORT PROPERTIES: *C. A. Angell*[1]; W. Richards[1]; K. Ito[1]; [1]Arizona State University, Dept. of Chem. and Biochemistry, Tempe, AZ 85287 USA

The factors which can affect the probability that a given liquid will fail to crystallize during cooling at moderate rates are not well understood, but usually favor liquids of complex structure. This complicates the problem of formulating analytical theories for their behavior. Fortunately there are some examples of molecularly simple systems that evidently fail to find low energy ordered packing schemes hence only become metastable at temperatures where their viscosities are already quite high. They therefore supercool more readily than would be expected at first sight. The four atom molecule S2Cl2 is one example and the even simpler (and much studied) liquid CS2, can be vitrified on addition of only small quantities of second components. In this contribution we take advantage of new and simple methods for fragility determination in liquids to discuss the behavior of these simple systems relative to that of other fragile liquids, especially molten salts. Finally we use their simple heat capacity characteristics to examine their fragilities in terms of their configuartion space densities of states.

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DIMERIZATION IN THE VAPORS OF THE RARE EARTH BROMIDES: Claus Gietmann[1]; Guido Gigli[2]; *Klaus Hilpert*[1]; [1]Research Centre Jülich, Institute for Mats. in Energy Systems, 52425 Jülich, North Rhine Westfalia Germany; [2]University of Rome, Dept. of Chem., Piazzale Aldo Moro 5, Rome, Toscana 00185 Italy

The vaporization of the rare earth bromides LnBr3 (Ln = La, Ce, Pr, Nd, Gd, Tb, Ho, Er, Tm) was investigated in the temperature range between 770 to 1040 K using Knudsen effusion mass spectrometry. The vapor species LnBr3(g) and (LnBr3)2(g) were identified in the equilibrium vapor over the different samples and their partial pressures determined. The enthalpy and entropy changes of the reactions 2 LnBr3(g) = (LnBr3)2(g) were evaluated according to the third- and second-law methods. The dimensional model by Blander and coworkers was used for the estimation of the unknown thermodynamic functions of LnBr3(g) and (LnBr3)2(g) necessary for the third law evaluation.

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THEORETICAL INVESTIGATION OF THE THERMOCHEMICAL PROPERTIES OF LITHIUM POLYMER ELECTROLYTES USED IN LITHIUM BATTERIES: *L. A. Curtiss*[1]; [1]Argonne National Laboratory, Chem. Division, 9700 S. Cass Ave., Argonne, IL 60439 USA

Ionically conducting lithium polymer electrolytes are used in lithium batteries. In this work we have used theoretical methods to study the ion-ion interactions in these systems. The structures and thermochemical properties of a series of lithium salts such as lithium perchlorate and lithium triflate have been calculated using ab initio molecular orbital theory and density functional theory. The results are used to help understand the role that they play in the conducting properties of the polymer electrolyte Mats.. In addition, the ion-polymer interactions were studied.

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ELECTROCHEMICAL BEHAVIOR OF WELDING FLUXES AND THEIR EFFECT ON WELD COMPOSITION AND MICROSTRUCTURE: *J. Ernesto Indacochea*[1]; [1]University of Illinois at Chicago, Civil and Mats. Eng. (MC246), 842 West Taylor St., Chicago, IL 60607-7023 USA

The influence of the chemical composition of the fluxes on the final chemistry of the weld metal is recognized. However, the mechanisms responsible for the transference of elements from the flux and electrode wire to the weld metal are not completely understood. In the submerged-arc welding (SAW) process, there are four phases (electrode, slag, plasma and weld pool) and five interfaces. The complexity of the system makes it difficult to apply a single model for the whole process. Attempts have been made to develop a thermodynamic equilibrium model but it has led to conflicting conclusions. In our investigation we proposed that in addition to thermochemistry, electrochemical mechanisms play an important role in the element transfer and thus, significantly influence the weld metal composition during the SAW process. In analyzing the electrochemical behavior of the SAW process, the welding wire and the workpiece become either the anode or cathode depending on the polarity used, with the molten flux and the plasm a being the electrolytes. In this presentation the role of electrochemistry, relative to other mechanisms, will be examined more directly from results on welding of alloy steels. Welding was performed by carefully selecting the flux composition and closely controlling the welding parameters. The relative importance of the electrochemical mechanism was enhanced by changing the polarity. The oxygen levels at the weld metal, droplet and electrode tip will be interpreted based on this mechanism. Also the changes in the levels of manganese in the weld metal and of manganese oxide in the slag will be explained based on thermodynamic and electrochemical mechanisms.

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THE SURFACE TENSION DRIVEN BENARD CONVECTION: A UNIVERSAL PHENOMENON: *Pierre Cerisier*[1]; [1]I.U.S.T.I. University of Provence, 5, rue Enrico Fermi, Marseille, Cedex 13 13453 France

A vertical temperature gradient is established between the lower and upper horizontal limiting surfaces of a shallow horizontal liquid layer. The upper surface is free. Beyond a critical temperature gradient the liquid is in convection. Generally the convective structure is composed of a mosaic of convective cells which form a two-dimensional (2D) hexagonal pattern. This phenomenon is considered as the canonical example for fundamental studies: instabilities, heat transfer, wave number selection, diffusion of dye in a liquid, spatio-temporal behavior, structural disorder, turbulence, temporal and spatio-temporal chaos etc. In this paper we consider four of many aspects of these studies: (1) Why a two-dimensional (2D) hexagonal structure? When can a roll or square or hexagonal structure be observed? (2) Influence of the earth's rotation on the structure. (3) Description of the regular and disordered structure. Analogies with 2D crystals. Tools to study such structures. Strong structural analogies and common properties with other 2D structures existing in minerals, metals, botany and zoology. Age of a convective structure and of a living structure. Formal analogy between the birth of a convective cell and that of a living cell. (4) The problem of surface deformation: convex, concave or "volcano"?

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LIMITATION OF THE RULE OF ADDITIVITY IN PREDICTING PHASE TRANSFORMATION DURING CONTINUOUS COOLING: *Yuntian T. Zhu*[1]; Terry C. Lowe[1]; Robert J. Asaro[2]; [1]Los Alamos National Laboratory, Mats. Sci. & Tech., MS G 755, Los Alamos, NM 87545 USA; [2]University of California at San Diego, Dept. of Applied Mech. and Eng. Sci., La Jolla, CA 92093 USA

The rule of additivity was first proposed for predicting the incubation time for nucleation of solid phases during continuous-cooling phase transformations, and has since been widely used for both the nucleation incubation and the entire process of phase transformation. While having been successfully used to calculate the transformed volume fraction during continuous cooling in many steel alloy systems, there is experimental evidence that shows rule of additivity to be invalid for describing the incubation time for nucleation. We have recently demonstrated using classical nucleation theory that the rule of additivity is invalid for the incubation time for nucleation. However, in practice, the relative error caused by using the rule of additivity could be very small in many cases due to the resolution limit of current experimental techniques. Experimental evidences and theoretical basis on the validity and limitations of the Rule of Additivity will be presented, and recommendations and cautions for its application will be given.

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HOMOGENEOUS NUCLEATION: Joseph L. Katz; Dep't. of Chemical Engineering, Johns Hopkins University, Baltimore, MD 21218 USA

Nucleation processes are very poorly understood. Often, one has no idea what constitutes a "nucleus" or what is the nucleating event. However, for vapor condensation processes, a kinetic description of homogeneous nucleation is possible. With it, one can clearly explain why nucleation occurs, and clear up significant confusion about the role of free energy and what is a critical nucleus. This approach can be generalized to cover condensation on surfaces, nucleation with simultaneous chemical reaction, and nucleation in the presence of an anti-nucleator. The limitations of these ideas and of our knowledge for bubble nucleation and for crystal nucleation also will be presented.

MINIATURE STRUCTURES & COMPONENTS UNDER CYCLIC LOADING; FATIGUE & INTERNAL FRICTION: Session II

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee; Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee; ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee,

Program Organizers: H. D. Merchant, Gould Electronics, Inc., Eastlake, OH 44095-4001 USA; Thomas R. Bieler, Michigan State University, Dept. of Mats. Sci. & Mech., East Lansing, MI 48824-1226 USA; James C. Earthman, University of California, Dept. of Chem. Eng. & Mats. Sci., Irvine, CA 92717-2535 USA; M. Wuttig, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20743-2115 USA

Tuesday PM Room: 11B
March 2, 1999 Location: Convention Center

Session Chair: Manfred Wuttig, University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742-2115 USA

2:00 PM INVITED PAPER

MECHANICAL PROPERTIES OF THIN-FILM MATS. EVALUATED FROM AMPLITUDE DEPENDENT INTERNAL FRICTION: *Yoichi Nishino*[1]; [1]Nagoya Institute of Technology, Dept. of Mats. Sci. and Eng., Gokiso-cho, Showa-ku, Nagoya, 466-8555 Japan

In our attempt to study the mechanical properties of thin films, we have examined the amplitude dependence of internal friction in aluminum thin films on silicon substrates. Procedures for analyzing the internal friction data are presented, firstly for evaluating the internal friction in the film separately from the measured internal friction of the composite system, and secondly for converting the internal friction in the film into the plastic strain as a function of effective stress on dislocation motion. The stress-strain responses thus obtained for aluminum films show that plastic strain of the order of 10^{-9} increases nonlinearly with increasing stress. The microflow stress at a constant level of plastic strain is inversely proportional to the film thickness, provided the grain size is larger than the film thickness. The film-thickness effect in the microplastic deformation can be associated with the bowing of dislocation segments whose ends are fixed at the film surface and at the film-substrate interface.

2:40 PM INVITED PAPER

THE REINFORCING EFFECT OF COVERLAYERS ON FATIGUE CRACK GROWTH IN FLEX CIRCUITS: EXPERIMENTS AND ANALYSIS: *Alan T. Zehnder*[1]; [1]Cornell University, Dept. of Theoretical and Applied Mech., Ithaca, NY 14853 USA

In numerous experiments it has been observed that the fatigue life of flex circuits, consisting typically of a thin copper layer on a flexible polyimide substrate, is greatly enhanced when the copper is sandwiched by the addition of a second polyimide layer known as a coverlayer. Through a series of fatigue crack growth experiments, and theoretical and computational fracture mechanics analyses we have shown that the increase in fatigue life can be attributed to the polyimide coverlayer bridging across cracks in copper. In this way the coverlayer provides reinforcement to the copper partially closing cracks, thus reducing the stresses at the tips of cracks in the copper. The reduction in crack tip stresses translates directly to a reduction in the rate of fatigue crack growth, hence an increase in fatigue life. That the polyimide could reinforce the copper in any significant way was at first surprising to us since the polyimide is much more compliant than the copper.

3:10 PM INVITED PAPER

EFFECT OF MATERIAL PROCESSING ON FATIGUE OF FPC ROLLED COPPER FOIL: *Junji Miyake*[1]; Yoshio Kurosawa[1]; Takaaki Hatano[1]; [1]Nippon Mining and Metals Company, Ltd., 10-1, Toranomon 2-Chome, Minato-Ku, Tokyo 105 Japan

The effect of cold rolling reduction and grain size at the final annealing in the rolled copper foil processing on the fatigue property of the foil were examined to attempt further fatigue property improvement. The fatigue property was characterized by the flex and fold fatigue cyclic tests. As the rolling reduction increases, more enhanced cubic texture develops after annealing for the recrystallization. Finer grain before the cold rolling was favorable for obtaining enhanced cubic texture. It is noted that the fatigue property of the foil processed depends on the fatigue cyclic mode. Enhanced cubic texture leads to higher flex fatigue property, but lower fold fatigue property. The system-relationship among materials processing variables(processing), texture evolution(microstructure) and fatigue properties(property) will be discussed.

3:40 PM BREAK

3:50 PM INVITED PAPER

INTERNAL FRICTION IN SHAPE MEMORY AND GIANT MAGNETOSTRICTIVE THIN FILMS: *Manfred Wuttig*[1]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., Stadium Dr., Bldg. 090, Room 1110, College Park, MD 20742-2115 USA

'The internal friction of thin films of NiTi, NiTiPd and CuNiAl shape memory alloy (SMA) poly- and graphoepitaxially grown single crystalline as well as giant magnetostrictive alloy (GMA) Terfenol-D single layer and (approx. 10nm FeTb)/(approx. 10nm (Co)Fe) multilayer films will be reported and analyzed. The friction gives insight into the evolution of the martensitic transformation in SMA films: For substrate constrained SMAs the equilibrium microstructure develops irreversibly with changing temperature, i.e. the microstructure evolution paths for the direct and reverse transformations are different. During the reverse transformation incompatibly stressed austensite forms from the martensite phase. Therefore, a considerable shift of the temperature interval showing maximal internal friction must be expected. Experimental studies on NiTi, NiTiPd and CuNiAI films on Si substrates support the principle thermodynamic conclusions. The magneto-mechanical properties of Terfenol-D thin films and (approx. 10nm FeTb)/(approx. 10nm (Co)Fe) multilayers also show evidence of substrate constraint: a pronounced damping maximum at a magnetic field of about 1 kOe oriented perpendicular to the plane of the film is the result of a magneto-mechanical instability in the Terfenol film. The magneto-mechanical response (of rare earth amorphous/high permeability) nanosized multilayers behaves similarly.

4:20 PM INVITED PAPER

THE EFFECTS OF UNDERFILL ON THE RELIABILITY OF FLIP CHIP SOLDER JOINTS: P. Su[1]; Sven Rzepka[1]; *Matt Korhonen*[1]; C. Y. Li[1]; [1]Cornell University, Dept. of Mats. Sci. and Eng., Ithaca, NY 14853 USA

Thermal fatigue damage of flip chip solder joints is a serious reliability concern, although it usually remains tolerable with the flip chip connections (of smaller chips) to ceramic boards as practiced by IBM over a quarter century by now. However, the recent trend in the microelectronics packaging towards bonding large chips or ceramic modules to organic boards, means a large differential thermal expansion mismatch between the board and the chip or ceramic module. Therefore, to reduce the thermal stresses and strains at solder joints, a polymer underfill is customarily added to fill the cavity between the chip or module and the organic board. This procedure has typically resulted in an increase of the thermal fatigue life by a factor of 10, at least, as compared to the non-underfilled case. In this contribution we first discuss the effects of the underfill to reduce solder joint stresses and strains as well as underfill effects on fatigue crack propagation, as based on a finite element analysis. Secondly, we shall probe the question of the importance of the effects of underfill defects, particularly that of its delamination from the chip side, on the effectiveness of the underfill to increase thermal fatigue life. Finally, we review recent experimental evidence from ther-

mal cycling of actual flip chip modules that appears to be in full support to the predictions of our model.

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TENSION-TENSION FATIGUE OF FREE-STANDING ELECTRON-BEAM-EVAPORATED FILMS OF COPPER AND ALUMINUM: *David T. Read*[1]; [1]National Institute of Standards and Technology, Mats. Reliability Division, 325 Broadway, Boulder, CO 80303-3328 USA

Previously published results of tension-tension fatigue tests of free-standing electron-beam- evaporated films of copper and aluminum from one set of aluminum and one set of copper specimens will be described. Both films were approximately 1 μm thick. The aluminum films, tested with an earlier, manually-operated version of the test device, had significantly poorer fatigue resistance than literature results for bulk sheet material. The copper films had fatigue resistance generally within the range expected for pure bulk copper. The copper films had an apparently abrupt transition from plastic ratchetting to mainly elastic behavior at about 1000 cycles. One copper film, tested at a stress of about 0.4 times the ultimate, endured over 100,000 load cycles without failure; and, its tensile behavior after cycling was indistinguishable from unfatigued specimens. Transmission-electron-microscope observations of the copper specimens were made, and will be described. It will be argued that the lack of correlation between visible geometric defects on the specimens and crack initiation sites indicates that optically invisible microcracks are a key factor in the fatigue failure process in these specimens.

NANOSTRUCTURED HYBRID MATERIALS: Applications of Nanostructured Materials

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Physical Metallurgy Committee

Program Organizers: Gan-Moog Chow, National University of Singapore, Dept. of Mats. Sci., Kent Ridge, Singapore 117600; Yeukuang Hwu, Institute of Physics, Academia Sinica, Nankang, Taipei Taiwan; Sara Majetich, Carnegie Mellon University, Dept. of Phys., Pittsburgh, PA 15213 USA; Luz Martinez-Miranda, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20742-2115 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Tuesday PM Room: 16A
March 2, 1999 Location: Convention Center

Session Chair: Luz Martinez Miranda, University of Maryland, Dept. of Mats. and Nuclear Eng., College Park, MD 20742 USA

2:00 PM INVITED PAPER
FTIR STUDY OF A NANOCRYSTALLINE BaTiO3/CuO COMPOSITE AS NOVEL ELECTRICAL CONDUCTANCE CO2 SENSOR: *Marie Isabelle Baraton*[1]; Lhadi Merhari[2]; Patrick Keller[3]; Karina Zweiacker[3]; Uwe Meyer[3]; [1]LMCTS, Faculty of Sciences, ESA 6015 CNRS, 123 Ave. Albert Thomas, Limoges F-87060 France; [2]Ceramac, Limoges F-87060 France; [3]Fraunhofer Institute for Biomedical Eng., Sensorsystems/Microsystems/Microsystems Dept., Sankt Ingbert Germany

The response of an electronic conductance sensor toward gases is due to the variation of the free carrier concentration induced by changes in the sensor environment. This mechanism, which essentially develops at the interface, reflects the adaptability of the semiconductor surface to the modifications of the surrounding gaseous milieu. From a chemical point of view, these surface changes correspond to adsorption/desorption processes and, therefore, are controlled by the chemical composition of the sensor surface. Hence the first steps toward the improvement of the sensor characteristics (sensitivity, response time, drift ...) are dependent on the comprehension of the chemical phenomena occurring at the gas-sensor interface. Both surface chemical composition and surface reactivity must be controlled to fabricate reproducible sensors. Obviously, this is particularly critical for nanostructured materials because the increase of the specific surface area and of the grain boundary volume implies an increase of the contaminant concentration at the surface, possibly leading to adverse effects. Conversely, a significant increase of the sensitivity can be expected from nanomaterials-based sensors. Fourier transform infrared (FTIR) spectrometry has already proved to be a particularly performant tool for the surface analysis of nanosized materials. In this paper, the technique is applied to the surface study of a BaTiO3/CuO nanocomposite powder which has been successfully used to fabricate electronic conductance sensors for CO2 detection. The chemical composition and the surface reactivity of the composite nanopowder are discussed before studying the interaction of CO2 with the sensor simulated by a pressed pellet of nanopowder. Both the formation of new surface species and the variation of the pellet electrical conductivity upon CO2 adsorption are simultaneously followed in situ by FTIR spectrometry. Then, the correlation between electrical conductivity changes and surface chemical modifications are discussed. This research is supported by the European Commission in the framework of the Brite-EuramIII program (contract BRPR-CT95-0002).

2:30 PM INVITED PAPER
NANOSTRUCTURED GAS SENSORS: *Hong Ming Lin*[1]; [1]Tatung Institute of Technology, Mats. Eng., Tatung Institue of Technologyhmlin, Taipei 104 ROC

For certain industries, the need of monitoring specific gases, such as CO, CO2, SO2, NO2 and H2S, at the specific location is increasing. Gas sensors are getting important and in great demand recently. Metal oxide semiconductor (MOS) type gas sensor is the most popular one because of the quicker response better than other types. The traditional MOS sensors are made of thin film or the Schottky diode. Ideally, the electrical signal generated by the sensing elements is linearly proportionate to the physical changes in the environment. However, in reality, due to the special characteristics and structure of the Mats., a linearly proportional output is uncommon. Nanocrystalline materials with the particle size smaller than 100 nm exhibit many amazing properties, which are not found in conventional Mats.. One of the distinctive features, the main effect of gas sensors, is an extremely large specific surface area. When sintered into porous nanocrystalline film, it will not only retain its gas-sensing surface, but also increase its sensitivity and response efficiency to gases and also decreasing the optimal operation temperature of gas sensors. The promotion of this technology will be very helpful for the industry to develop the nanostructured gas sensors. This study will profoundly to examine the gas-sensing materials of nanostructured WO3, TiO2 and ZnO and the doping concentration effects on sensing properties of the CO, CO2, NO2 and H2S gases.

3:00 PM INVITED PAPER
NANOSTRUCTURED MATERIALS FOR GAS SENSORS: *Virgil Provenzano*[1]; Michel Trudeau[2]; [1]Naval Research Laboratory, Dept. of Mats. Sci. and Tech., Code 6323, 4555 Overlook Ave. SW, Washington, D.C. 20375 USA; [2]Hydro-Quebec, Emerging Technologies, 1800 Boul. Lionel-Boulet, Varennes, Quebec Canada

Nanostructured materials, with their small grain size, large number of grain boundaries and surfaces, together with their strong reactivity with gaseous species, are very interesting materials for gas-reactive applications, including gas sensors. In this paper, the basic characteristics of nanostructured materials, especially those related to gas sensors, will be reviewed. This will be followed by presenting a synopsis of recent results obtained from a joint research and development effort involving the Naval Research Laboratory and the Research Laboratory of Hydro-Quebec on nanostructured gas sensors for naval and dual-use applications. The paper will conclude by briefly considering both the challenges as well as the opportunities offered by nanostructured materials for developing the next generation of gas sensors with significantly improved properties.

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4:00 PM INVITED PAPER

NANOSTRUCTURED MATERIALS FOR THERMAL MANAGEMENT: Jackie Y. Ying[1]; *M. L. Panchula*[1]; [1]MIT, Chem. Eng., Rm 66-544, 25 Ames St., Cambridge, MA 02139 USA

Improved thermal management materials are necessary to increase the efficiency, speed, and reliability of many common devices. In some instances, such as turbine blades and other heat engine components, the material must insulate the underlying metal from the extremely high temperatures of the operating environment. In order to fulfill this protective role the material must have a low thermal conductivity, high damage tolerance, and low oxygen permeability. At the other extreme, the next generation of computer chips will be running faster, and most likely, hotter. In order to reduce the temperature of the chips and prevent premature aging and failure, the chips must be cooled by mounting them on a ceramic with high thermal conductivity, low dielectric losses, and good mechanical properties. While the requirements for the two applications described are very different, nanostructured materials may provide the answer for both cases. This talk will focus on our research on (i) Al2O3/ZrO2 nanocomposite for thermal barrier coating applications and (ii) AlN as a high thermal conductivity substrate.

4:30 PM INVITED PAPER

NANOSTRUCTURED MATERIALS USING ELECTROLYTIC PROCESSES: *Michel L. Trudeau*[1]; [1]Hydro Quebec, IREQ, 1800 Boul. Lionel-Boulet, Varennes, Quebec J3X 1S1 Canada

The last ten years have clearly revealed the technological potentials of nanostructured materials. However the developments in some technological fields are still in desperate need of synthesis processes that can generate large amount of fully dense nanostructured products. This is the case for instance for soft magnetic materials. It has been showed a number of years ago, that by decreasing the average crystal size of soft magnetic materials in the nanometer regime, it is possible to reduce drastically their magnetic losses. However, many studies done in recent years have demonstrated that large-scale soft magnets can not be obtained by the densification of nanostructured powders. On the other hand, a number of works have showed that by controlling the current profile during electrodeposition and through the addition of grain growth of the inhibitors, it is possible to control the nucleation and growth of the deposited materials. Dense samples, with a crystalline size as low as 5 to 7 nm, can thus be synthesized. Compared to other techniques, pulse-electrodeposition has received little attention as a synthesis method for producing large quantities of fully dense nanostructued materials. In this work we will discuss the synthesis of soft magnetic materials, in particular Fe and Fe-riched Fe-Ni alloys obtained by controlling different electropdeposition parameters. We will also present data on the synthesis of new gas sensing nanostructured materials, in which presence of atmospheric pollutants. These examples will demonstrate that electrolytic processes can be the major synthesis technique for large-scale development of dense nanostructured materials.

REACTIVE METALS: General Session II

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: John N. Hryn, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 32, Argonne, IL 60439 USA

Tuesday PM — Room: 5B
March 2, 1999 — Location: Convention Center

Session Chairs: Renato G. Bautista, University of Nevada -- Reno, Mackay School of Mines, Dept. of Chem. and Metall. Eng., Reno, NV 89557-0136 USA; David L. Olson, Colorado Scool of Mines, Dept. of Met. and Mat. Eng., Golden, CO 80401 USA

2:00 PM

THE PRECIPITATION KINETICS OF MAGNESIUM CARBONATE BY USE OF POPULATION BALANCE METHOD: *Zhiang Sun*[1]; Renato G. Bautista[1]; [1]University of Nevada, Reno, Dept. of Chem. and Metall. Eng., Mackay School of Mines, Reno, Nevada 89557-0136 USA

The mathematical modeling of the precipitation process involves the kinetics of the nucleation, growth, and agglomeration of the crystal particle. In this paper, the population balance technique was used to develop the precipitation kinetics of magnesium carbonate. The experimental magnesium carbonate precipitation data from a solution of $MgCl_2$ and Na_2CO_3 reported by Devasahayram and Khangoaukar [Minerals and Metall. Processing, 12, (3), 1995, 157-160] were used in this study. The nucleation rate, B°, can be represented by the equation $B° = 1.768 \times 10^{-9} G^{0.995}$ and the linear growth rate, G, can be represented by the equation $G = -L/t \ln \{n_i/n°\}$. The kinetics is first order, indicating that the growth of the particulate is dependent on size. These results can be used to predict the behavior of continuous precipitation of magnesium carbonate, to control the particle size, and the size distribution of the precipitates.

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DUNITE (OLIVINE) AS A SOURCE OF MAGNESIUM METAL: *Kermit B. Bengtson*[1]; [1]Consultant, 7411 Chippewa Trail, Yucca Valley, CA 92284-4710 USA

Dunite (olivine) occurs as large masses at several surface locations in the world. It analyzes 49.2% MgO and 42.1% SiO_2 together with FeO, MnO, NiO, and CoO. Na_2O, K_2O, and CaO are present in only trace amounts; B and S are absent. Olivine is known to be decomposed by HCl, but under most conditions silica gel is formed, rendering economic separation of the reaction products impreactical. Leaching conditions are reported which recover 92% of the total MgO in a simple exothermic leach of a few minutes' duration as a solution containing 25% dissolved chlorides. The easily filterable silica has a BET surface area of 110 m^2/g. FeO, MnO, NiO, and CoO also dissolve during leaching. Means are described for removing these metals as filterable oxides without the addition of extraneous chemicals. The resulting pure concentrated $MgCl_2$ solution, prepared without discharge of chlorides to the environment, could provide an economically advantageous feed to the dehydration step of a chloride electrolysis for the production of Mg metal.

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RECOVERY OF PURE MnO2 FROM MEDIUM-GRADE LOCAL MANGANESE ORES: *M. B. Morsi*[1]; [1]Central Metall. Research and Development Institute, Pyrometallurgy Lab., P.O. Box 87, Helwan, Cairo Egypt

Local medium-grade manganese ore was blended with different amounts of sodium bisulphate as sulphatizing agent and roasted at temperatures up to 800°C for various periods. The roasted products were subjected to water leaching to recover the soluble manganese sulphate. Maximum recovery of manganese as soluble sulphate achieved under optimum conditions was 98.5%. The leached liquors of manganese sulphate were purified and chemically treated to obtain pure γMnO_2 suitable for dry cell batteries. The purified solution was concentrated to obtain crystallized manganese sulphate and other valuable manganese chemicals. The mechanism and kinetics of the sulphatizing roasting process were suggested.

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KINETICS OF DISSOLUTION OF MOLYBDENUM FROM SECONDARY SOURCES / SCRAPS: *Raj P. Singh*[1]; Michael J. Miller[1]; [1]OSRAM SYLVANIA Products Inc., Research and Development Division, Chem. Development Dept., Hawes St., Towanda, PA 14848 USA

This paper pertains to the kinetics of dissolution of molybdenum from secondary sources containing metallic molybdenum. According to the literature, molybdenum can be dissolved in oxidizing acidic media such as nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). Therefore, two nitric acid containing media, HNO_3-HCl (aqua regia) and HNO_3-H_2SO_4 and one H_2O_2 medium were investigated for this purpose. Reaction in all solvents i.e., HNO_3-HCl, HNO_3-H_2SO_4 and H_2O_2 was of double decomposition type with first order rate kinetics. Although dissolution of only a few molybdenum sources was studied, results can be applied to all type of molybdenum metal secondary source types.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Diamond and Related Coatings

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

Tuesday PM Room: 7B
March 2, 1999 Location: Convention Center

Session Chairs: Jeffrey S. Zabinski, AFRL/MLBT, Nonstructural Mats. Branch, Wright-Patterson AFB, OH 45433-7750 USA; G. Radhakrishnan, The Aerospace Corporation, Mech. and Mats. Tech. Center, Los Angeles, CA USA

2:00 PM INVITED PAPER

DIAMOND AND DIAMOND-LIKE COMPOSITE COATINGS: *J. Narayan*[1]; R. Q. Wei[1]; V. Godbole[1]; A. K. Sharma[1]; [1]North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695-7916 USA

We have improved adhesion and wear of diamond and diamond-like coatings by reducing internal stresses in the films and providing appropriate buffer layers between the film and the substrate. Discontinuous layers of diamond are coated with layers of titanium carbide and aluminum nitride to manage local stresses within the diamond composite films. The adhesion of diamond layers can be improved by providing appropriate interposing layers, which promote strong bonding and graphitization. For diamond-like films, doping with appropriate elements reduces internal stresses and effects mechanical properties to produce functionally gradient Mats.. The adhesion in diamond-like films has also been improved via a buffer layer approach. The structural and medical applications of these novel structures are discussed.

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DEPOSITION, CHARACTERIZATION, AND APPLICATIONS OF METAL-CONTAINING, DIAMOND-LIKE CARBON FILMS: *Gary L. Doll*[1]; [1]Timken Research, Mats. Sci. Dept., Canton, OH 44706 and North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695 USA

Metal-containing diamond-like carbon films can be deposited by DC magnetron sputtering of metals such as titanium, tantalum, or tungsten in a partial atmosphere of argon and a hydrocarbon gas. Films deposited by this technique can be done so at substrate temperatures well below 150ϒC, making them attractive tribological coatings for case carburized steel components. The physical, mechanical, and tribological properties of these coatings are strongly dependent on the deposition conditions. Characterization experiments used in the development of these films are examined, and sever applications where metal-containing diamond-like carbon films are used as tribological coatings are reviewed.

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STRUCTURAL STUDY AND MECHANISMS OF NUCLEATION AND GROWTH OF DIAMOND CRYSTAL GROWN ON SCRATCHED Si(100) SUBSTRATES BY HOT FILAMENT CHEMICAL VAPOR DEPOSITION METHOD: *M. Shamsuzzoha*[1] and Ashok Kumar[2], [1]Department of Metallurgical and Materials Engineering, The University of Alabama, Tuscaloosa, AL 35487; [2]Department of Electrical Engineering, University of South Alabama, Mobile, AL 36688

CVD diamond films deposited on intentionally scratched Si(100) substrates, both free and containing seeded diamond crystals, have been investigated by x-ray diffraction and transmission electron microscopy techniques. Films grown on substrates scratched by a diamond paste and containing seeded diamond crystals appear to distribute on the substrate well and show little evidence of any existing void in the microstructure. The film microstructure is comprised of sparsely populated diamond crystals of faceted morphology embedded in the matrix of graphite and amorphous carbon. Substrates scratched either by diamond paste or by an abrading tool, but containing no seeded crystal, develops a film with a high density of diamond particle and a lower concentration of graphite and amorphous carbon. Diamond particles found in these two types of substrate show difference in their crystal morphology; exhibiting faceted crystal morphology on substrates treated with abrading tool and complex crystal morphology, with ragged surfaces on substrate treated with diamond paste. A distinctive feature found common to diamond of either crystals morphology is the appearances of a zigzag network of constituent twins. The observed structural features of grown diamond crystals are explained on the basis of an existing nucleation and growth mechanism suggested for the freezing of diamond cubic crystals from melt.

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A COMPARISON OF THE CVD DIAMOND NUCLEATION AND GROWTH PROCESSES ON MONOCRSTALLINE COPPER AND SILICON: *J. C. Arnault*[1]; L. Demuynck[1]; L. Constant[1]; C. Speisser[1]; F. Le Normand[1]; [1]Instut de Physique et Chimie de Strasbourg, Groue Surfaces-Interfaces, IPCMS-GSI, UMR 46, Bat 69, 23, Rue du ess, Straabourg 67037 France

CVD diamond synthesis is now well established. However, main diamond applications require a better control of the nucleation and growth mechanisms. At the early states, there is a strong competition between diamond nucleation and carbon phases formation (carbide, graphite, DLC). We will show that the nucleation, studied by electron spectroscopies, SEM, HRTEM and AFM, is quite very different depending on the substrate. On silicon surfaces, a carbide layer of 1-2 nm thick is formed within the first minutes of deposition (1). Furthermore, an etching process by the radical hydrogen occurs, including surface defects, possible preferential sites for the diamond nucleation (2). The part of controlled defects in the nucleation mechanism will be illustrated. On copper, where no carbon miscibility occurs, oriented turbostratic graphite layers are stabilized during the early stages of deposition (3). Some of them are quite new (oignons, polyhedral graphite). (1) F. Le Normand, J. C. Arnault, V. Parasote, L. Fayette, B. Marcus and M. Mermoux, J. Appl. Phys. 80 (3), 1830 (1996); (2) J. C. Arnault, S. Hubert and F. Le Normand, J. Phys. Chem. B 102, 4856 (1998); (3) L. Constant, C. Speisser and F. Le Normand, surf. Sci. 387, 28 (1997)

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THE FORMATION OF SiC INTERFACE AND ITS EFFECTS ON DIAMOND NUCLEATION: *S. T. Lee*[1]; I. Bello[1]; [1]University of Hong Kong, Dept. of Phys. and Mats. Sci., Center of Super-Diamond and Advanced Films, Hong Kong

Three different techniques, namely, bias-enhanced nucleation, ECR-enhanced nucleation and direct ion beam deposition, were employed to enhance diamond nucleation on mirror-polished silicon substrates. It was observed that a SiC layer formed prior to the diamond nucleation by all three nucleation methods. The formation and characteristics of this SiC interface was investigated by high-resolution TEM, selected-area diffraction, and micro-Raman spectroscopy. A theoretical model of diamond/SiC/Si interfacial structure was established with the molecular orbital theory and molecular mechanics/dynamics simulations. The interaction of reactant radicals in the plasma with the substrate surface and the effects of SiC formation on diamond nucleation are discussed.

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RECENT STUDIES ON THE ELECTRON FIELD EMISSION CHARACTERISTICS OF DIAMOND AND DIAMONDLIKE CARBON EMITTERS: H. F. Cheng[1]; I. N. Lin[1]; C. T. Hu[1]; C. Y. Sun[1]; M. Yokoyama[1]

Diamond-like-carbon (DLC) films synthesized by pulsed laser deposition process can be turned-on at low applied field and exhibit large electron field emission capacity, showing great potential for the appli-

cations as electron emitters. The substrate temperature used for preparing the diamond-like carbon (DLC) films by the pulsed laser deposition process pronouncedly modifies the sp3-bonds content and morphology of the DLC films. large proportion of sp3-bonds results in high emission current density (J_e), whereas spherical geometry of the clusters induced low turn-on field (E_0). By contrast, the Au-precoatings reduced the resistance for the electron to transport from the substrate to the DLC clusters without modifying the nature of the DLC clusters. The incorporation of boron-species into the DLC clusters further improved their electron field emission properties via the induction of impurity energy levels Moreover, the post-annealing and the (Ar, N_2 or O_2) post-treatment processes alter the morphology and the proportion of sp3-bonds for the DLC films, resulting in marked improvement on their electron field emission behavior.

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A FINITE ELEMENT MODELING OF THERMAL RESIDUAL STRESSES DURING BRAZING OF DIAMOND TO TUNGSTEN CARBIDES: *R. Torres*[1]; [1]Colorado School of Mines, Dept. of Metall. and Mats. Eng., Advanced Coatings and Surfaces Eng. Laboratory (ACSEL, Golden, CO 80401-1887 USA

The use of polycrystalline diamonds for shear bits used in rock drilling has been expanding rapidly in recent years. These diamond bits are brazed to tungsten carbide using an alloy filler material at high temperatures. During cool down from the braze temperature, the diamond-tungsten carbide joint develops residual stresses. The origin of the stresses is due to the mismatch between the coefficient of thermal expansion, Young's modulus, and Poisson's ratio of the two components involved. Depending on the magnitude and sign of these stresses, delamination or debonding of the joint may occur; or failure may occur either in the diamond or the tungsten carbide piece. The purpose of this investigation is to calculate the thermal residual stresses developed in these joints using a finite element model. Modeling was conducted by independently varying the temperature from which the joint was cooled down (850 to 1150ϒC), the thickness of the braze layer (25 to 150 microns thick), and the diameters of the diamond and tungsten carbide samples (5.2 to 19 mm). The shear, axial, and radial stresses were computed. It was observed that lower brazing temperatures minimize the thermal stresses and also minimize the risk of graphitization of diamond. It was also noted that the thicker the braze layer the greater is the stress relaxation, and maximum relaxation occurred when the braze layer deformed plastically. Optimum parameters for brazing are being developed for a given filler material.

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ADHESION AND WETTING IN DIAMOND AND CARBIDE SYSTEMS: *S. V. Yulyugin*[1]; [1]Institute for Mats. Sci. Problems of National Academy of Sci., 3 Krzhyzhanovsky Str., Kyiv 252142 Ukraine

The common fundamental conformity to natural laws of adhesion and wetting of diamond by liquid metals has been confirmed. These data have been compared with dependencies on adhesion and wetting of carbides. It has been found that high adhesion and wetting of solids by melts is determined mainly by chemical interface interaction. The measurements of the contact angles and adhesion of liquid metals have been complemented by the estimation of the value mechanical strength of metal-solid contact and data of chemical structure of the interface. Experimental studies of liquid sintering of powder composites diamond/melt and diamond//TiC/melt were carried out. The influence of some factors such as the powder granularity, chemical composition and wetting of Cu/Sn/Ti melts and other capillarity characteristics has been studied.

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THE EFFECT OF SURFACE MODIFICATION Al OR Ti FILM ON BOND STRENGTH OF JOINING AIN TO METALS: *Wladyslaw Wlosinki*[1]; [1]Warsaw University of Technology, 85 Narbutta, Warsaw 02-524 Poland

The process conditions, microstructures, reaction products and residual stress as well as their effects on the bond strength of joining surface modified aluminum nitride (AlN) ceramic to metals were studied. Radio frequency sputtering was applied to deposit surface modification aluminum or titanium film on AlN ceramic. Brazing with AgCu19.5Ti3In5 filler and diffusion bonding processes were investigated to join the different surface modified AlN ceramics to Cu and FeNi4 metals. The strength of the joints was improved because the interfacial Al or Ti film improved the wettability of AlN. The maximum bond strength of 127 MPa and 176 MPa could be obtained for brazing surface modified AlN to Cu and to FeNi42 respectively at 1173K for 20 min. And the bond strength of diffusion bonding AlN to Cu joint could be improved further with a functional gradient material(FGM) interlayer coated using plasma spraying.

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STRUCTURE AND PROPERTIES OF PVD-COATINGS BY MEANS OF IMPACT TESTER: Techn. E. Lugscheider, Univ.-Prof. Dr. techn. O. Knotek, Dipl.-Ing. Christian Wolff, Dipl.-Ing. Stephan Berwulf, Materials Science Institute-Aachen University of Technology, RWTH-Aachen, WW, Augustinerbach 4-22, 52062 Aachen, Germany

Machine parts like rolling bearings or gears are stressed during operation in a changing mechanical strain. This causes wear by impacts and wear by rolling which is marked by the so called surface ruin. The appearance of surface fatigue is based upon structural transformation, cracking and cracking-growth processes and ends with the separation of debris particles caused by the above mentioned permanent changing strain . The final stage, which is equivalent to the component failure, is the so called pitting on the technical surface, which is characteristically named surface fatigue. The impact tester is used for detailed research about failure mechanisms of thin films. Statements about the adherence of hard material coatings under dynamic compressive stress can be made using this test method, due to the possibility to simulate some effects of rolling strain. Therefore a hard metal ball strikes with a frequency of up to 50 Hz onto the surface. The altitude stress can be varied to get a detailed evaluation of fatigue strength under reversal strain. Selected hard material coatings were analyzed after testing with the described method applying an impact force of 300 N, 500 N and 700 N. In the framework of this presentation MSIP (Magnetron-Sputter-Ion-Plating) coatings on titanium- and chromium basis were used. The fatigue defects and the results of this study will be discussed depending on structure and morphology of thin films.

SYNTHESIS OF LIGHTWEIGHT METALS III: Titanium - II

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

Tuesday PM Room: 10
March 2, 1999 Location: Convention Center

Session Chairs: C. G. Li, Beijing Institute of Aeronautical Materials (BIAM), Beijing, 100095 China; Oleg Senkov, University of Idaho, IMAP, Moscow, ID 83844-3026 USA

2:00 PM INVITED PAPER

RECENT TITANIUM DEVELOPMENTS - PART I: *Rod Boyer*[1]; Oleg N. Senkov[2]; F.H. (Sam) Froes[2]; [1]Boeing Mats. Technology, P.O. Box 3707, M/S 73-43, Seattle, WA 98124 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Traditionally the majority of titanium used in the USA has been in aerospace applications. However, after "peace broke out" in the world there has been a move to expand non-aerospace use of titanium and its alloys. Recent developments will be discussed including low cost titanium advances.

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RECENT TITANIUM DEVELOPMENTS - PART II : *Rod Boyer*[1]; Oleg N. Senkov[2]; F.H. (Sam) Froes[2]; [1]Boeing Mats. Technology, P.O. Box 3707, M/S 73-43, Seattle, WA 98124 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Traditionally the majority of titanium used in the USA has been in aerospace applications. However, after "peace broke out" in the world there has been a move to expand non-aerospace use of titanium and its alloys. Recent developments will be discussed including low cost titanium advances.

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EXTENSION OF SOLID SOLUBILITY OF Mg IN Ti BY MECHANICAL ALLOYING: *D. Carl Powell*[1]; E.G. Baburaj[2]; F.H. (Sam) Froes[2]; [1](present address) Lockheed-Martin Skunk Works, Mechanical Test Lab, B/633 P/10, 1011 Lockheed Way, Palmdale, CA 93599 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The light weight elements Li, Be, B, Mg, Al, Si, Ca and Sc are the potential alloying components for reducing the density of Ti. Consideration of atomic size, electronegativity and crystal structure of alloying elements, along with material cost suggests Mg as the suitable alloying element for the production of low density Ti alloys. However, alloying Mg with Ti by conventional melting routes is not feasible, since Mg boils, before Ti melts. Therefore, the synthesis of this alloy necessitates the application of non-conventional methods of alloying. In the present study, mechanical alloying has been employed to extent the solid solubility of Mg in Ti. Mechanical milling of the blended elemental powders, using a Spex 8000 mill, showed extension of solid solubility of Mg up to 24 at%. Solubility has been estimated on the basis of changes in lattice parameters of Ti. Contamination by oxygen and nitrogen also increases the lattice parameter of Ti and this effect has been taken into account. The Ti-Mg alloys formed by MA has been consolidated by HIP'ing. Electron microscopic examination of the HIP'ed samples revealed a fine a distribution of Mg particles, in the size range of 10-50 nm, in Ti matrix.

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SYNTHESIS OF COPPER BASE Cu-Ti BINARY ALLOYS BY MECHANOCHEMICAL PROCESSING: *Swati Ghosh*[1]; E.G. Baburaj[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Copper base Cu-Ti alloys have mechanical and electrical properties comparable to those of the precipitation hardenable Cu-Be alloys. Cost effective production of Cu-Ti alloys has the potential to substitute Cu-Be alloys. Present work is an attempt to produce Cu-Ti alloys by mechanochemical processing which involves co-reduction of the $CuCl_2$ and $TiCl_4$ using CaH_2 and Mg at ambient temperatures. The reduction reaction results in the formation of Cu-Ti alloys in a matrix of $CaCl_2$-$MgCl_2$ salt mixture. The salts are leached out to obtain the alloys in the form of fine powder, in a size range of 10 - 500nm. Three copper base alloys containing 2.5, 4.7 and 11.0 % Ti have been synthesized by this process. Detailed examination of the reaction products revealed uniform powder mixtures containing Cu_4Ti and Cu-Ti solid solution. The volume fraction of the intermetallic compound has been found to increase in proportion to the Ti content.

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SYNTHESIS OF Ti-Al INTERMETALLICS BY MECHANOCHEMICAL PROCESSING: *Swati Ghosh*[1]; Dwight Linch[1]; E.G. Baburaj[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Among the light weight intermetallics, Ti_3Al and TiAl are potential candidates for applications in aerospace systems and automobiles because of their low density and high temperature strength properties. In spite of the combination of excellent properties, the use of these intermetallics is limited because of the poor ductility and high cost. Ductility of these alloys is known to increase when the grain size is reduced to nanoscale range. It is difficult to achieve the goals of either fine grain size or the low cost of production of Ti-Al alloys by conventional methods of alloy production. The present work is aimed at the synthesis of Ti-Al intermetallics in the ultrafine powder form and at low cost, by mechanochemical processing. The process involves the co-reduction of chlorides of titanium and aluminum by CaH_2 and Mg by mechanical alloying of the chemicals to induce the reduction reaction resulting in the formation of Ti-Al intermetallics. The use of CaH_2 for the reduction reaction leads to the formation of hydrided intermetallic compounds, Ti_3Al and TiAl. Electron microscopic examination of the intermetallics reveals faceted crystals in the size range of 10 to 300 nm. Further work on consolidation of the alloy powders is in progress.

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DEVELOPMENT AND CHARACTERIZATION OF A TiAl/Ti5Si3 COMPOSITE WITH A SUBMICROCRYSTALLINE STRUCTURE: *O. N. Senkov*[1]; M. Cavusoglu[1]; G. Popescu[2]; M.L. Ovecoglu[3]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Polytechnic University, Dept. of Non-ferrous Metals and Alloys, 313 Independentei Splai, Bucharest, . R - 77206 Romania; [3]Istanbul Technical University, Dept. of Metall. Eng., Faculty of Chem. Metall., Maslak Istanbul, 80626 Turkey

A Ti-47Al-3Cr/Ti_5Si_3 composite with a submicrocrystalline structure was produced by mechanical alloying, thermal treatment and hot isostatic pressing. Blends of elemental and pre-alloyed powders were used for mechanical alloying. Phase reactions and microstructure stability in the mechanically alloyed powders and hot isostatically pressed compacts were studied during heating with the use of DTA, XRD, and TEM. As produced composite consisted of a homogeneously distributed mixture of gamma-TiAl and Ti_5Si_3 grains. The average grain size increased when the hot-isostatic-pressing temperature increased and it was about 200 nm after processing at 1050ϒC. During annealing at 1100ϒC for up to 500h, slow grain growth occurred, however the microstructure of the composite was much more stable than the microstructure of the Ti-47Al-3Cr alloy produced by using identical conditions. Even after annealing for 500 hours, the grains were less than 1 μm in size. Microhardness measurements were also performed which showed high hardness of this material after compaction as well as following annealing.

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SYNTHESIS OF A LOW DENSITY Ti-Mg-Si ALLOY: *O. N. Senkov*[1]; M. Cavusoglu[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

A low density titanium alloy was synthesized from blended elemental powders of Ti, Mg, and Si by mechanical alloying and heating. In some experiments a titanium hydride was used instead of titanium. Phase transformations which occurred in the system during heating were studied with the use of DTA and XRD. The powder annealing was performed in the DTA unit in an argon atmosphere. It was found that during heating of the blended powders some silicon went into solid solution in titanium while most of it exothermically reacted with magnesium at about 500ϒC producing an intermediate phase Mg_2Si. This phase was stable in titanium until the temperature of 950ϒC where it began to decompose into Mg_2Si and liquid Si, and a reaction of the silicon with titanium occurred by formation of a Ti_5Si_3 phase. The third reaction in the system was detected at about 1100ϒC and was due to formation of MgO, so that after annealing at 1100ϒC three stable phases, Ti(Si), Ti_5Si_3, and MgO, were present in the alloy. No decomposition of Ti_5Si_3 phase and formation of Mg_2Si were detected during subsequent cooling and second heating of the alloy. Essentially different phase reactions occurred in the mechanically alloyed powders. The Mg_2Si phase was already formed after heating at 450ϒC, and Ti_5Si_3 phase was detected after heating at 570ϒC. The Mg_2Si decomposed completely at a temperature of 650ϒC with the formation of MgO and Ti_5Si_3. After heating to 1100ϒC, three stable phases, $TiN_{0.3}$, Ti_5Si_3, and MgO, were present in the alloy. A discussion of the results is given.

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CHARACTERIZATION OF A REFRACTORY Ti-Al-Si ALLOY POWDER: *Y. Lytvinenko*[1]; L.D. Kulak[1]; S.A. Firstov[1]; J. Qazi[2]; O.N. Senkov[2]; F.H. (Sam) Froes[2]; [1]Ukrainian Academy of Sci., Institute for Problems of Mats. Sciences, 3 Krzhizhanovsky St., Kiev 252680 Ukraine; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

A novel refractory powdered alloy produced by plasma rotating electrode process (PREP) in the Institute for Problems of Mats. Sci. was characterized. The main composition of the alloy was Ti-6Si-5Al-7Zr (wt.%) and a specific density of 4.6 g/cm^3. The powder particles were spherical in shape with a very small variation in size (the average particle size was about 0.35 mm). They had a dendrite microstructure and consisted of three phases, i.e. disordered alpha-Ti, ordered Ti_3Al and ordered Ti_5Si_3. The Ti_5Si_3 phase was stable during heating up to 1100ϒC while disordering of the Ti_3Al phase occurred near 1000ϒC. Oxidation resistance of the alloy was much superior to conventional titanium alloys and similar to that of a silicon nitride ceramic at temperatures up to 950ϒC. Tensile properties of ingots produced from the alloy were studied at temperatures of 20ϒC to 800ϒC. It was shown that the yield strength and tensile strength of the alloy were higher than those of a gamma-TiAl-based alloy. The fracture toughness of the alloy was about 17-20 MPavm within a temperature range of 20ϒC to 600ϒC. The results showed that the alloy could be potentially used in high-temperature (up to 800ϒC) engine applications.

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FABRICATION OF ADVANCED METAL AND INTERMETALLIC MATRIX COMPOSITES BY CONCURRENT FIBRE WINDING AND LOW PRESSURE PLASMA SPRAYING: *Kyeong Ho Baik*[1]; Patrick S. Grant[1]; Brian Cantor[1]; [1]University of Oxford, Dept. of Mats., Oxford Centre for Advanced Mats. and Composites, Parks Rd., Oxford OX1 3PH UK

A concurrent fibre winding and low pressure plasma spraying process has been developed to manufacture multi-ply fibre reinforced metal and intermetallic matrix composites in a single spraying operation. In this study, Sigma 1140+ SiC fibre reinforced Al, Ti and $MoSi_2$ composites have been manufactured using optimum plasma spraying conditions and evaluated in terms of fibre distribution, matrix/fibre cracking and fibre damage. Fibre breakage occurred during powder spraying because of an increase in fibre axial tensile stress which was caused by the difference of coefficient of thermal expansion between the matrix and the fibre. A reduction in fibre winding tension inhibited fibre breakage, but a significant fibre displacement was observed below fibre winding tension of 10 N. Fibre distribution was mainly affected by surface roughness of predeposit and a close fibre spacing gave rise to interface cracking in SiC/Ti composite and matrix cracking in SiC/$MoSi_2$ composite during secondary consolidation. It was found that the surface roughness was determined by unmelted powder particles and a fine powder spraying was favoured for uniform fibre spacing. However, Ti matrix composite using fine powder (20~50 μm) had poor mechanical properties because of oxidation embrittlement. A reduction of surface roughness was also achieved using low pressure surface rolling during powder spraying and subsequent consolidation. Surface flaws were observed on fibre coating, which degraded tensile strength. The SiC fibres extracted from Ti matrix composites had a large reduction in tensile strength compared with those extracted from Al matrix composites.

THE MARTIN E. GLICKSMAN SYMPOSIUM ON SOLIDIFICATION AND CRYSTAL GROWTH: 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

Tuesday PM — Room: 11A
March 2, 1999 — Location: Convention Center

Session Chairs: Richard H. Hopkins, Northrop Grumman, STC-ESSD, Pittsburgh, PA 15235 USA; Rose Scripa, University of Alabama at Birmingham, Dept. of Mats. and Mech., Birmingham, AL 35294 USA; Krishna Rajan, R.P.I., Troy, NY 12180 USA

2:00 PM INTRODUCTION

2:15 PM INVITED PAPER
PHASE FIELD MODELING OF DENDRITIC GROWTH: *Robert F. Sekerka*[1]; Stanislav Pavlik[1]; [1]Carnegie Mellon University, Dept. of Phys. and Math., Pittsburgh, PA 15213 USA

This talk will focus on modeling of dendritic growth by the phase field model. The model is based on an entropy functional that is the integral of a entropy density assumed to be a function of energy density and a phase field variable (order parameter). The entropy density is that for a homogeneous phase, augmented by a "gradient entropy" correction. By assuming that the local rate of entropy production is positive (local form of second law) subject to the constraints of conservation, we obtain self-consistent coupled linear constitutive laws constitutive laws for energy flux and for phase field evolution. We review the results of dendrites computed from this model at large supercoolings, with emphasis on scaling laws and dendrite morphologies. At large supercoolings, dendrite shapes are found to be more nearly hyperboloidal than paraboloidal. We discuss the modification of the phase field equations needed to account for fluctuations and noise, and the influence of such fluctuations on dendrite sidebranching. This work is supported by the National Sci. Foundation under grant DMR 9634056.

2:45 PM
CSK-1C, TITUS AND ADVANCED TITUS FACILITIES FOR MATS. EXPERIMENTS IN SPACE: *Cestmir Barta*[1]; [1]BBT-Mats. Processing, Doubicka 11, 184 00 Prague 8, Prague Czech Republic

There are described and compared three generations of special facilities for a variety of materials experiments both on Earth and in Space. CSK-1C facility (first generation) was developed in BBT-Mats. Processing Company (BBT) and it is still operational on board MIR Orbital Station for more than 10 years. TITUS facility (second generation) was developed by BBT in cooperation with the Humboldt University (HU) in Berlin for ESA and is also operational on board MIR. Advanced TITUS facility (third generation) is being developed by BBT and HU for the German Aerospace Establishment - Microgravity Users Support Centre (DLR-MUSC) for the International Space Station (ISS Alpha). Advanced TITUS Facility represents a new conception of the space facility based on the "TITUS" currently operational on board MIR and using our experience with CSK-1C facility. It is equipped with a multizone furnace which is expected to be used for metallurgy experiments, glass processing, sublimation techniques, chemical vapour transport, solidification of melts, alloys and glasses, directional solidification of melts,

Bridgeman crystal growth, zone rafination, experiments of undercooling, thermophysical properties measurements (DTA, calorimetry), travelling heater methods, fluid physics, etc.

3:05 PM

ON THE MORPHOLOGY OF A SOLIDIFYING FRONT NEAR AN INSOLUBLE SPHERICAL PARTICLE: *Layachi Hadji*[1]; [1]University of Alabama, Dept. of Mathematics, 345 Gordon Palmer Hall, Box 870350, Tuscaloosa, AL 35487-0350 USA

During the unidirectional solidification of a pure substance, the presence in the melt of an inclusion is known to induce local deformations in the solid-liquid interface. In this work, the interaction between an insoluble spherical particle and a solidifying front is investigated by means of an asymptotic analysis using the width of the gap between the particle and the interface as a small parameter. An axisymmetric model is considered which accounts for (1) the thermal conductivities of the melt and the particle, (2) the disjoining pressure in the thin melt film that is sandwiched between the particle and the moving front and (3) the hydrostatic pressure in the film. An evolution equation for the deformed front is derived whose solution yields a steady state equation for the interfacial shape. The dependence of this solution, which is valid only locally, on the factors listed above is investigated. A linear stability analysis is also undertaken to isolate the stable morphology.

3:25 PM

DENSITY AND DISTRIBUTION OF POROSITY IN ALUMINUM ALLOYS: *C. J. Paradies*[1]; J. Wolla[2]; [1]Northrop Grumman, AS&T, M/S A01-26, Bethpage, NY 11731 USA; [2]Naval Research Laboratory, Code 6323, Bldg. 42, 4555 Overlook Ave., SW, Washington, D.C. 20375 USA

Hydrogen was intentionally introduced into several different aluminum alloys to evaluate the size, shape, density and distribution of the resulting hydrogen porosity that developed during solidification. Directional solidification experiments were performed to reveal the effect of processing conditions on the pore formation. The final pore size appears to be defined by the hydrostatic pressure and the hydrogen concentration in the melt. Bubbles nucleate at heterogeneous sites and grow by diffusion of hydrogen from the melt to the bubbles. The shape and distribution of the resulting pores depends upon both the microstructure of the solid and the time and location that the bubble nucleated within the solid structure. The density of pores probably depended upon a combination of factors including the density of nucleation sites, the local supersaturation, the local solidification time and the number of bubbles that found their way to a free surface. The results of the experiments are analyzed and discussed.

3:45 PM BREAK

4:05 PM INVITED PAPER

SYNTHESIS OF ZIRCONIA-8WT.% YTTRIA NANOCOMPOSITE BY PLASMA SPRAY: *Ramasis Goswami*[1]; Guo-Xiang Wang[1]; Sanjay Sampath[1]; Herbert Herman[1]; [1]SUNY at Stony Brook, Dept. of Mats. Sci., Stony Brook, NY 11794-2275 USA

Zirconia-8wt.% Yttria has been plasma sprayed to a thickness of 2mm on a steel substrate kept at two different temperatures. A fine dispersion of monoclinic phase with a size range of 5-20nm has been observed to be embedded at the cell boundary of metastable tetragonal (t') phase when it is deposited on a steel substrate held at 100ϒC. At a higher substrate temperature (450ϒC) an alternate layer of thick metastable tetragonal and very thin monoclinic phases was observed. The microstructural transition observed in this experiment has been explained by the use solute trapping models.

4:35 PM

DYNAMIC CALCULATIONS FOR PARTICLE PUSHING: *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Faculty of Sciences, 1552 Azara St., Posadas, Misiones 3300 Argentina

The interaction of particles with a solidifying interface is studied dynamically employing mathematical modeling. The two main physical forces involved which are the drag and pushing force, are calculated for different particle and interface morphologies. Both forces are integrated analytically and the dynamic equation resulting from application of Newton's law is integrated numerically. The pushing forces come from the Lifshitz-Van der Waals interactions. The results show stable steady state interactions for flat interface and spherical or flat particles. However for curved interface shapes the steady state is reached for values of the interface velocity and particle radius which depend on the curvature of the interface. In the present report the results of the calculations are presented and the conditions for pushing are discussed.

4:55 PM INVITED PAPER

HETEROGENEOUS NUCLEATION ON CATALYTIC PARTICLES: *P. G. Höckel*[1]; G. Wilde[1]; J. H. Perpezko[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1500 Eng. Dr., Madison, WI 53706 USA

Crystallization studies on undercooled droplets and bulk samples offer an effective method to examine nucleation kinetics due to catalysis by primary phases and incorporated particles. Droplet studies, where the interface between primary solid and the undercooled liquid acts as the catalytic substrate for heterogeneous nucleation, can provide information on nucleation behavior which is crucial for solidification modeling. In order to identify nucleation mechanisms, controlled thermal cycles have been performed in eutectic and peritectic systems. The results suggest some deficiencies in the spherical cap model of catalysis and identify new directions for kinetics analysis and modeling. Experiments on bulk samples of Cu and Ni containing inoculant particles yielded various amounts of undercooling, exceeding 200K for some composite systems following fluxing with a glass slag. The results indicate that many crystalline particles can be relatively inert in nucleation catalysis. The support of NSF (DMR-9712523) and NASA (NAG8-1278) is gratefully acknowledged.

5:15 PM INVITED PAPER

DIRECT NUMERICAL SIMULATION OF DENDRITIC MICROSTRUCTURES: *N. Provatas*[1]; N. Goldenfield[1]; *J. A. Dantzig*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. and Indust. Eng., Dept. of Phys., Urbana-Champaign, IL 61801 USA

Prof.Glicksman has made invaluable contributions to the understanding of pattern selection during dendritic solidification. His experiments have served to guide the theoretical development of the industrially important problem. In this talk, we describe recent computations using phase-field models to directly simulate dendritic growth. In this method, the liquid-solid interface is modeled as a diffuse region whose thickness is characterized by an order parameter, known as the phase field. One of the difficulties encountered when applying the phase field method is the conflicting requirements of high resolution needed to successfully capture the physical phenomena at the interface, and the simultaneous need to fully resolve the diffusion field ahead of the advancing front. We employ an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion field. Examples are given comparing calculations to experimental results from Prof. Glicksman's experiments.

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

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

Wednesday AM Room: 15B
March 3, 1999 Location: Convention Center

Session Chair: Reginald W. Smith, Queen's University, Dept. of Mats. and Metall. Eng., Kingston K7L 3N Canada

8:30 AM

TEMPUS CONTAINERLESS PROCESSING FACILITY FOR SPACE STATION - PRELIMINARY DESIGN AND SPECIFICATION: *J. Piller*[1]; A. Siedel[1]; M. Stauber[1]; W. Dreier[2]; [1]Dornier Gambh, Daimler-Benz Aerospace, Friedrichshafen D-88039 Germany; [2]Deutsches Zentrum fur Luft-und Raumfahrt (DLR), Bonn D-53227 Germany

The containerless processing facility TEMPUS was successfully used for electromagnetic levitation experiments during the MSL-1 Spacelab mission in 1997. Scientific goals have been the study of nucleation statistics and solidification speeds in undercooled melts, and the determination of thermophysical properties of the melt above and below the melting point. For future research in this promising field the accommodation of an Advanced TEMPUS facility on board of the International Space Station is under discussion. The results of a design study are presented to show that the well-proven technology of TEMPUS Spacelab can be transferred to a Space Station facility. In particular to better serve the individual and often interfering experiment requirements exchangeable experiment containers shall be made available for processing which are equipped with a set of samples, coil system, windows and other experiment specific devices.

8:50 AM

SCIENCE OPPORTUNITIES OF THE MATERIALS SCIENCE LABORATORY (MSL): *A. Lundstrom*[1]; P. Behrmann[1]; H. Lenski[2]; C. Cordelle[3]; [1]ESA/ESTEC, P.O. Box 299, AG, Noordwijk NL-2200 The Netherlands; [2]DASA-Dornier, Friedrichshafen Germany; [3]SNECMA-SEP, Villaroch France

The European contribution to the microgravity payloads of International Space Station includes the Materials Science Laboratory (MSL), which is part of ESA Microgravity Facilities for Columbus programme. MSL is a multi-user facility intended to support four areas of microgravity research: solidification physics, Bridgeman crystal growth crystal growth by zone processing and measurement of thermo physical properties. MSL will be developed in two versions: one to be accommodated in NASA's Materials Science Research Facility in the US-module and one self-standing facility for Columbus. The MSL provides a unique scientific flexibility by its concept of exchangeable furnace inserts and will by this concept support different branches of high temperature research. So the Low Gradient Furnace (LGF) insert is optimised for crystal growth of semiconductors and electronic materials under restricted, well-controlled thermal gradients. The Solidification and Quenching Furnace (SQF) is mainly intended for metallurgical solidification under strong gradients with the possibility of quenching the solidification interface at the end of processing. The furnace insert concept also supports incorporation of furnaces from international partners or external customers into MSL; NASA's Quench Module Insert (QMI) and Diffusion Module Insert (DMI) as well as the German Float-zone furnace with Magnetic Field (FMF) are presently in the planning. For the experiment cartridge, various diagnostics and stimuli are offered, including high-resolution thermocouples, Peltier pulsing, shear cell activation for diffusion experiments and reservoir heating. The scientific performance of MSL is further enhanced by the concept of experiment-dedicated electronics, which allows incorporation diagnostics targeted to individual experiments. In this context ESA is studying Seebeck voltage and resistance measurement and ultrasonic measurement of the solidification velocity, but this interface will also allow for e g video observation or PI-developed electronics.

9:10 AM

THE EUROPEAN FLUID SCIENCE LABORATORY (FSL) A HIGHLY FLEXIBLE, MODULAR MULTI-USER FACILITY: *J. R. Becker*[1]; H. Mundorf[1]; Cestmir Barta[1]; [1]ESA/ESTEC, TOS-MMG, P.O. Box 299, AG, Noordwijk NL-2200 The Netherlands

The FSL is one of the large multi-user facilities being developed under ESA's Microgravity Facilities for Columbus (MFC) Programme. Extending ESA's earlier fluid science research programs, the FSL will allow, among others, for the investigation of areas such as flows and induced instabilities, diffusive instabilities, interfacial tension and adsorbtion mechanisms, mechanisms of boiling, critical point phenomena, crystal growth and directional solidification within transparent media. Owing to its adaptable diagnostic tools and its modularity on several levels, complementary science areas such as colloid and aerosol physics, particle agglomeration and plasma crystal physics are envisaged. FSL's most important diagnostic tools are four different types of interferometers. An electronic speckle pattern interferometer (ESPI), a differential (shearing) interferometer and a holographic interferometer for measurements on transparent media and in addition an ESPI to perform surface measurements. For the first time in the history of microgravity facilities these diagnostics tools will become available on one facility by converting one type of interferometer into another by switching of optical components. Each type of interferometer represents unique features in terms of spatial resolution, time resolution and dynamic range in such a way, that the scientist will be able to select the best choice with respect to his particular experiment. Compared with earlier facilities, such as the Bubble, Drop and Particle Unit (BDPU), flown on Spacelab, FSL offers a greatly enhanced flexibility by having a larger exchangable experimental volume (the 'Experiment Container (EC)'), intelligent interfaces and modularity down to that level, improved control capabilities and extended research autonomy. The large experimental volume together with the intelligent interfaces will even allow for the application of dedicated three-dimensional measurement tools. The introduction of electronic imaging improves the telescience performance significantly.

9:30 AM

CSK-1C, TITUS AND ADVANCED TITUS FACILITIES FOR MATERIALS EXPERIMENTS IN SPACE: *Cestmir Barta*[1]; [1]BBT-Materials Processing, Doubicka 11, Prague 8, Czech Republic 184 00 Russia

There are described and compared three generations of special facilities for a variety of materials experiments both on Earth and in Space. CSK-1C facility (first generation) was developed in BBT-Materials Processing company (BBT) and it is still operational on board MIR Orbital Station for more than 10 years. TITUS facility (second generation) was developed by BBT in cooperation with the Humboldt University (HU) in Berlin for ESA and is also operational on board MIR. Advanced TITUS facility (third generation) is being developed now by BBT and HU for the German Aerospace Establishment - Microgravity Users Support Centre (DLR-MUSC) for the International Space Station (ISS Alpha). Advanced TITUS Facility represents a new conception of the space facility based on the "TITUS" currently operational on board MIR and using our experience with CSK-1C facility. It is equipped with

a multizone furnace which is expected to be used for metallurgical experiments, glass processing, sublimation techniques, chemical vapour transport, solidification of melts, alloys and glasses, directional solidification of melts, Bridgeman crystal growth, zone rafination, experiments of undercooling, thermophysical properties measurements (DTA, calorimetry), travelling heater methods, fluid physics, etc.

9:50 AM BREAK

10:10 AM

QUALITATIVE AND STATISTICAL ANALYSIS OF CELLULAR ARRAYS IN DIRECTIONAL SOLIDIFIED SUCCIONITRILE-ACETONE: *B. Kauerauf*[1]; G. Zimmermann[1]; S. Rex[1]; [1]Access e.V., Intzestr. 5, Aachen 52072 Germany

Two-dimensional cellular arrays which occurred during directional solidification in a Bridgman configuration under microgravity conditions in the transparent alloy succinonitrile-acetone were evaluated with respect to the time dependent behaviour of the pattern. Images of the cellular patterns observed in-situ in top view from the melt by an endoscope optic show continuous restructuring of the cellular patterns accompanied by arising and elimination of cells on a time-scale of several minutes. Contrary to this qualitative analysis a statistical quantitative evaluation of pattern characteristics shows no significant changes in the primary spacing of the cells or in the distribution of the cell sizes. Therefore, it can be concluded that during the directional solidification a steady-state for the patterns was reached without changes in quantitative characteristics but with a continuous rearrangement of the structure.

10:30 AM

NUMERICAL SIMULATION OF MONOTECTIC SOLIDIFICATION USING PHASE-FIELD MODEL: *Y. Arikawa*[1]; J. B. Andrews[1]; S. R. Coriell[2]; W. F. Mitchell[2]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., Birmingham, AL 35294 USA; [2]National Institute of Standards and Technology, Gaithersburg, MD USA

A numerical simulation scheme using a phase-field formulation has been constructed to model a directional solidification front in monotectic systems. The scheme has been tailored to suit the atypical characteristics of the underlying physical system, i.e. one of the product phases in the monotectic system is still liquid after the reaction. This atypical characteristics suggest a strong asymmetry in the production of latent heat at the interface which should have a impact on the microscopic interface curvature. Although the liquid-liquid interface and accompanying Marangoni flow at the interface are expected to play a role in determining the interface shape, inclusion of the effect of fluid motion into the model is still underway. Numerical results are compared to experimental results obtained for succinonitrile-glycerol system.

10:50 AM

LSW, LSEM AND MEAN FIELD THEORY DISTRIBUTIONS FOR Fe-Cu LPS MICROGRAVITY RESULTS: *J. Naser*[1]; Y. He[1]; S. Ye[1]; A. K. Kuruvilla[2]; J. E. Smith[1]; [1]University of Alabama in Huntsville, Consortium for Materials Development in Space and Dept. of Chemical & Materials Engineering, Huntsville, AL 35899 USA; [2]Marshall Space Flight Center, IITRI/MRF, Bldg. 4618, MSFC, AL 35812 USA

Testing coarsening theories against experimental LPS results is complicated by the gravity driven convection in unit gravity. The absence of gravitational force in microgravity represents a unique environment for testing coarsening theories. Lifshitz Slyozov and Wagner theory (LSW) and the modified Lifshitz Slyozov Encounter Modified theory (LSEM) distributions were compared against experimental results obtained from microgravity processed liquid phase sintered Fe-Cu samples. The agreement between the LSEM and the experimentally measured distributions were far better than using LSW theory. The mean Field theory did not fit the experimental distribution as well as the LSEM, but predicted a better cut off than the LSW distribution. The reason for this disparity resulted from the fact that Fe-Cu had agglomerated particles and copper pools. Results for several systems, and proposed theory modification will be presented.

11:10 AM

THE THERMOPHYSICAL PROPERTIES OF Zr-Nb-Ni-Cu-Al AND Zr-Ti-Ni-Cu GLASS FORMING ALLOYS: *Charles G. Hays*[1]; Jan Schroers; William L. Johnson[1]; [1]California Institute of Technology, Dept. of Mat. Sci. 138-78, Pasadena, CA 91125 USA

The thermophysical properties of the recently discovered bulk metallic glasses are an area of both scientific and technological interest. To further our understanding of the glass forming ability of these alloys a ground-based examination of the undercooling behavior in these alloy systems was implemented using the Electrostatic Levitation (ESL) technique. This paper presents results of undercooling and step modulation calorimetry measurements conducted on two bulk glass forming alloy systems: Zr-Nb-Ni-Cu-Al and Zr-Ti-Ni-Cu. In each system large undercooling levels of the order of 225K were observed. The undercooling was found to be dependent on the degree of overheating above the liquidus temperature. Discontinuous levels of undercooling were observed for overheats of 300K. The properties of the ESL processed specimens were examined by x-ray diffraction, electron- and optical-microscopy, and thermal analysis. The ESL results are compared with the results from the MSL-1 space shuttle flight experiments. The experimental results are discussed in relation to the predictions of classical nucleation theory and are related to the known features of the multicomponent phase diagrams.

11:30 AM

CHARACTERIZATION OF A CERAMIC SPRING FOR USE IN SEMICONDUCTOR CRYSTAL GROWTH IN MICROGRAVITY: *Monica L. Kaforey*[1]; Christopher W. Deeb[1]; David H. Matthiesen[1]; [1]Case Western Reserve University, Dept. Mat. Sci. & Eng., 10900 Euclid Ave., White 330, Cleveland, OH 44106 USA

Traditionally, semiconductor crystals have been grown in microgravity to reduce convection in the hopes of achieving diffusion controlled growth. In many of these experiments, a ceramic spring was used to prevent free surface formation to avoid Marangoni convection. Pyrolytic boron nitride (PBN) leaf springs will withstand the temperature and atmosphere requirements for the growth of semiconductor materials such as germanium and gallium arsenide. A theoretical model based on a simply supported section cut from a cylinder was developed to predict the spring constant of a stack of PBN leaf springs. Experiments were done, based upon a statistically rigorous design of experiments (DOE), in which the spring constant was measured. The experimental data was empirically fit with a full quadratic model in terms of 4 variables: spring width, spring thickness, spring radius, and the number of springs in the stack. The empirically fit model was compared with the theoretically developed model.

11:50 AM

AN OVERVIEW OF THE ELECTROSTATIC LEVITATION FACILITY AT NASA'S MARSHALL SPACE FLIGHT CENTER: *Jan R. Rogers*[1]; Michael B. Robinson[1]; Larry Savage[1]; Wolfgang Soellner[1]; [1]NASA/MSFC, ES76, Marshall Space Flight Center, Huntsville, AL 35812 USA

Containerless processing represents an important area of research in microgravity materials science. This method provides access to the metastable state of an undercooled melt. Containerless processing provides a high-purity environment for the study of reactive, high-temperature materials. Reduced gravity affords several benefits for containerless processing, for example greatly reduced positioning forces are required and therefore samples of greater mass can be studied. Additionally in reduced gravity, larger specimens will maintain spherical shape which will facilitate modeling efforts. Space Systems/LORAL developed an Electrostatic Containerless Processing System (ESCAPES) as a materials science research tool for investigations of refractory solids and melts. ESCAPES is designed for the investigation of thermophysical properties, phase equilibria, metastable phase formation, undercooling and nucleation, time-temperature-transformation diagrams and other aspects of materials processing. These capabilities are critical to the research programs of several Principal Investigators supported by the Microgravity Materials Science Program of NASA. NASA's Marshall Space Flight Center (MSFC) recently acquired the ESCAPES system from LORAL. MSFC is now developing a levitation facility to provide a critical resource to the microgravity materials science re-

search community to continue and enhance ground-based research in the support of the development of flight experiments during the transition to Space Station.

ABATEMENT OF GREENHOUSE GAS EMISSIONS IN THE METALLURGICAL & MATERIALS PROCESS INDUSTRY

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee

Program Organizers: C. Lupis, MIT, Room 13-5114, Cambridge, MA 02139 USA; V. I. Lakshmanan, Ortech Corporation, Mississauga, Ontario L5K1B3k Canada

Wednesday AM Room: 1B
March 3, 1999 Location: Convention Center

Session Chairs: Claude Lupis, MIT, Materials Science and Engineering, Cambridge, MA 02139 USA; V. I. Lakshmanan, Mississauga, Ontario L5K1B3k Canada

8:30 AM INTRODUCTION

8:40 AM
CO2 REDUCTION STRATEGIES IN THE BASIC METALS INDUSTRY: A SYSTEMS APPROACH: *Dolf J. Gielen*[1]; *Antonius W. N. VanDril*[1]; [1]ECN, Beleidsstudies, P.O. Box 1, Westerduinweg, Petten, Noord-Holland 1755 ZG Netherlands

To find strategies for reducing greenhouse gas emissions in the basic metals industry, a study is made for the Western European steel and aluminium market up to 2020. Taken into account are competition of cheap energy locations, alternative policy scenarios for CO2/energy taxes, energy efficiency improvement for existing technologies, new technologies for iron and steelmaking, additional CO2 removal technologies, possibilities and limitations for recycling, and improved materials efficiency in product manufacturing. These strategies are simultaneously analysed within a MARKAL model approach that includes the energy supply system. Results state that a factor two reduction of CO2 is readily attainable. Favourable solutions for steel include DRI combined with EAF and new Cyclone converter technology. CO2 removal and the CO2 emission for electricity generation are essential factors. The limitation for recycling is scrap availability, rather than quality. For aluminium, scrap availability and the electricity emission factor are essential limitations. However, taking all options into account, greenhouse gas reductions with a factor 5 to 10 are attainable for the European basic metals industry.

9:05 AM
CO2 EMISSIONS AND THE STEEL INDUSTRY'S POSSIBLE RESPONSES TO THE GREENHOUSE EFFECT: *Jean-Pierre Birat*[1]; Yann de Lassat[2]; Michel Schneider[2]; Michel Jeanneau[2]; [1]IRSID, BP 30320, Maizieres-les-Metz 57283 France; [2]Usinor, Cours Valmy, Paris-La-Defense France

The Steel Industry, is responsible in France for 6% of CO_2 emissions, through a small number of large sources. The carbon consumption of the Steel Industry ends up eventually as CO_2 in the atmosphere, to a level which is directly proportional to the amount of steel production. In 1989, the French Steel Industry used 423kg of carbon per ton of rolled steel, which means emissions of 1550kg of CO_2 per ton. Globally, for a 16.9Mt production, this adds up to 26.3Mt CO_2; the energy savings policy, which has been rigourously enforced since before the first energy crisis, has led in France to a reduction of carbon consumption from 1100kg/t of steel to 423kg/t over this period of 20 years. This result has been achieved thanks to radical modifications of process routes and to better process control: increase in the size of reactors such as the blast furnace or the basic oxygen converter, general introduction of strand sintering and continuous casting, increase in the number of electric furnace steelshops are typical examples of such changes; the carbon which the Steel Industry uses today is close to the minimum amount that is required by process thermodynamics and technologies. An exhaustive assessment of steel production processes, either existing, under development or still in the research field, has been carried out in terms of CO_2 emissions, using process models available at IRSID. Industrial realism has been introduced by taking into account the magnitude of available sources of iron, by estimating time for process development when a process route is not readily available in the industry, and by balancing various possible processes against the quality needs of the product mix that a modern steel production company has to make available to customers. The major conclusions are as follows: energy savings should be continued, especially in countries where the actions outlined before have not yet all been made (step 1); another step would consist in substituting iron ore by scrap, within the limits set by product quality and by the availability of recycled steel. This should be easier to carry out in developed countries, where scrap should become more readily available, due to used metal accumulation and to government regulations that foster materials recycling. Typically, scrap use could go up from 35 to more than 50% of source iron in a country like France (step 2); beyond that, other solutions are possible, although they are not cost effective today. The use of plasma torches in the blast furnace on a year round operation and also natural gas or hydrogen prereduction are typical examples (step 3).

9:30 AM
THE ABATEMENT OF CO2-EMISSIONS IN THE PRODUCTION OF FERROALLOYS: *Tor Lindstad*[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2B, Trondheim N-7034 Norway

Specific CO2-emissions are given for the production of silicon-metal, ferrosilicon, silicomanganese, ferromanganese and ferrochrome. Emissions come from the mining of raw materials and reduction materials, from coke production and generation of electric energy. CO2-emissions can be abated by direct and by indirect methods. A direct method is to replace fossil carbon as reduction material wholly or partially by endogenous carbon (biocarbon, charcoal). Another possibility is to produce metals by electrolysis instead of the present electric smelting furnace. Indirectly CO2-emissions can be reduced by energy recovery in the off-gas, thus saving coal, oil or natural gas for heat generation. Some reduction of global CO2-emissions can be obtained by giving credit for the use of silica dust partially replacing cement in concrete. In addition to an overview of possible methods, costs will be discussed.

9:55 AM
ABATEMENT OF SOX EMISSIONS IN RELATION WITH BASIC METAL SULFIDES PROCESSING: *Ibrahim Gaballah*[1]; Eric Allain[2]; Ndue Kanari[1]; Kawan Malau[3]; [1]INPL - CNRS, ENSG-LEM, 2 Avenue de la foret de Haye, BP 3, Rue du Doyen M. Roubault, BP 40, Vanduvre F - 54501 France; [2]University of Missouri Rolla, Dept. of Metall. Eng., 215, Fulton Hall, Rolla, MO 654098 USA; [3]Mineral Technology Development Center, Jalan Jenderal Sudiman 623, Bandung Indonesia

About forty percent of the SOx emissions are generated during the processing of metal sulfides of Cu, Pb, Zn, Ni, etc. Although new technologies for metal sulfide's smelting allow the production of H2SO4 with a reasonable cost and decrease the emissions of sulfur oxides, the generated quantity of SOx is considered as a serious threat to environment. This paper suggests alternatives for low temperature processing of complex sulfide concentrates "CSC" under neutral, reducing and chlorinating atmospheres or their combination. The neutral and reducing treatments, at T > 400ϒC, allow the decomposition of pyrite and the partial recovery of sulfur as Sϒ; and H2S. At 300ϒC, the chlorinating treatment of CSO is fast, exothermic and allows the separation of FeCl3 from the valuable elements: chlorides. It also generates SyClx that can be either reduced by H2 or hydrolyzed in presence of sodium sulfide. The recovery rate of elemental sulfur with respect to the sulfur input exceeds 90 percent.

10:20 AM BREAK

10:30 AM

MORE PROGRESS IN IMPLEMENTING THE ALUMINUM TECHNOLOGY ROADMAP: *Jack Eisenhauer*[1]; *Henry Kenchington*[2]; John Green[3]; [1]Energetics, Inc., 7164 Gateway Dr., Columbia, MD 21046 USA; [2]U.S. Department of Energy, Office of Industrial Technologies, 1000 Independence Ave., Washington, D.C. 20585 USA; [3]The Aluminum Association, 900 19th St., Washington, D.C. 20006 USA

Leaders in the U.S. aluminum industry have recognized that success in the competitive global marketplace will depend on new business strategies that leverage R&D investments across industry and government. To this end, the aluminum industry has entered into a partnership with the U.S. Department of Energy's (DOE's) Office of Industrial Technologies (OIT) to address issues of competitiveness, the environment, and energy use from a long-term perspective. The Aluminum Industry Technology Roadmap published in 1997 has laid out concrete technology strategies for achieving industry goals and has established a focused research and development agenda for aluminum. A follow-on technology roadmap on inert anodes addresses one of the highest priorities in the first roadmap, the development of inert (non-consumable) anodes that can reduce emissions of CO_2 and perfluorocarbons from smelting. A separate technology roadmap focusing on R&D that will facilitate the use of aluminum in the automotive market is currently being developed. This effort complements technology roadmap activities undertaken by the U.S. automotive industry as part of the Partnership for a New Generation of Vehicles. As a result of the aluminum industry's roadmapping efforts, a number of collaborative R&D projects have been launched and are making considerable progress.

10:55 AM

PRIMARY ALUMINUM PRODUCTION: PROJECTED GREENHOUSE GAS EMISSIONS AND THE COSTS OF CLIMATE CHANGE POLICY: *Jochen Harnisch*[1]; Ian S. Wing[1]; Ron G. Prinn[1]; Henry D. Jacoby[1]; [1]Massachusetts Institute of Technology, Joint Program on the Science and Policy of Global Change, 77 Massachusetts Ave., Building E40-390, Cambridge, MA 02139-4307 USA

Climate policy may gain significant influence on investment decisions made associated with the production of primary aluminum. This work is intended to demonstrate the potential of an integrated analysis for an improved understanding of the environmental effectiveness and economic consequences of different climate policies. In this study we first compare atmospheric observations to the available emission estimates for CF_4 and C_2F_6 for the baseline years 1990 and 1995. We then present projections for regional emissions of PFCs from the aluminum industry under different climate change policy scenarios using the MIT Emission Projection and Policy Analysis (EPPA) energy-economy model. Finally, we use CO_2 abatement costs to analyze the environmental effectiveness of investments into emission reductions of PFCs in the context of the Kyoto-protocol.

11:20 AM

A BIOMIMETIC APPROACH TO CO2 REMEDIATION: *Gillian M. Bond*[1]; Gerald Egeland[1]; Donald K. Brandvold[2]; Margaret Gail Medina[1]; John Stringer[3]; [1]New Mexico Tech, Materials & Metallurgical Engineering, Campus Station, Socorro, NM 87801 USA; [2]New Mexico Tech, Chemistry, Campus Station, Socorro, NM 87801 USA; [3]Electric Power Research Institute, 3412 Hillview Ave., Palo Alto, CA 94304 USA

Public concerns over the "greenhouse effect" have resulted in research efforts into various possible approaches to CO_2 remediation. Fixation of CO_2 into calcium carbonate is potentially an ideal way of disposing of the large quantities of CO_2 produced by many industrial processes. It is a proven method in geological terms, with an environmentally friendly end product. Early in the earth's geological history, the atmosphere contained around three orders of magnitude more CO_2 than it does now. This level gradually decreased as large quantities of carbon became locked up in various reservoirs, of which carbonate minerals, notably limestone and chalk, comprise a major example. Much of the deposition into carbonate reservoirs occurred biologically, through the action of a variety of marine organisms. The problem with such an approach to commercial CO_2 remediation, of course, is one of rate. We have followed a biomimetic approach to accelerating CO_2 sequestration; we have examined the chemistry of CO_2 fixation into calcium carbonate in aqueous solution, and the rate-limiting steps, and then considered what lessons we could learn from biological systems in terms of how to accelerate those steps. As a result, we are studying an enzymatic approach to accelerated CO_2 sequestration, in which carbonic anhydrase is used to catalyze the hydration of CO_2.

11:40 AM

HIGH-TEMPERATURE SOLAR THERMOCHEMISTRY FOR GREENHOUSE GAS MITIGATION IN THE EXTRACTIVE METALLURGICAL INDUSTRY: *Jean P. Murray*[1]; Aldo Steinfeld[2]; [1]Colorado School of Mines, Engineering, Golden, CO 80401 USA; [2]Paul Scherrer Institute, Solar Process Technology, Villigen PSI 5232 Switzerland

The extractive metallurgical industry is a major consumer of high-temperature process heat. It is, consequently, a major contributor of CO2 emissions and other greenhouse gases derived from the combustion of fossil fuels for heat and electricity generation. These emissions can be substantially reduced by replacing fossil fuels with solar energy as the source of process heat. Concentrated solar radiation can supply thermal energy to endothermic reactions at temperatures exceeding 2000 K. Examples of metal oxides reduction processes that have been studied experimentally in solar furnaces include the production of Fe, Al, Mg, Zn, TiC, SiC, CaC2, TiN, Si3N4, and AlN by carbothermic reduction of their oxides in Ar or N2 atmospheres.

ALUMINA AND BAUXITE: Bayer Process Design and Simulation

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

Wednesday AM Room: 6E
March 3, 1999 Location: Convention Center

Session Chair: Seymour Brown, Kaiser Aluminum & Chemical Corporation, Gramercy Works, Gramercy, LA 70052 USA

8:30 AM

SIMULATIONS OF BAYER LIQUOR PIPEFLOWS USING A ROTATING CYLINDER ELECTRODE: *N. Andy Darby*[1]; Chris J. Newton[1]; Jonathan D. B. Sharman[1]; [1]Alcan International, Ltd., Banbury Laboratory, Southam Rd., Banbury, Oxon OX16 7SP England

The investigations of flow-related corrosion and erosion occurring in process plants can be aided by the use of a rotating cylinder electrode. Effects of changes in flow velocity, liquid composition and operating temperature can be considered using a relatively small scale apparatus. To achieve successful simulations of plant conditions, a reliable method of correlating plant flows with those present in this type of laboratory equipment is necessary. It is necessary to include thermophysical fluid property data since many plants, such as those in the alumina industry, operate at temperatures and pressures significantly above ambient. Also, Bayer process plants operate with finely divided solids slurries and turbulent flow regimes are utilised to ensure particulates are transported in suspension to prevent scaling of pipework. This paper extends the theories put forward by Silverman and allows a much wider range of fluid Reynolds numbers to be used as well as the characterisation of a rotating drum/cylinder device in use for errosion/corrosion testing at Alcan.

9:00 AM

SIMULATION OF THE BAYER PROCESS: *Emmanouil C. Papadopoulos*[1]; Olga Dimitropoulou[1]; Ioannis L. Paspaliaris[1]; [1]National Technical University of Athens, Laboratory of Metallurgy, Zografou Campus, Zografos, Athens GR 157 80 Greece

Today, off-the-shelf personal computers have enough power to perform complicated calculations of material and energy balance in an extremely short time. In order to study the response of the Bayer Process to various changes in its parameters, a simulation tool was developed in the Laboratory of Metallurgy. The tool was developed

using an object oriented programming language (Microsoft VC++) and can be run under any MS-Windows 32 bit operating system. The simulation tool was developed so that it could be easily operated by anyone with a minimum working knowledge of Windows and that no programming experience should be required. The result of this effort is a complete graphical user interface, which is easy to use and yet gives the flexibility to explore the different possibilities in the flowsheet design. Because of the modular approach, the same tool can be applied to simulate other steady state hydrometalurgical processes. An example is presented showing the close agreement of the model with those from an existing alumina plant.

9:30 AM
1998 VISIONS OF THE FUTURE BAYER PROCESS: *Lester A. D. Chin*[1]; [1]Chin's Consultants International, Inc., 426 Kibbee Rd., McDonough, GA 30252 USA

Karl Joseph Bayer invented and patented the Bayer Process in 1888, and in 110 years since its invention, Bayer Technologists have modified and developed the Process to economically supply the growing aluminium and alumina chemicals industries with Smelter-Grade Alumina and alumina chemicals feedstocks of the desired qualities from different bauxites, under increasing costs of fuels and energy, under increasing concerns for the environmental impacts of the industries and in the environments of developing countries where some bauxites are found. A Bayer Technologist's visions of the future Bayer Process are presented, with the suggestion that some of these visions must be developed to continue the provision of viable solutions to the future challenges of our industries, posed by (a) decreasing grades of bauxite, (b) more stringent Product quality requirements, (c) more stringent environmental requirements for bauxite mining and alumina refining operations, and (d) increasing costs of operating supplies such as fuels, energy, and caustic soda.

10:00 AM BREAK

10:30 AM
ENERGY UTILISATION AND COST REDUCTION IN ALUMINA REFINERIES: *John McFeaters*[1]; [1]Queensland Alumina, Ltd., Parsons Point, Gladstone, Queensland 4680 Australia

Energy comprises a significant proportion of the cost of alumina refining and consequently offers a large potential for cost reduction. Pinch technology can be used to help analyze energy use and identify potential areas for savings. Although, in general, larger opportunities are often limited by process, capital or logistical constraints. However, there are often good opportunities for energy savings through operational and maintenance practices and strategies. These opportunities usually result in less capital intensive projects which do not significantly affect the process. This paper includes a generalized analysis of energy utilization in high temperature alumina plants and the development of guidelines for evaluating energy utilization, identification of potential savings and continuous monitoring of energy efficiency. The paper focuses on energy cost reduction through operation and maintenance practices and control strategies.

11:00 AM
CO-GENERATION—AN EFFECTIVE AND ECONOMIC SOLUTION TO POWER INSTABILITY: *B. K. Mishra*[1]; R. Waris[1]; [1]National Aluminium Company, Ltd., Mines and Refinery Complex, Damanjodi District, Koraput, Orissa 763008 India

Uninterrupted power supply to an alumina refinery assumes great importance for its continuous operation as well as to keep many of its vital equipment in healthy state, even when they are not engaged for achieving production. In various units of the Bayer circuit, bauxite, mud or hydrate slurry, with solid consistency varying between 10-50%, power interruptions for a prolonged period can cause major damage to equipment resulting in production and financial losses. Therefore, it becomes obligatory for alumina plants to have an uninterrupted power source especially when a state owned grid is unstable because of a wide gap between supply and demand. Co-generation of power which generates both electricity and process steam is an effective and economic means to solve the problems associated with power supply instability. The paper highlights the benefits associated with establishing a co-generation unit and its economics at the 800,000 MTPY, NALCO, alumina refinery in Orissa, India.

ALUMINUM ALLOYS FOR PACKAGING IV: Session I — General

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

Wednesday AM Room: 3
March 3, 1999 Location: Convention Center

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

8:30 AM INVITED PAPER
WILL PET DO TO ALUMINUM CANS WHAT ALUMINUM DID TO STEEL?: *Firoze Katrak*[1]; [1]Charles River Associates, Inc., 200 Clarendon St., T-33, Boston, MA 02116 USA

Aluminum containers have lost a significant share of the market to PET over the last few years. What are the key issues driving the competition among aluminum, PET, and steel for soft drink and other types of containers? How do they differ globally? Also, given the different rates of growth for soft drink containers around the world, what are the overall implications for aluminum? This paper will discuss key issues related to the future use of aluminum in containers.

9:00 AM INVITED PAPER
ALUMINUM BEVERAGE CAN STOCK- ENTERING THE 21ST CENTURY: *Tom Thomsen*[1]; [1]231352 Loges Lane, Evergreen, CO 80439 USA

This presentation will include-growth of the beverage industry, global consolidation, threat of plastic & glass bottle, global can makers, can stock producers, and environmental issues.

9:30 AM
ALUMINUM PROPERTIES IN BEVERAGE CANS DURING NECKING: *Dean Johnson*[1]; [1]Ball Packaging Operations, Research Dept., 9343 W. 108th Ave., Broomfield, CO 80021 USA

It is safe to say that the beverage can industry finally understands how to neck 204 and 202 beverage cans at the current gauge. It is the industry challenge to reduce the metal in the can once again. The largest hurdle seems to be reducing the metal in the neck area. In order to understand the necking operation, we need to investigate the metal properties during the entire can making process. There have been several good papers written on necking and how the metal properties change through the necking operations. This paper will further investigate how the metal properties change in the Die Necking process and Spin Necking process.

10:00 AM INVITED PAPER
OPPORTUNITIES, PROBLEMS, AND PERILS FOR ALUMINUM IN PACKAGING: *George J. Binczewski*[1]; [1]SC Systems, P.O. Box 6154, Moraga, CA 94570 USA

The enormous tonnage of aluminum consumed for packaging usages raises serious concerns when semblances of a decline in various sectors are intimated or actually experienced. Significant changes occurring in the associated aluminum manufacturing operations have raised profound questions about the future. Some of the reasons for this are openly obvious while others are somewhat subtle, seldom mentioned and should be examined. Various aspects of the most relevant factors involving fabrications, applications, marketing, and recycling are discussed and recommendations for achieving positive results are made.

ALUMINUM REDUCTION TECHNOLOGY: Prediction & Validation of Performance

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

Wednesday AM Room: 6F
March 3, 1999 Location: Convention Center

Session Chair: John Chen, The University of Auckland, Dept. of Chem. & Mats. Eng., Auckland 1001 New Zealand

8:30 AM
WHY 'BEST' POTS OPERATE BETWEEN 955 AND 970ϒC: *T. A. Utigard*[1]; [1]University of Toronto, Metallurgy & Mat. Science, 184 College St., Toronto, Ontario M5S 3E4 Canada

From fundamental physical and chemical properties of the electrolyte it is shown that for the Hall- Heroult process, the optimum operating temperature is in the range from 955 to 970ϒC. This corresponds to an electrolyte containing 9 to 12 wt% excess AlF_3, 4 to 7 wt% CaF_2 and 2 to 4 wt% Al_2O_3. For operations using LiF, the optimum operating temperature can be lowered by approximately 10ϒC. The present analysis which is supported by industrial practice, is based on i) phase diagrams, ii) density behavior, iii) aluminum-electrolyte interfacial tension, iv) electrolyte electrical conductivity, and v) heat loss through sidewalls and cathode bottom.

9:05 AM
INSTABILITY MECHANISMS IN ALUMINIUM REDUCTION CELLS: *P. A. Davidson*[1]; W. R. Graham[1]; H. O'Brien[1]; [1]Univeristy of Cambridge, Dept. of Engineering, Trumpington St., Cambridge CB2 1PZ UK

We have developed a simple model of reduction cell instabilities which highlights the critical role played by the cryolite thickness. We have extended the model to incorporate movement of the anode blocks and have investigated the effect of small, controlled movements of these blocks. Using a simple control strategy we find that the stability threshold of a cell can, in principle, be greatly improved.

9:35 AM
EIGENMODES AND INTERFACE DESCRIPTION IN A HALL-HEROULT: J. P. Antille[2]; *J. Descloux*[1]; M. Flueck[1]; M. V. Romerio[1]; [1]Swiss Federal Institut of Technology, Dept. of Mathematics, Lausanne 1015 Switzerland; [2]Alusuisse Technology & Management, Ltd., Technology Center Chippis CH - 3965 Switzerland

In a series of papers, the authors have introduced a general method for calculating the stability of a Hall-Heroult cell with high accuracy. The stability of the fluid motions, and particularly the geometry of the interface between aluminum and bath, is modeled using a linearization of the magnetohydrodynamic equations around a steady-state solution of the full set of equations under the given operating conditions of the cell. Measurements were performed on an unstable cell, in which the current in the 16 anodes rods were recorded simultaneously. These measured currents are used as input disturbances to the stability model. The resulting slightly modified force field in turn excites the different modes of oscillation of the cell with amplitudes which are directly related to the amplitudes of the fluctuation of the current. The amplitudes of the different modes are thus determined, so that the time-dependent behavior of the different fields, in particular those of metal surface contour and of the velocity, can be described. A video-recording of the simulated metal surface will be presented.

10:00 AM
DIMENSIONING OF COOLING FINS FOR HIGH-AMPERAGE REDUCTION CELLS: *I. Eick*[1]; D. Vogelsang[1]; [1]VAW Aluminium-Technologie GmbH, Process Modelling, Georg-von-Boeselager-Str. 25, 53117 Bonn Germany

The heat generated in the bath region of high-amperage reduction cells has to be dissipated in a controlled manner to establish a stable side ledge which protects the potlining sufficiently. This can be achieved by designing the potlining of the cells using thermo-electric simulations which also take into account the cooling effect of the shell cradles. In cases of spatial restrictions, due to short distances inside the pot, strong metal flow against the ledge or insufficient ventilation between the pot shells, additional cooling fins welded to the pot shell can be used to avoid "hot spots" and vanishing side ledge thickness. The position and dimensions of such cooling fins can be designed using Computational Fluid Dynamics (CFD) simulations of the heat transfer conditions, assuming turbulent natural convection of the ambient air. The local heat transfer coefficients determined in this way can be implemented in thermo-electric heat balance models, thus offering the possibility to optimize the dimensions of the cooling fins and to analyze the impact of different ventilation conditions. Examples of computational results for different fin configurations are given. The calculated temperature fields and heat fluxes are compared to corresponding plant measurements.

10:25 AM BREAK

10:45 AM
DYNAMIC LEDGE RESPONSE IN HALL-HEROULT CELLS: *K. A. Rye*[1]; T. Eidet[2]; K. Torklep[2]; [1]Elkem Aluminium, R&D-Group, PB 566, Mosjoen N-8650 Norway; [2]Elkem Research, Aluminium Reduction, PB 40, Kristiansand N-4602 Norway

Measurements of the side ledge thickness, bath temperature, electrolyte composition and cell resistance have been performed to verify the dynamic response of Hall-Heroult cells to the extra heat generated during anode effects. Results indicate that melting of the freeze ledge is less severe than predicted by thermal models.

11:10 AM
THREE DIMENSIONAL MODEL FOR CURRENT EFFICIENCY BASED ON THE RATE OF ALUMINIUM TRANSFER TO ANODE: *M. F. El-demerdash*[1]; S. M. El-Raghy[1]; F. A. Moustafa[2]; [1]Cairo University, Faculty of Engineering, Cairo Egypt; [2]The Aluminium Company of Egypt, Cairo

The already published two dimensional model is developed to three dimensional model to estimate current efficiency of the Aluminium Cell. The transfer rate of a aluminium from cathode to anode is estimated as a result of diffusion and mass transfer under velocity field. The mixing differential equation in the homogenous conditions is solved numerically with suitable boundary conditions at Al / electrolyte and electrolyte / anode interfaces. The diffusion coefficient of aluminium in the electrolyte is taken as 1*10-8 m^2s and the maximum solubility of aluminium in electrolyte as 0.03 wt./wt. Results show the effect of three dimensional velocity field on current efficiency.

11:35 AM
SIDE LINING EFFECTS ON THERMAL BEHAVIOUR OF PREBAKED ALUMINIUM CELL: *S. M. El-Raghy*[1]; H. A. Ahmed[1]; S. A. Kaseb[1]; Z. Bassuony[2]; M. M. Ali[1]; [1]Cairo University, Metallurgical Engineering, Faculty of Engineering, Cairo Egypt; [2]Aluminium Company of Egypt, Cairo Egypt

A 203 KA prebaked cell has been designed and operated by Aluminium Company of Egypt (Egyptalum). The cells are side lined by carbon blocks of 20 cm thick at the top of the cell and 40 cm thick at the cathode bottom. One of the cells is side lined by 10 cm thick blocks of silicon carbide. The thermal behaviour of the cell which effects cell life and energy efficiency was determined using a computer thermal model. This model calculates bath temperature and temperatures across the side walls and the bottom of the cell, ledge profile and energy losses through different parts of the cell. The model was used to perform parametric studies to determine the effect of side lining material, namely, carbon and silicon carbide with different thicknesses on thermal behaviour of the cell. Theoretical analysis comparing the results with an actual cell is presented.

ANALYTICAL TECHNOLOGY IN THE MINERAL INDUSTRIES: Microbeam Techniques in the Mineral Industry

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

Wednesday AM Room: 7A
March 3, 1999 Location: Convention Center

Session Chairs: Steven L. Chryssoulis, Amtel, London, Ontario NG6 4X8 Canada; Stamen Dimov, Amtel, London, Ontario NG6 4X8 Canada

8:30 AM INTRODUCTORY REMARKS BY STEPHEN CHRYSSOULIS

8:40 AM INVITED PAPER
QUANTIFICATION OF SILICATE MINERALS BY SEM-BASED IMAGE ANALYSIS: *John Wilson*[1]; [1]Natural Resources Canada, CANMET/MMSL, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

An analytical program using a combination of SEM-based image analysis techniques is developed for automatic modal analysis of ores containing silicates and other gangue minerals. Previous knowledge of which minerals are present and their elemental composition is used to establish discrimination criteria in the image analysis program. The program initially discriminates minerals in polished section on the basis of relative grey values in a backscattered electron image. For those minerals which have overlapping grey levels the minerals are identified by energy dispersive X-ray spectrometry with X-ray counting of minerals grains for twelve or more elements. The minerals are quantified by measuring the accumulated areal proportion of each mineral over several fields. CANMET uses this technique to quantify gangue minerals where high accuracy is required. Two cases of application are presented.

9:00 AM INVITED PAPER
AUTOMATED PRECIOUS MINERAL SEARCH AND APPLICATIONS IN PROCESS MINERALOGY: *Rolando Lastra*[1]; Louis J. Cabri[1]; John M. D. Wilson[1]; [1]Natural Resources Canada, CANMET, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

An image analysis program, written at CANMET, performs an automatic precious minerals search. The program can search, for example, gold minerals in samples in polished sections. The search involves a meander of approximately 100x100 fields (BSE images) at a magnification of 400X. This magnification allows detection of gold grains as small as one micrometer in diameter. Typically, an overnight search of a -270+400 mesh product will scan ~300,000 particles, providing data not easily obtained by manual optical microscopy. The program consists of four parts: the location of grains of correct brightness, WDS dot mapping of such grains, filtering to remove artifacts and recording images and stage location for subsequent retrieval. At the end of the run, the program allows the operator to verify and identify grains classified as gold. CANMET has applied this gold search program in many studies of gold ores of commercial interest. Some case histories will be described.

9:20 AM
TIME-OF-FLIGHT RESONANT IONIZATION MASS SPECTROMETRY (TOF-RIMS) TRACE ELEMENT ANALYSIS OF PRECIOUS METALS IN MINERALS: *Stephen L. Chryssoulis*[1]; Stamen Dimov[1]; [1]AMTEL, 100 Collip Circle, UWO Research Park, London, Ontario N6G 4X8 Canada

Detection and quantification of precious metals (Au, Pd, Pt, and Rh) in rock minerals (quartz, silicates, and carbonates) is important for establishing the maximum attainable recovery. To achieve this goal subpart per billion (ppb) detection limits are required plus the ability to quantify results. Existing quantitative microbeam techniques (EPMA, PIXE, SIMS) have limits of detection in the 0.5-200ppm range. Time-of-flight resonant ionization mass spectrometry (TOF-RIMS) is a microbeam analytical technique that allows for the identification and quantification of atomic species in very small quantities. It utilized a tunable laser source for resonant excitation and ionization of a particular element of interest and has some unique features: elemental selectivity, linear response over a large dynamic range and very high sensitivity. The analysis consist of three steps: i) the solid sample is vaporized by laser ablation to form a plume of neutral atoms, ii) the neutral atoms of a specific atomic element in the plume are resonantly excited and ionized by another tunable laser source, finally iii) the ions created are mass analyzed in a time-of-flight mass spectrometer. The technique was developed at AMTEL for the mining industry for trace element analysis of Au, Pd, and Rh in rock minerals. The TOF-RIMS mass spectrometer was calibrated using NIST reference samples that provide trace concentrations of Au, Pd, and Rh within three decades of dynamic range. Detection limits attained, for the above mentioned elements are in the low ppb range.

9:40 AM INVITED PAPER
DETECTION OF SURFACE SPECIES ON SULPHIDE MINERAL GRAINS BY TIME OF FLIGHT SECONDARY-ION MASS SPECTROMETRY (TOF-SIMS): *Joo Y. Kim*[1]; Stephen Chryssoulis[2]; [1]Noranda Technology Centre, 240 Hymus Blvd., Pointe Claire, Quebec H9R 1G5 Canada; [2]Advanced Mineral Technology Lab (AMTEL), 100 Collip Circle, UWO Research Park, London, Ontario N6G 4X8 Canada

TOF-SIMS (time of flight secondary-ion mass spectrometry) was used to detect, identify and determine the types of species adsorbed on the surface of mineral grains obtained from the process streams of Brunswick concentrator. Lead on the surface of pyrite and sphalerite particles was detected and its distribution was mapped. Pyrite of the Pb-upgrading concentrate has comparable amount of surface lead wit the pyrite grains in the final tails. Sphalerite of the zinc concentrate and plant tails also have comparable surface lead. On pyrite surfaces, lead is more unevenly distributed. The only galena grains detected on the pyrite and sphalerite particles are unliberated remnants attached phases. The principal forms in which lead is lost in the Brunswick circuits are galena attached onto pyrite and halena in the size fraction of minus 5 microns. This accounts for 2/3 of the total lead content in the pyrite concentrate. The study demonstrated the ability of TOF-SIMS to detect the nature of surface species on the mineral particles and showed the technique as an excellent tool for establishing the surface compositions of minerals.

10:00 AM
THE USE OF CATHODOLUMINESCENCE MICROSCOPY AS AN ANALYTICAL TECHNIQUE IN THE MINERAL INDUSTRIES: *Richard D. Hagni*[1]; [1]University of Missouri-Rolla, Dept. of Geology and Geophysics, 125 McNutt Hall, Rolla, MI 65409-0410 USA

Cathodoluminescence Microscopy (CLM) is a valuable technique whose unique character and range of applications to problems in the mineral industries is not adequately recognized and appreciated. Many of the valuable non-metallic minerals exhibit strong cathodoluminescence that makes their recognition and study of grain size, shape, and distribution remarkably easy in exploration and mine samples. CLM forms an especially valuable tool in the analysis of beneficiation problems involving non-metallic ore and gangue minerals. CLM is often the best technique for the study of mineral textures in refractory and pyrometallurgical problems. CLM is an analytical technique that should be more wide used in the minerals industry.

10:20 AM BREAK

10:40 AM

ADVANCED MICROBEAM TECHNIQUES IN THE DETERMINATION OF GOLD MINERALOGICAL BALANCES: *Stephen L. Chryssoulis*[1]; *Stephen Knipe*[1]; [1]AMTEL, 100 Collip Circle, UWO Research Park, London, Ontario N6G 4X8 Canada

The accurate determination of the mineralogical distribution of gold is important in determining the metallurgical treatment of ores and in optimizing recovery. The mineralogical forms of gold and the microbeam techniques applied to their study include: (i) visible gold minerals which can be studied by electron probe (EPMA) to evaluate grain size, composition, and association; (ii) submicroscopic gold which can be quantified in sulphide and oxide minerals by SIMS; (iii) surface gold which may be detected and quantified by TOF-LIMS on a number of substrates (e.g.) carbonaceous matter, iron oxides, and sulphides; colloidal and fine gold associated with quartz and other gangue minerals for which PIXE is used and TOF-RIMS analysis is being developed. The study procedure, instrumentation, and data evaluation are discussed using representative examples from ores showing each form of gold.

11:00 AM

DETERMINATION OF QUANTITATIVE MINERALOGICAL BALANCES FOR MAJOR AND TRACE ELEMENTS: *Louis J. Cabri*[1]; William Petruk[1]; J. H. Gilles Laflamme[1]; [1]CANMET, MMSL, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

A quantitative mineralogical study using ore microscopy, electron microscopy, proton-microprobe, image analysis, and material balances was done on samples from the circuit of a mill in Québec, with particular emphasis on the Cu concentrate. The ore contains many different minerals, of which the principal ones, in decreasing order of abundance, are pyrite, sphalerite, chalcopyrite, bornite, galena, tennantite, and pyrrhotite. Minor to trace amounts of covellite, chalcocite, stannoidite, mawsonite, colusite, wittichenite, miharaite, hessite, petzite, electrum, and an unidentified Cu-Pb-Bi-sulfide also occur. Liberation of pyrite is very high in all size fractions for all samples (averages range from 80-91%). Liberation of chalcopyrite, bornite, sphalerite, and tennantite is relatively high, on average, in the head sample and Cu concentrate (58-74% and 66-88%, respectively). Most of the chalcopyrite in the flotation tails is not liberated, whereas about one half of the tennantite and sphalerite are liberated in both the flotation and final tails. Recoveries calculated for the Cu concentrate (on the basis of assays, mineralogy, and image analysis) are chalcopyrite 87.7%, bornite 67.5%, sphalerite 41.7%, tennantite 88.1%, galena (together with some trace minerals) 33.0%, pyrrhotite 26.2%, and pyrite 2.4%. Materials balance calculations, were done on the five major sulfide minerals, to determine the mineralogical distribution of 13 major, minor, and trace elements (as determined by proton-microprobe analyses). It was determined that, in the Cu concentrate, tennantite is the principal carrier of Bi (60%), As (83%), and Te (80%). Therefore, removal of tennantite, which is mostly liberated in the Cu concentrate (average 88%) would significantly reduce these three contaminant elements, but at a loss of 8% of the Cu, 1.9% of the Zn, and 0.6% of the Ag. The precious metals (Au, Ag) occur as discrete minerals (electrum, petzite, hessite); some of the Ag occurs in solid solution in bornite (19.4% of Ag in Cu concentrate) and in sphalerite. Four of the major sulfides (chalcopyrite, bornite, sphalerite, tennantite) in the ore also contain measurable concentrations of the following additional elements: Se, Cd, In, Sn, and Sb, occurring mostly as solid solutions. Some of these elements are also concentrated (up to ab out 18%) in minor and trace minerals such as colusite, stannoidite, and mawsonite.

11:20 AM

MINERALOGICAL TRANSFORMATION OF A COMPLEX CU-ZN CONCENTRATE DURING PARTIAL ROASTING IN A NICHOLS HERRESHOFF MONOHEARTH FURNACE: E. Boydens[1]; S. Brouwer[2]; *L. J. Evrard*[1]; E. Bosly[2]; [1]Université Catholique de Louvain, Dépt. des Sciences des Matériaux et des Procédés, PCIM, Place Sainte Barbe, 2, Louvain-la-Neuve 1348 Belgium; [2]Union Minière, UM Research, Kasteelstraat, 7, Olen 2250 Belgium

A partial desulphurization roasting process has been tested on a typical copper-zinc sulfide concentrate in a Nichols Herreshoff monohearth pilot furnace whereby the sulfur is partially removed and iron is to a certain degree preferentially oxidized. The phase changes that occur in the particles during selective roasting at 650°C and 800°C over a range of residence times were investigated by the use of chemical analysis, light microscopy, X-ray diffraction and electron microprobe analyses. The purpose of this work was to get an insight into the mineral assemblages by controlled oxidation roasting and to evaluate if this non traditional approach could be afterwards used to recover zinc, copper and iron by a physical separation method.

11:40 AM

CERAMIC MATRIX COMPOSITE ALUMINUM NITRIDATION MINERALOGICAL CHARACTERIZATION UTILIZING ELECTRON MICROSOCOPY, OPTICAL MICROSCOPY, AND X-RAY DIFFRACTION TECHNOLOGY: *Ann M. Hagni*[1]; [1]A. P. Green Refractories, Inc., Research Division, 1 Green Blvd., Mexico, MO 65265 USA

Ceramic matrix composites are inorganic materials that are particularly resistant to heat and abrasion. A magnesia (MgO) and aluminum nitride (AlN) based composite is formed by exposing a magnesia preform to a metallic alloy block in a nitrogen atmosphere at 950°C. The metallic alloy, which is composed primarily of aluminum, is wicked into the preform. Aluminum nitride matrix is grown between MgO particles as the wicked metallic aluminum reacts with nitrogen. Unused metallic alloy, remnant metallic alloy (carcass), and composite produced after 8 hours, 50 hours, and 225 hours of processing are characterized mineralogically. Reflected light microscopy, transmitted light microscopy, scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction techniques reveal that in addition to anhedral to euhedral Al_3Ni, Al_2Si_2Sr, and Al crystallizing in the metallic alloy carcass, AlN and (Al,Si)N have formed as well. Nitride rings and nodules in the carcass may hinder AlN growth in the composite. Fine-grained residual Al_3Ni and Al_2Si_2Sr are dispersed throughout the composite matrix. The nitride matrix is composed of AlN and (Al,Si)N. Rim replacement of MgO by spinel occurs in the composite. Aluminum nitrides in the metallic carcass cathodoluminesce deep blue-purple, unlike non-luminescent AlN in the composite matrix. This distinction in cathodoluminescence suggests a different mode of formation for composite AlN than for carcass AlN. Phase interpretations and textural characterizations deduced from the implementation of sophisticated mineralogical techniques have contributed to the understanding, and will aid in the improvement, of MgO/AlN-based ceramic matrix composite processing.

CAST SHOP TECHNOLOGY: DC Casting/ Modeling II

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

Wednesday AM Room: 6C
March 3, 1999 Location: Convention Center

Session Chair: Dr. James W. Evans, University at California, Berkeley, CA 94720 USA

8:30 AM
A MICRO/MACRO MODEL FOR THE EQUIAXED GRAIN SIZE DISTRIBUTION IN DC-CAST ALUMINIUM INGOTS: *Arild Hakonsen*[1]; Dag Mortensen[2]; Steinar Benum[1]; Hans Erik Vatne[1]; [1]Hydro Aluminium, R&D Materials Technology, P.O. Box 219, Sunndalsora, More & Romsdal 6601 Norway; [2]Institute for Energy Technology, Mats. and Corrosion Tech. Dept., P.B. 40, Kjeller, Akershus 2007 Norway

A micro/macro model for dendritic equiaxed grain nucleation and growth is applied on the DC-casting process of sheet ingots. Different nucleation laws, which all relates the nucleation to the undercooling of the melt, are compared. The growth velocity of the grain envelope is restricted by the alloying elements. The results of the micro model are the evolution of the solid fraction until impingement of the grains, as well as the mean grain size. The model is also able to calculate the grain size distribution around the mean size. The macro model for heat and fluid flow is coupled with the micro model by an iterative micro/macro time step scheme. This coupling makes it possible to calculate the grain size distribution versus position in the ingot. The modelling results are compared with measurements of secondary dendrite arm spacing and mean grain size in ingots with varying grain refinement additions.

8:55 AM
THERMOMECHANICAL BEHAVIOR OF AN AA3004 ALLOY AT LOW STRAIN RATE: *W. M. Van Haaften*[1]; B. Magnin[2]; W. H. Kool[1]; L. Katgerman[1]; [1]Delft University of Technology, Laboratory of Materials, Rotterdamseweg 137, Delft, 2628 AL The Netherlands; [2]Pechiney CRV, BP 27, Voreppe 38340 France

Recent thermomechanical modelling to calculate the stress field in industrially DC cast slabs has been successful, but lack of material data limits the accuracy of these calculations. The aim of this study is to determine the constitutive behaviour of AA3104 in as-cast condition. This was done by tensile testing at low strain rates in a broad temperature range. The parameters of a modified Ludwig equation were determined and are now being used in thermo-mechanical models. In order to study the material during later stages of casting, its behaviour at 50°C after prestraining at higher temperatures was also investigated.

9:20 AM
PHYSICAL MODELING OF THE EFFECTS OF THERMAL BUOYANCY DRIVEN FLOWS IN EM AND DC CASTERS: *D. Xu*[1]; [1]University of California - Berkeley, Dept. of Mats. Sci. and Mineral Eng., 516 Evans Hall, Berkeley, CA 94720 USA

Particle Imaging Velocimetry (PIV) was used to illustrate the role of thermal buoyancy driven flows in a laboratory scale model of an aluminum caster. The use of PIV enables the entire flow field to be captured at once yielding a quantitative description of the fluid. The water model, described in preceding papers, was equipped with a water heater than enabled preheated water to enter the system. The Tundish number, along with the Reynolds number, was matched with operational properties of an actual aluminum caster to obtain thermal and dynamic similarity. Results were obtained for representative superheats of 50, 75 and 100°C in two different combo bags and a channel bag. The results suggest that thermal buoyancy does modify the flow and is therefore important in proper representation of the actual system, however; the flow driven by the inflow through the nozzle/ bag system was still dominant in the sump.

9:45 AM
EFFECTS OF COMBO BAG GEOMETRY ON THE THERMAL HISTORY AND SUMP PROFILE OF A 3104 DC CAST INGOT: *W. K. Jones*[1]; D. Xu[1]; J. W. Evans[1]; E. Williams[1]; [1]University of California, Dept. of Mats. Sci. and Mineral Eng., 516 Evans Hall, Berkeley, CA 94720 USA

An experimental campaign was performed at the Reynolds Metals Company Cast House, Richmond, VA, to study the influence of combo bag geometry on a DC cast aluminum alloy. Two different size combo bags were chosen and subject to two different casting practices. Sacrificial thermocouples were used to track the steady state thermal history. The results clearly show that the longer bag created a deeper sump, up to four inches in the center. This is anticipated as previous experiments on a water model at Berkeley shows that the longer bag directs more aluminum into the lower regions of the sump. This result is important noting that previous research has shown a deeper sump to have greater centerline macrosegregation. A different set of stationary thermocouples was used to obtain the liquid temperature in an attempt to resolve the turbulent nature of the flow. The measurements showed that the shorter bag produced greater thermal fluctuations, which are believed to due to a greater level of turbulence. Fourier analysis was performed to determine the periodicity of the turbulence. Macrosegregation profiles were obtained across the rolling faces and the larger bag showed worse negative macrosegregation.

CAST SHOP TECHNOLOGY: Molten Metal Processing - Final Properties

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

Wednesday AM Room: 6D
March 3, 1999 Location: Convention Center

Session Chair: Dr. Ray D. Peterson, IMCO Recycling, Irving, TX 75039 USA

8:30 AM
PROCIAS: A JOINT PROJECT ON PROCESS CONTROL OF ALUMINIUM MELTS: *François De Schutter*[1]; Johan Verwimp[1]; Willy Engelen[1]; Stefan Kuypers[1]; Marc Ryckeboer[2]; Werner Verberckt[3]; Peter Hermans[4]; Cees Castelijns[5]; [1]Vlaamse Instelling Voor Technologisch Onderzoek, Boeretang 200, Mol 2400 Belgium; [2]Remi Claeys Aluminium NV, Kortemarkstraat 52, Lichtervelde B-8810 Belgium; [3]Hayes Lemmerz Belgium BVBA, Lage Weg 392, Hoboken B-2660 Belgium; [4]Lamitref Metals NV, Fr. Sheidlaan, Hemiksem B-2620 Belgium; [5]Hoogovens Aluminium NV, Stockletlaan 87, Duffel B-2570 Belgium

In order to ameliorate the current melting processes of the Flemish aluminium industry a joint project between a research institute and four foundries/cast houses was set up. The aluminium companies cover a large product area ranging from cast wheels, high frequency welded tubes, rolled electricity wire up to rolling slabs and extrusion billets for transportation, construction and engineering purposes. During the in-depth analysis of the industrial melts the chemical composition is monitored using optical emission spectroscopy, hydrogen concentration us-

ing the Alscan device and finally the qualitative as well as the quantitative inclusion concentration using the novel PoDFA-f and LiMCA-II. The aim is to make a coupling between melt parameters and the final material properties. In order to achieve this goal, several process parameters have been identified as important: material supply, temperature, fluxing, modifiers, degassing, grain refinement, metal velocity and filtering. The role of each of these parameters and their influence on the melt properties as well as the final end product properties will be investigated by modern design of experiment techniques. This paper will discuss the practical introduction of these techniques for these purposes as well as the introduction of the state-of-the-art measuring tools into the Flemish aluminium industry.

8:50 AM

SCRAP VARIABILITY AND ITS EFFECTS ON PRODUCING ALLOYS TO SPECIFICATION: *Ray D. Peterson*[1]; [1]IMCO Recycling, Inc., 5215 North O'Connor Blvd., Irving, TX 75039 USA

Historically, aluminum products were fabricated from prime metal and master alloys to achieve the desired final alloy composition. Today, many aluminum products are made entirely from scrap-based charges. Aluminum scraps are broken down into a myriad of subcategories based on source, alloy chemistry, and cleanliness. Yet there is still significant variability within a single scrap type. This degree of variability is different for various types of scraps. The variability also changes for different elements within a single scrap type. All of this variability in the scrap complicates the charge calculation and the subsequent manufacture of specification alloys. Sources of variability within a scrap type are discussed along with methods of characterizing the scrap. The implications of scrap variability and its effect on meeting specifications and determining production cost are considered.

9:10 AM

THERMODYNAMIC COMPUTER PROGRAMS AS AN AID IN REFINING OF ALUMINIUM: *Aud Nina Waernes*[1]; Johan Kr. Tuset[2]; Soeren Groenborg Hansen[2]; [1]SINTEF, Materials Technology, Alfred Getz v 2B, Trondheim N-7034 Norway; [2]Norwegian University of Science and Technology, Dept. of Metall., Alfred Getz v 2B, Trondheim N-7034 Norway

Computer Programs for calculation of complex chemical equilibrium is a powerful tool for solving problems in the metallurgical industry, for instance in refining of aluminium containing dissolved impurities. Since the refining process probably take place under conditions close to chemical equilibrium, modelling based on thermodynamic calculations is an important tool in optimising the process or when new processes are being planned. Most metallurgical processes and systems depend on the properties of solutions such as alloys, slags and salts. It is well known that when different substances are mixed, the resulting mixture often display properties different from those expected on basis of ideal mixing. Computer programs like ChemSage, include different solution models that describe the deviation from ideal mixing for alloy systems. In the refining of aluminium and aluminium alloys, knowledge of the interaction between both the dissolved impurity elements (Na, K, Ca, Li etc.) and the alloying elements are essential. This means that in order to achieve reliable results from the computer calculation, consistent thermochemical data must be available. By combining data from the literature with data from the databases that comes with the computer programs, equilibrium condition for the progress of the refining of aluminium with different fluxing agent (Cl2 and AlF3), has been calculated. The advantage of using a thermodynamic computer program in the calculation of the progress of aluminium refining is that the influence of temperature, amount of fluxing agents and alloy composition, on the final metal composition can very easily be established.

9:30 AM

SOLIDIFICATION STRUCTURES AND PROPERTIES OF CAST SAMPLES OF ZINC-ALUMINUM ALLOYS AND COMPOSITES: *Carlos Enrique Schvezov*[1]; Rafael Auras[2]; [1]University of Misiones, Faculty of Sciences, 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/UNSAM-CNEA, 1552 Azara St., Posadas, Misiones 3300 Argentina

Zinc-Aluminum alloys with different additions of Silicon and Copper and the respectives composite containing Silicon Carbides particles as reinforcements were cast in small samples. The cast alloys and composites were analysed using quantitative metallography and the size of the solidification structure were determined along with the particle size distribution in number and location. In addition, SEM analysis of the different alloying element distribution in the microstructure were performed in different sample positions which show different size structures. The Silicon precipitates were identified and their size and location determined. The samples were tested for hardness and wear. The hardness values were correlated with the local structure, alloy composition and particle distribution. The wear tests were performed at different pressures and speeds. The results of this investigation will be presented and discussed. Particular attention is given to the correlation between the microstructure and the mechanical properties which can be employed to improve the use of these alloys.

9:50 AM

A THERMODYNAMIC DATABASE ON ALUMINUM ALLOYS FOR PRACTICAL ALLOY DESIGN: *Haiyan Liang*[1]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

A thermodynamic database has been developed for multicomponent aluminum alloys. All the major alloying elements used in commercial aluminum alloys have been included in this database. By integrating the thermodynamic database with kinetic solidification models we can predict solidification paths of commercial aluminum alloys. In this presentation, we report the model-calculated solidification paths for several alloys with compositions close to real commercial aluminum alloys. The calculated results are in good agreement with experimental data reported in the literature. To further confirm the reliability of the database, our group performed detailed experimental investigations on the microstructure and microsegregation of the Al-Cu-Mg-Zn quaternary alloys. The experimental results are in accord with calculations. The good agreement obtained between calculations and experiments demonstrated that the thermodynamic database can be used for practical alloy design to achieve desired microstructures and properties for commercial alloys.

10:10 AM BREAK

10:30 AM

ELEMENT DISTRIBUTION IN Al-Si ALLOYS: *Heng-Xian Zhao*[1]; Xiao-Fei Xu[1]; [1]Northeastern University, Shenyang 110006 China

Investigation on element distribution in Al-Si alloys has been made. A number of ingot samples were analyzed using a Foundry Spectrovac (FSQ,BAIRD,USA). The contents of some elements are found different in different zones of the ingots. Discussion and conclusion are given in the paper.

10:50 AM

PREPARATION OF Al-Si ALLOYS USING SODIUM- FLUOSILICATE AND MOLTEN ALUMINIUM: *Ibrahim Hamed Aly*[1]; A. A. Nofal[2]; E. E. Ebrahiem[1]; F. M. Ahmed[3]; A. M. Omran[3]; [1]Minia University, Chem. Eng. Dept., Faculty of Engineering, El-Minia Egypt; [2]Central Metallurgical Research and Development Institute, President, El-Tebbin Egypt; [3]Aluminium Company of Egypt, Nag-Hammady Egypt

Al-Si alloys, and sodium aluminium fluorides were produced by reacting molten pure aluminium with sodium fluosilicate (by-product from super-phosphate fertilizer plants). The obtained products can be easily separated and used directly in casting shops or aluminium reduction cells in aluminum smelters. The produced Al-Si alloys are high quality, containing up to 20% silicon and less than 0.12% iron. Different factors affecting the composition of the produced Al-Si alloys were studied. These factors are: temperatures, mixing intensity, sodium fluosilicate to aluminium ratio, feeding rate and particle size. The results obtained were correlated and empirical equations representing the silicon-content in the produced alloy with each factor. Microstructure examination and x-ray diffraction were carried out on the produced alloys as well as the fluorine salts.

11:10 AM

MICROSTRUCTURES AND ELECTRICAL PROPERTIES OF STRONTIUM TREATED ALUMINUM FOUNDRY AND WROUGHT

ALLOYS: *B. Closset*[1]; [1]Timminco, 44, Chemin Petite-Boissiere, Geneve CH-1208 Switzerland

Aluminum foundry and wrought alloys treated with strontium were examined by optimal microscopy. It is shown that strontium affects mainly the eutectic silicon in Al-Si foundry alloys and to a lesser extend other compounds such as Mg2Si and AlFeSi. In wrought aluminum 6XXX series the strontium addition results in a change of morphology of the ternary AlFeSi intermetallics. The shape of coarse - AlFeSi is altered and a more finely dispersed - AlFeSi intermetallics are obtained. The electrical resistivity or conductivity of both types of aluminum alloys were measured before and after strontium treatment Strontium modified Al-Si foundry alloys exhibit a higher electrical conductivity. It has also been demonstrated that the strontium treated alloys react differently to a heat treatment cycle (T4 or T6) than unmodified alloys. In this work microstructural changes are correlated to electrical resistivity or conductivity measurements.

11:30 AM

DISSOLUTION MECHANISMS OF COMPRESSED ADDITIVES IN ALUMINIUM: *David John Bristow*[1]; Sarah Lockwood[2]; Tom Woodcock[2]; Ray Cook[1]; [1]London & Scandinavian Metallurgical Company, Ltd., Technical Centre, Fullerton Rd., Rotherham, S. Yorks S60 1DL UK; [2]University of Sheffield, Dept. of Eng. Mats., Mappin St., Sheffield, S. Yorks S1 3JD UK

The addition of compressed powder additives to aluminium for alloying purposes is well established. In this investigation, the mechanisms by which tablet dissolution occurs, and the reasons for variable rates of dissolution have been investigated. The dissolution of additives containing Cr, Fe, Mn and Ti have each been investigated, by quenching the aluminium at various times after tablet addition. Tablet disintegration is monitored, and metallography reveals the successive phases which develop. These are related to the diffusion rates of species through the successive layers of intermetallics. Fe-based additives are shown to exhibit a tabletting pressure sensitivity whose practical implications are discussed.

CREEP BEHAVIOR OF ADVANCED MATERIALS FOR THE 21ST CENTURY: Low Stress Creep Mechanisms: A Discussion 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 USA; Amiya K. Mukherjee, University of California, Dept. of Chem. Eng. & Mats. Sci., Davis, CA USA; K. Linga Murty, North Carolina State University, Box 7909, Raleigh, NC 27695-7909 USA

Wednesday AM Room: 15A
March 3, 1999 Location: Convention Center

Session Chair: B. Dyson, Imperial College of Science, London, England; M.J. Mills, Ohio State University, OColumbus, OH USA

8:30 AM INTRODUCTION

8:35 AM KEYNOTE

PARTICLE DISTRIBUTION AFTER DIFFUSIONAL CREEP: *F. R. N. Nabarro*[1]; [1]Division of Materials Science and Technology, CSIR, PO Box 395, Pretoria 0001 and, Condensed Matter Physics Research Unit, University of Witwatersrand, Private Bag 3, Wits 2050, Johannesburg South Africa

The theory of diffusional creep implies that in a particle-strengthened alloy there will be zones denuded of particles on grain boundaries transverse to a tensile stress and zones enriched in particles on lateral boundaries. Within this framework, Bilde-Sorensen, Smith and Thorsen have studied the complications which arise when one considers both the glide and the climb forces on dislocations in the grain boundaries. Ruano, Sherby, Wadsworth and Wolfenstine have argued that the presence of denuded zones is not conclusive evidence for diffusional creep, and have proposed two other mechanisms. We argue here that one mechanism, the dragging of particles by a migrating boundary will produce an enriched zone immediately adjacent to the denuded zone, and cannot account for isolated enriched and denuded zones, while the other mechanism, re-solution of the particles, is implausible and cannot account for enriched zones.

9:05 AM KEYNOTE

DEFORMATION MECHANISMS IN CRYSTALLINE SOLIDS AND NEWTONIAN VISCOUS BEHAVIOR: *Oleg D. Sherby*[1]; Oscar A. Ruano[2]; Jeffrey Wadsworth[3]; [1]Stanford University, Dept. of Materials Science and Engineering, Stanford, CA 94305 USA; [2]CENIM, CSIC, Dept. of Phys. Metall., Av. Gregorio del Amo 28040 Spain; [3]Lawrence Livermore National Laboratory, P.O. Box 808, L-353, Livermore, CA 94550 USA

The three principal mechanisms of plastic flow in crystalline solids at elevated temperature are crystal slip, grain boundary sliding and diffusional flow. All these mechanisms involve the diffusion of atoms as the rate controlling process, either in the lattice or in the grain boundary. Under the correct condition of microstructure, temperature and stress, each mechanism can lead to Newtonian-viscous flow behavior. That is, the strain rate increases linearly with the applied stress. In the case of crystal slip, Newtonian-viscous behavior is observed at very low stresses, and, in pure metals, is known as Harper-Dorn (H-D) creep. It is also observed in anisotropic crystalline solids when deformed under thermal cycling conditions. The dislocation density and the stacking fault energy are important structural factors that contribute to crystal slip-controlled Newtonian flow. In the case of grain boundary sliding, Newtonian -viscous behavior is observed in fine-grained solid solution alloys under conditions where grain-boundary sliding is accommodated by dislocation glide controlled by the diffusion of solute atoms. In the case of diffusional creep, which is rigorously described by the Nabarro-Herring (N-H) theory, the creep rate is controlled by grain size and by the rate of atom diffusion in the lattice and in the grain boundary. Deformation mechanism maps permit establishing the conditions of dislocation density, grain size, stress and temperature where each deformation process can be expected to be rate-controlling.

9:35 AM KEYNOTE

THE IDENTIFICATION AND SIGNIFICANCE OF DIFFUSIONAL CREEP PROCESSES: *G. W. Greenwood*[1]; [1]University of Sheffield, Dept. of Engineering Materials, Sheffield S1 3JD UK

The theory of diffusional creep was first put forward 50 years ago and the physical basis of the theory has never been disputed. It provides equations that predict creep rates in terms of parameters that can separately be determined. Whilst several experimental studies have supported the theory, others have illustrated situations where these equations do not apply. Such results have led to proposals that this form of creep does not occur or that, in some situations, the original theory requires modification. In the present paper attention is particularly given to identifying the conditions under which this form of creep may predominate and the relationships that are then applicable. The validity of the original theory requires that lattice dislocations remain immobile and that grain boundaries act as perfect sources and sinks for vacancie and do not undergo Rachinger type sliding. It is now established that microstructural features can be important and specific microstructural changes, including grain size and shape, precipitate location and grain boundary profiles can be observed. These can influence the time, temperature and stress dependence of strain. Below specific temperatures grain boundary diffusion coefficients are important and can be highly sensitive to impurities. Some appreciation can be obtained of the transition from diffusional to dislocation creep through a knowledge of the patterns of variation of internal stress. Finally, the importance of

identifying the mode of deformation and the part that may be played by directional diffusion are shown to be of crucial significance in understanding mechanisms of fracture at elevated temperatures, in radiation damage and in joining and sintering processes as well as in diffusional creep.

10:05 AM INVITED PAPER
DEFORMATION BY GRAIN BOUNDARY SLIDING AND SLIP CREEP VERSUS DIFFUSIONAL CREEP: *Jeffrey Wadsworth*[1]; Oscar A. Ruano[2]; Oleg D. Sherby[3]; [1]Lawrence Livermore National Laboratory, P.O. Box 808, L-353, Livermore, CA 94550 USA; [2]CENIM, CSIC, Dept. of Physical Metallurgy, Av. Gregorio del Amo 28040 Spain; [3]Stanford University, Dept. of Materials Science and Engineering, Stanford, CA 94305 USA

A review is presented of the debates between the present authors and other investigators regarding the possible role of diffusional creep in the plastic flow of polycrystalline metals at low stresses. These debates are recorded in eleven papers over the past seventeen years. in these papers it has been shown that the creep rates of materials in the so-called "diffusional creep region" are almost always higher than those predicted by the diffusional creep theory. Additionally, the predictions of grain size effects and stress exponents from diffusional creep theory are often not found in the experimental data. Finally, denuded zones have been universally considered to be direct evidence for diffusional creep; but, those reported in the literature shown to be found only under conditions where a high stress exponent is observed. It is proposed that diffusion-controlled dislocation creep is the dominant deformation process during the formation of such denuded zones by stress-directed grain boundary migration with the precipitates dissolving in the moving grain boundaries. The above observations have led us to the conclusion that grain boundary sliding and slip creep are the principal mechanisms for plastic flow in the so-called "diffusional creep regions."

10:30 AM BREAK

10:40 AM INVITED PAPER
EVIDENCE FOR DIFFUSIONAL CREEP IN Cu-2wt%Ni: *J. B. Bilde-Sørensen*[1]; P. A. Thorsen[1]; [1]Risø National Laboratory, Materials Research Dept., Roskilde DK-4000 Denmark

A sample of Cu-2wt%Ni was crept in tension to an elongation of 2.4% at 1073 and 1103 K under a stress of 1.14 MPa. Prior to creep the sample was covered with a regular grid of alumina. After creep the local deformation could be measured from the grid. The deformation was seen to be localized to the boundaries. With a knowledge of the orientation of the grain boundary plane the deformation can be divided into a sliding component and a component arising from removal or deposition of material at the boundary. It was demonstrated that material had been deposited at some of the transverse boundaries and removed from some of the longitudinal boundaries. The results are discussed in terms of the coincident site lattice (CSL) model for the grain boundary structure. On this basis it is possible to explain a number of features that have all been observed experimentally: (i) some boundaries close to an exact CSL orientation are inactive, (ii) the ratio of sliding to deposition/removal varies from boundary to boundary, (iii) some transverse boundaries exhibits negative climb (iv) material is deposited on some of the longitudinal boundaries. It is suggested that contemporary grain boundary theory should be included in a revised model for diffusional creep.

11:05 AM INVITED PAPER
CASE STUDIES IN DIFFUSIONAL CREEP: *Brian Wilshire*[1]; [1]University of Wales, Swansea, Materials Engineering, Singleton Park, Swansea, Wales SA2 8PP UK

Diffusional creep theories are undoubtedly elegant and have been widely assumed to account for the high temperature creep behaviour displayed by many metallic and ceramic materials at low stresses. However, the experimental observations commonly quoted as evidence for diffusional creep are, at best, inconclusive. The limitations of this evidence are reviewed for pure metals and particle-strengthened alloys, as well as for monolithic ceramics and ceramic-fibre-reinforced ceramic matrix composites.

11:30 AM
A FINITE ELEMENT STUDY OF GRAIN BOUNDARY SLIDING IN INCANDESCENT LAMP FILAMENTS: *John Selverian*[1]; [1]Osram Sylvania Development Inc., 71 Cherry Hill Drive, Beverly, MA 01915

Finite element analysis was used to study the effect of grain boundary creep on sag and kinking of incandescent lamp filaments. Grain boundary creep can explain most of the sag and kinking seen in standard filaments. As the number of grain boundaries increase the amount of sag increases. As the degree of grain boundary interlocking decreases the sag increases. Kinks can be explained by grain boundaries with a low degree of interlocking, i.e. flatter. Not every flat grain boundary kinks. Kinks result whenever there are a few grain boundaries that are significantly flatter (by approximately a factor of 3 than the majority of grain boundaries.

ELECTRICAL AND THERMAL PROPERTIES OF MATERIALS: Session I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sungho Jin, Lucent Technologies Bell Labs, Murray Hill, NJ 07974 USA; Anthony Mulligan, Advanced Ceramics Research, 851 East 47th St., Tuscon, AZ USA; King Ning Tu, University of California, Dept. of Mats. Sci. and Eng., Los Angeles, CA 90095 USA

Wednesday AM Room: 16B
March 3, 1999 Location: Convention Center

Session Chair: K. N. Tu, University of California, Dept. of Mats. Sci. and Eng., Los Angeles, CA 90095 USA

8:30 AM INVITED PAPER
CONTACT ISSUES FOR GaN TECHNOLOGY: D. Qiao[1]; *S. S. Lau*[1]; [1]University of California, Dept. of Elect. and Computer Eng., San Diego, CA 92093-0407 USA

In this presentation, we review the metal-GaN contact technology to shed light on some of the critical issues such as GaN surface cleaning before metallization, low-resistance contacts to GaN/AlGaN hetero-structures, barrier heights of metals on AlGaN and on hetero-structures. Some general conclusions are drawn on the electrical behaviour of contacts for further advances in this field.

9:00 AM INVITED PAPER
MICROSTRUCTURE AND THERMOELECTRIC PROPERTIES OF SINTERED Bi2Te3: Gil-Geun Lee[1]; Byoung-Kee Kim[1]; *Hyung-Sik Chung*[1]; [1]Korea Institute of Machinery and Materials, Chanwon 641-010 Korea

Thermoelectric materials have been studied to increase the figure of merit by controlling of microstructure using powder metallurgical process. Various kinds of Bi2e3 starting powders which have different particle size distribution and second phase content are synthesized by mechanical grinding process, and their powders are sintered. The sintered bodies have shown different microstructure and characteristics, i.e.: grain size distribution and thermal/electric conductivity, etc.. This paper discusses the effect of microstructure on the thermoelectric properties of Bi2Te3 based on the effective medium and percolation theories.

9:30 AM
SEEBECK EFFECT ON AMORPHOUS-CRYSTALLINE INTERFACE AND AMORPHOUS-CRYSTALLINE THERMOCOUPLE: *Mikhail V. Finkel*[1]; Jim S.-J. Chen[2]; [1]DAATH-Scientific Center, Dept. of Mats. Sci., 9926 Haldeman Ave., #36A, Philadelphia, PA 19115 USA;

[2]Temple University, Mechanical Engineering Dept., Norris & 12th St., Philadelphia, PA 19022 USA

Seebeck phenomenon of an amorphous-crystalline interface has been discovered and studied for various alloy systems. Thermocouples consisting of amorphous and crystalline parts of a single alloy composition were created. A thermocouple made of as-cast Co-Fe-Si-B metallic glass demonstrates constant thermopower of 8.1mV/K in the temperature range from 293 to 593K. It has been shown that amorphous-crystalline thermocouple (ACT) can be stabilized by high-temperature anneal. Several methods are proposed for ACT fabrication. One of them involves crystallization of amorphous ribbon on the part of its length using heterogeneous temperature field. Sharp natural amorphous-crystalline transition zone plays a role of the thermocouple hot junction. It is suggested that for low and moderate temperatures ACT could be techni cally and economically more advantageous than traditional crystalline thermocouples.

9:50 AM

THE APPARENT ACTIVATION ENERGY AND CURRENT DENSITY EXPONENT OF ELECTROMIGRATION DAMAGE IN CHIP LEVEL INTERCONNECT LINES: A GRAIN STRUCTURE BASED STATISTICAL APPROACH: *T. M. Korhonen*[1]; Y. -K. Liu[1]; D. D. Brown[2]; M. A. Korhonen[1]; C. -Y. Li[1]; [1]Cornell University, Dept. of Mats. Sci. and Eng., Ithaca, NY 14583 USA; [2]Advanced Micro Devices, Sunnyvale, CA 94088 USA

Electromigration reliability assessment of chip-level interconnects is based on accelerated testing at a higher temperature and larger current density that expected in service conditions. The critical parameters needed to extrolate accelerated test data to service conditions are the activation energy, Q, and the current density exponent, n. Although these are well-known for the elemental processes there is no consensus which apparent activation energy or currently density exponent values would be appropriate in reliability estimates for realistic line structures comprising regimes of bamboo and polycrystalline sections. In this contribution we shall apply a Monte-Carlo approach to generate realistic-like ID line structures, and model their electromigration (EM) lifetimes. For a given grain structure distribution, we follow the stress evolution along the interconnect line and let a void nucleated at the growth of nucleated voids as a function of time until the largest of them reaches a specified size, resulting in a 'failure' of the particular line. By repeating this process for various temperatures and current densities we can extract the apparent activation energies and current density exponents from the modelling data. The results obtained compare well with literature data and prevalent theoretical concepts.

10:10 AM BREAK

10:30 AM INVITED PAPER

PATTERN FORMATION BY METALLIC AND NON-METALLIC MICROSPHERES IN A PASSIVE FLUID UNDER ZERO OR APPLIED FIELDS: *Weijia Wen*[1]; K. N. Tu[1]; [1]UCLA, Dept. of Mats. Sci. & Eng., Los Angeles, CA 90095-1595 USA

Electric-field-induced fractal and chain patterns formed respectively by metallic and non-metallic microspheres in silicone oil have been investigated. The two patterns are interchangeable if we start with the same glassy microspheres and coat them sequentially with metallic and by surface conductivity of the microspheres. Under zero field, Ni-coated glassy microspheres tend to form rings and chains. The ring and chain formation can be simulated on the basis of dipole-dipole interaction, without thermal noise, between microspheres.

11:00 AM

ELECTRICAL RESISTIVITY CHANGES AND PHASE TRANSFORMATIONS IN Ti-(25-52) AT.% Al ALLOYS: D. Veeraraghavan[2]; Uwe Pilchowski[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Mats. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]VLSI Technology, 9651 Westover Hills Blvd., San Antonio, TX 78251 USA

Phase transformations in Ti-(25-52) at.% Al were studied by electrical resistivity measurements over a range of temperatures using a special device. The alpha2, alpha, beta and gamma phases were observed to have distinctly different resistivities and temperature dependencies, owing to which phase transformations could be monitored. The room temperature resistivity of stoichiometric alpha2-Ti3Al and gamma-TiAl are 118 and 31 micro-ohm.cm, with a difference of 87 micro-ohm.cm. The changes in resistivity with temperature are also significantly different in that in alpha2 the resistivity saturates to a near-constant value near 750Ɣ, whereas that of the gamma phase shows a linear and near-constant slope with temperature like most metallic materials. In order to explain these differences, the electrical resistivity of alpha2 and gamma phases has been modeled by fitting the data using the Bloch-Gruneisen formulation with certain simplifying assumptions. Good agreement between the calculated and experimental resistivity-temperature curves, and between calculated and experimental values of residual resistivity and Debye temperature, were obtained. From the model, parameters such as Fermi velocity, effective mass of a conduction electron, the number of electrons participating in conduction and electron mean free path have been calculated for the two phases. The calculations reveal that the mean free path is of the order of the lattice parameter in the case of alpha2, which leads to high resistivity and resistivity saturation. The resistivity of the alpha2 phase is also higher than that of the gamma phase due to the fact the Fermi velocity of the electrons is lower, effective electron mass higher and fewer electrons participitate in conduction. These factors, coupled with hybridization and localization effects, cause the different electrical resistivity behavior of the two phases.

11:20 AM

THE EFFECT OF IMPURITY ON THE MECHANICAL AND THE ELECTRICAL PROPERTIES OF Cu-Cr IN SITU COMPOSITE: *Eiju Takakura*[1]; Kuniteru Mihara[2]; Hirowo G. Suzuki[1]; [1]National Research Institute for Metals, Materials Processing Division, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]The Furukawa Electric Company, Ltd., Metal Research Center, No. 5 Research Section, 500 Kiyotaki, Nikko, Tochigi 321-1493 Japan

Cu-15wt%Cr in situ composite that has high strength and high electrical conductivity was developed, but it was not still economical because of its high purity ingredients. Lowering purity of ingredients is one of the candidates to decrease the production costs. In this study, we investigate the effect of impurity on the mechanical and the electrical properties of Cu-Cr in situ composite using two nine level of purity ingredients. Main impurity was Fe and it was scavenged into the Cr fiber. Vickers hardness of the cold drawn low purity material was 10Hv higher than the high purity one. This can be attributable to the hardening of Cr fiber. The electrical conductivity of the low purity material was almost the same as of the high purity one. It was clarified that the low purity materials can be utilized as the ingredient for the composite.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Metals Extraction and Smelting

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

Wednesday AM Room: 2
March 3, 1999 Location: Convention Center

Session Chairs: Hong Yong Shon, University of Utah, Salt Lake City, UT 84112-1183 USA; Stavros A. Argyropoulos, Dept. of Metall. and Mats. Sci., Toronto, Ontario M5S 3E4 Canada

8:30 AM
STUDY ON COMBUSTION OF BURNERS IN BLAST FURNACE STOVES: *Youduo He*[1]; [1]Baotou University of Iron & Steel Tech., Baotou, Inner Mongolia 014010 P.R. China

Burners are very important facilities for blast furnace stoves to raise hot-blast temperature up to 1200°. Nowadays, since the fuel being combusted in stoves is blast furnace gas only, to provide a suitable length of flame in combustion chambers of stove is drawing more attention. The length of flame should satisfy that the location of highest temperature of flue gas after full combustion is in the dome. The length of flame must be not too long so that part of the gas combustion does take place in upper part of checkerwork which will cause damage, subside and displacement of that part of checkerwork. Since the heat content of blast furnace gas is going down and the volume of gas passed through the burner become very large, the over long length of flame is often happened in practice. The length of flame should also not be too short because very large temperature gradient will cause large heat stress and the life of chamber wall be shorter. The turbulent diffusion flame of ceramic burners were simulated by three dimensional turbulent combustion programs, Which contained two equation models for turbulence, mixture fraction f and concentration fluctuation g for diffusion flame. Three kind of ceramic burners has been studied. They were common type of two rings (one ring linked with another), Hoogovens and a new burner designed by us. The flow pattern, the concentration and temperature distributions, the shape of flame, and the average length of flame with different conditions have been obtained. Some useful results can be extracted for practice. Besides, a pilot experiment of the new burner has been carried out at situ of Anshan Steel Complex to compare with the simulation results. The new ceramic burner has been accepted by Anshan Steel Complex and will put in practice at blast furnace number 4 which has 1000 cubic meters of working volume at the end of this year.

8:50 AM
MODELLING GAS INDUCED CONVECTION IN ALUMINA REDUCTION CELLS: *Knut Halvard Bech*[1]; Pål Tetlie[1]; *Asbjørn Solheim*[1]; *Torstein Haarberg*[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2b, Trondheim N-7034 Norway

Experiments were conducted in a water model of a two-dimensional (2D) section of a typical reduction cell. The metal-bath interface was modelled by a solid, non-deformable surface, so that convection was restricted to the "bath" (water) phase. Nitrogen gas flowed through a semi-permeable membrane under the model anodes, inducing an approximately two-dimensional circulation pattern in the side channel. The mean velocities and fluctuations were measured applying Laser Doppler Velocimetry. Side channel width, bath height and total gas flow rate were varied between the different experiments. A computer model of the experimental setup was established in the computational fluid dynamics (CFD) program Fluent. The bubbles were modelled as discrete, buoyant particles. However, due to the strongly localized momentum transfer and some physical shortcomings in the Fluent model, an adapted version of the program was constructed, incorporating the following changes: 1) The mass middle point of the bubbles were forced to at a distance away from the anode due to a repelling potential. 2) The momentum transfer was distributed over a region corresponding to the anticipated bubble size. 3) The momentum transfer was reformulated, ensuring conservation of momentum. The modified model reproduced the experimental data both qualitatively and quantitatively, except in narrow channels (10 cm width), where the real bubbles are of the same size as the channel width. After verifying the CFD model for the 2D flow situation, the model was applied in 3D computations, predicting the steady state flow around and between prebaked anodes. Results from the 3D model computations are presented.

9:10 AM
APPLICATION OF A REACTING CFD MODEL TO DROP TUBE KINETICS AND SMELTER SIMULATIONS: *B. R. Adams*[1]; K. A. Davis[1]; M. P. Heap[1]; A. F. Sarofim[1]; G. A. Eltringham[2]; A. A. Shook[2]; [1]Reaction Engineering International, 77 West 200 South, Suite 210, Salt Lake City, UT 84101 USA; [2]BHP, 7400 North Oracle Rd., Suite 200, Tucson, AZ 85704 USA

This paper discusses the use of a reacting CFD model to determine chalcopyrite kinetics in a drop tube furnace and to predict the reaction of a chalcopyrite concentrate in the reaction shaft of an industrial smelter. Drop tube furnaces are commonly used to derive kinetic parameters on pyrolysis and oxidation of different grade ores, since they can provide well-defined temperatures, oxygen concentrations, and residence times. For injection of particles at very low concentrations the gas concentration and temperature are not appreciably affected by the injected particles. Only in-furnace optical observations via pyrometry or high-speed photography or analysis of extracted particles by electron microscopy can be conducted at mass flow rates which are low enough to neglect perturbations of the gas temperature or concentration. At the higher concentrations needed for chemical or instrumental analysis, significant perturbations of the composition and temperature of the gas in the drop tubes can be expected. For such cases, reacting CFD codes can be applied to determine the consequences of the interaction of gas with particles and therefore improve the ability to derive kinetic parameters that take into account the different temperature and oxidation histories that different particles will see. This paper describes the application of such a model to derive improved kinetic parameters for the pyrolysis and oxidation of chalcopyrite. The improved kinetic parameters derived from the drop tube studies are then used to simulate chalcopyrite concentrate reactions in the reaction shaft of an industrial smelter. The reaction shaft model includes the effects of turbulent fluid mechanics, entrained flow mixing, turbulent particle dispersion, heterogeneous particle reactions, radiative and convective heat transfer, and surface and bath deposition rates. Particle reaction and composition characteristics are predicted as a function of particle trajectory and deposition and are used to aid in evaluating shaft performance.

9:30 AM
A 3-D COMPUTER MODEL OF THE FLASH CONVERTING FURNACE SHAFT: *Perez-Tello Manuel*[1]; *Hong Yong Sohn*[2]; Philip John Smith[1]; Kirsi M. Riihilahti[2]; [1]University of Utah, Dept. of Chem. and Fuels Eng., Salt Lake City, UT 84112 USA; [2]University of Utah, Dept. of Metallurgical Engineering, 412 William Browning Building, Salt Lake City, UT 84112 USA

A three-dimensional computer model for the Kennecott-Outokumpu flash converting process for copper matte is presented. The model incorporates the transport of momentum, heat, mass, and reaction kinetics between the gas and particles in a particle-laden turbulent gas jet. The standard k-e model is used to describe gas-phase turbulence in an Eulerian framework. The particle-phase is treated from a Lagrangian

viewpoint which is coupled to the gas-phase through the source terms in the Eulerian gas-phase governing equations. Matte particles were represented as Cu2S.yFeSx. Based on experimental observation, the oxidation products were assumed to be Cu2O, CuO, Fe3O4, and SO2. A reaction mechanism involving the external mass transfer of oxygen to the particle surface and diffusion of the oxygen through the oxide layers of Cu2O/Fe3O4 and CuO/Fe3O4 is proposed. Predictions of the computer model were compared with the experimental data collected in a large laboratory furnace. A reasonable agreement between the model predictions and the measurements was obtained in terms of fractional conversion of the oxidation reactions and of sulfur remaining in the reacted particles. The simulation of an industrial flash converting furnace was also performed. Higher oxidation rates, more even distribution of particles, and thus a more efficient use of the reactor volume are predicted with a burner having a distributor cone, than with a single-entry burner.

9:50 AM

MODEL STUDY ON DISPERSED-PHASE HOLDUP IN FERROALLOY REFINING PROCESSES: *G. Akdogan*[1]; B. Yoruc[1]; R. H. Eric[1]; [1]University of the Witwatersrand, School of Process and Materials Engineering, Private Bag 3, Johannesburg, Wits 2050 South Africa

An experimental study has been performed to investigate the dispersed phase holdup induced by a high-strength submerged gas injection in a bottom blown air-stirred one-seventh water model of CLU (Creusot-Loire Uddeholm) reactor usine two different tuyere configurations. The air flow rates varied from 0.00599 m3/s to 0.01312 m3/s. In these series of experiments, water height and kerosene height were kept constant at 0.23m and 0.02m respectively. The dispersed phase holdup was determined at various gas flow rates and nozzle orientations. The dispersed phase holdup increased with gas injection rate and decreased with vertical distance from the original interface. At a constant vertical distance, the radial distribution of the holdup was found to increase with increasing gas flow rate. At constant vertical distance and gas injection rate the dispersed holdup increased with increasing radial distance from the centerline. At constant gas flow rate, as the vertical distance increased in the centeline, off-center orientation of nozzles displayed higher values of the dispersed phase holdup than those of the center configuration. The variation of percentage of dispersed phase holdup with respect to vertical radial distances was also discussed in terms of the swirling action of the bath. The maximum dispersed phase holdup of 22% was achieved at the interface (vertical distance of zero) with a radial distance of 5cm from the centerline using 0.01081m3/s gas flow rate for off-center configuration of the nozzles. The minimum dispersed phase holdup of 3.64% was seen at a vertical distance of 0.22m from the interface with a radial distance of 10cm from the centerline at a flow rate of 0.00599m3/s for center configuration of nozzles.

10:10 AM BREAK

10:30 AM

FLOW VISUALIZATION AND TEMPERATURE MEASUREMENTS CLOSE TO THE ARC ATTACHMENT ZONE OF A LABORATORY SCALE DC FURNACE FOR SLAG CLEANING: *Adrian Christian Deneys*[1]; David G. C. Robertson[1]; [1]University of Missouri - Rolla, Center for Pyrometallurgy, 215 Fulton Hall, Rolla, MO 65409-1460 USA

Documentation of fluid flow phenomena on the surface of a molten slag have been made using video photography in a laboratory scale D.C. arc furnace. In addition, temperatures have been measured close to the D.C. arc attachment zone. A molybdenum thermocouple (with a 90Ɣ bend, 50 mm from tip) was inserted under the electrode, 5-30 mm below the surface of the molten slag bath. Two other temperatures were simultaneously measured; one submerged approximately 70 mm in the bath, against the crucible wall, and another 5-10 mm below the surface of the bath (also against the crucible wall). Initially induction heating melted approximately 2 kg of nickel flash smelter slag which has a liquidus temperature around 1150Ɣ. Transient temperature measurements were then obtained during discrete periods with the plasma arc turned on. Temperatures (and rates of temperature rise) were found to be a function of the depth of the thermocouple in the bath. As the depth decreased, the peak temperatures increased. With 3.3 kW of D.C. arc power supplied for 35 seconds (and the bent molybdenum thermocouple submerged 10 mm beneath the surface). The temperature under the electrode tip rose from 1181Ɣ to 1302Ɣ in 45 seconds. At the same power level, the temperature stabilized at 1420Ɣ on the surface of the bath (at the wall). Directly below the electrode (10mm beneath the slag surface), the temperature stabilized at 1398Ɣ. This work has been conducted to quantify the temperatures, fluid flow and reductant behavior present in the arc attachment zone of a D.C. arc furnace, and has relevance to optimizing furnace design for pyrometallurgical slag cleaning processes. A description of the experimental apparatus and experimental results will be presented.

10:50 AM

FLUID FLOW MODELING IN ELECTRIC ARC FURNACES: L. P. Gu[1]; *G. A. Irons*[1]; [1]McMaster University, Dept. of Mats. Sci. and Eng., 1280 Main St. West, Hamilton, Ontario L8S 4L7 Canada

A 1/3-scale "thin-slice" model of an electric arc furnaces was developed so that gas could be injected to simulate slag foaming. The influence of arc jets, oxygen lancing and bottom bubbling was also simulated. Liquid velocities were measured by a white light particle image velocimetry system. The results are compared with mathematical models.

11:10 AM

PHYSICAL AND MATHEMATICAL MODELLING INVOLVING EXOTHERMIC REACTIONS IN LIQUID METALS: *Stavros A. Argyropoulos*[1]; Hongfa Hu[2]; Stephan Ferenczy[3]; [1]University of Toronto, Dept. of Metall. and Mats. Sci., Walberg Bldg., Room 142, 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Meridian Magnesium North America, Strathroy, Ontario N7G 1H4 Canada; [3]FUCHS Systems, Inc., Salisbury, NC USA

Physical as well as mathematical modeling was carried out to investigate the exothermic mass transfer in liquid metals systems. In these systems, the heat, mass and momentum transfer is deeply coupled. Moreover, these deeply coupled systems are further complicated by the presence of a moving boundary. In the physical model used, ice cylinders were immersed into sulfuric acid solutions, because the intermixing of ice with sulfuric acid is very exothermic. In this low temperature system, velocity and temperature measurements in the sulfuric acid were made. In addition, measurements of ice melting in the sulfuric acid were carried out. The mathematical model developed solved the heat, mass and momentum equations numerically, utilizing the control-volume finite difference approach. The enthalpy method was adopted to track the moving boundary. Predictions made by the mathematical model were in close agreement with the results obtained by the physical model. The results from the mathematical model were further validated with high temperature liquid metals. Specifically, data were obtained in an experimental setup involving (a) dipping silicon cylinders into high carbon liquid iron and (b) dipping nickel cylinders into liquid aluminum. Results from these experimental studies supported the predictions that had been made using the mathematical model.

11:30 AM

MODEL STUDIES OF HEAT TRANSFER AND FLOW IN SLAG-CLEANING FURNACES: S. Kang[1]; *David Gordon Campbel Robertson*[1]; [1]University of Missouri Rolla, Dept. of Metall. Eng., 215 Fulton Hall, Rolla, MO 65409-1460 USA

Slag cleaning furnaces require extensive wall cooling because they involve the use of a superheated slag bath. This is in direct contrast to smelting furnaces, where the wall is usually (but not always) protected by "banks" of solid charge. In order to understand the heat transfer and flow in slag cleaning furnaces a number of model studies have been carried out using molten wax to simulate the slag. Wax is a good model fluid because, like slag, it has low Prandtl number. Wax models cannot simulate the behavior close to the electrodes (DC arc or AC submerged), but they can and do give a good simulation of the flow and heat transfer at the wall. An understanding of these phenomena is of great importance is the design of cooling panels in the walls of the furnace. Since superheated molten slag will eventually wear away any reasonably priced refractory bricks, the wall cooling must be designed to form a "freeze lining". The heat flux density (Q/A) required to do this is given by Q/A = h*(deltaT), where h is the heat transfer coefficient in the slag phase at the wall, and (deltaT) is the superheat (bulk slag temperature - liquidus

temperature). Both Q/A and h vary greatly with position and both are usually a maximum at the slag line. In the experimental work molten wax baths were heated by block heaters to provide similar energy densities (MW/m3) to those encountered in commercial slag cleaning furnaces. This procedure was considered to be valid since most of the electrical energy into a furnace bath is dissipated as heat. Gas stirring was used to simulate the gas evolved at the electrode in real furnaces - the quantity of this gas could be estimated from a knowledge of electrode consumption. The similarity criterion was equal gas flow per unit area of surface, in model and prototype. Heat transfer coefficients were measured in the models as a function of power density, gas stirring, superheat, etc., and could be related to the full scale operations by the use of dimensional analysis. The flows, heat transfer coefficients, etc., were also measured in a simple rectangular geometry and the flow was modeled using the COMPACT-2D software package. Good agreement was obtained between the experimental data and the model predictions.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Solidification and Casting: Computation and Experiments

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

Wednesday AM Room: 5B
March 3, 1999 Location: Convention Center

Session Chairs: Doru M. Stefanescu, The University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Robert A. Stoehr, University of Pittsburgh, Dept. of Mats. Sci. and Eng., Pittsburgh, PA 15261 USA

8:30 AM
FLUID FLOW PHENOMENA AND SOLIDIFICATION BEHAVIOR DURING THE CASTING OF A Pb-Sb-Sn ALLOY: *Matthew John M. Krane*[1]; [1]Purdue University, School of Materials Engineering, 1289 MSEE Bldg., West Lafayette, IN 47907 USA

Using a new numerical treatment of transport phenomena occurring during the solidification of ternary metal systems, the macrosegregation patterns and convective behavior of a Pb-5wt%Sb- 35wt%Sn alloy are studied. At this composition, the solidification which occurs while the local liquid composition is in the (Pb)-(SbSn) binary eutectic trough has a significant effect on these phenomena. The effects of this solidification path on fluid flow and macrosegregation are compared to the more familiar binary alloy patterns. The effects of solid particle transport and mushy zone permeability are also evaluated. The final predictions of macrosegregation are compared to measured composition profiles.

8:50 AM
GRAIN STRUCTURE EVOLUTION IN Al-Cu ALLOYS SOLIDIFYING DURING UNIDIRECTIONAL FLOW OVER A CHILL: COMPARISON OF A CA MODEL WITH EXPERIMENTS: *Robert A. Stoehr*[1]; Chidchai Loyprasert[1]; [1]University of Pittsburgh, Dept. of Mats. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

A computer model has been developed to predict grain structure in alloys solidified under unidirectional flow using a combination of three techniques — solution of the equations for flow of an incompressible fluid bounded by curved surfaces by the SOLA-SURF algorithm, calculation of heat flow by a finite volume method, and simulation of grain formation by a two-dimensional cellular automaton (CA) model. The changing rigid surface has been taken into account. Heterogeneous and homogeneous nucleation are considered, and the growth kinetics of a dendrite tip are evaluated by the Kurz-Giovanola-Trived (KGT) model. These calculations were applied to solidification of an Al-4.5% Cu alloy flowing over a chill on the bottom of a channel, a system for which experimental results were available. Using a variety of flow rates and superheats, agreement between the computed and experimental results were very good including transitions between columnar and quasi-equiaxed grains, the aspect ratio of the grains, and the inclination of the grains to the flow.

9:10 AM
AN EXPERIMENTAL STUDY OF THE SOLIDIFICATION OF PURE SUCCINONITRILE AND A SUCCINONITRILE-ACETONE ALLOY: James E. Simpson[1]; Henry C. de Groh[2]; Mark McDowell[2]; *Suresh V. Garimella*[1]; [1]University of Wisconsin - Milwaukee, Mech. Eng. Dept., P.O. Box 784, Milwaukee, WI 53201 USA; [2]NASA Lewis Research Center, MS 105-1, Cleveland, OH 44135 USA

An experimental study of the horizontal Bridgman growth of pure succinonitrile (SCN) and of a succinonitrile-1.0 mol.% acetone alloy (SCN-1.0 mol.% ACE) has been performed. Experiments involving both a stationary thermal field (no-growth case) and a translating thermal field (growth case) were conducted. Growth rates of 40 and 2 micro-m/s were investigated. For the pure SCN experiments, the velocity field in the melt was measured using Stereo Imaging Velocimetry (SIV). Measurements indicate that a primary longitudinal convective cell is formed. The maximum magnitude of the convective velocity in the pure case was 1.6 mm/s for both no-growth and steady growth. The shape of the solid/liquid interface was also quantitatively determined. The solid/liquid interface was stable (non-dendritic and non-cellular); however, it was not flat. Rather, it was significantly distorted by the influence of convection in the melt and, for the growth case, by the moving temperature boundary conditions along the ampoule. It was found that the interface shape and position were highly dependent on the alignment of the ampoule in the apparatus. Consequently, the ampoule was carefully aligned for all experiments. The values for front location agree with those determined in previous experiments. For the alloy experiments, the solid/liquid interface was determined to be unstable at growth rates of greater than 2.8 micro-m/s, but stable for the no-growth and growth at 2 micro-m/s cases. When compared to the shape of the pure SCN interface, the alloy interface forms closer to the cold zone, indicating that the melting temperature has been suppressed by the addition of the alloying element. Temperature measurements were taken at various locations on the outside of the ampoule, in order to determine the thermal boundary condition on the ampoule. The resulting thermal profiles are presented in detail. It is intended that the interface shape, thermal boundary condition and velocity field data presented in this paper be used to test numerical simulations.

9:30 AM
CONVECTION INDUCED PATTERN FORMATION IN DIRECTIONAL SOLIDIFICATION: *Prantik Mazumder*[2]; Rohit M.I.K. Trivedi[1]; [1]Ames Laboratory, US-DOE, Dept. of Mat. Sci. and Eng., Ames, IA 50011 USA; [2]Iowa State University, Dept. of Mech. Eng., Ames, IA 50011 USA

Numerical analysis is carried out to examine the effects of thermosolutal convection on formation of patterns in directionally solidified binary alloys. A finite-difference analysis is used for dynamic modeling of a prototype Vertical Bridgman system that takes into account heat transfer in the melt, crystal, and the ampoule; melt flow and solute transport. Actual temperature data from experimental measurement are used for accurately describing the thermal boundary conditions. The convection is found to consist of two vertically stacked torroidal cells. The flow in the upper cell is upwards along the ampoule wall and is driven by the large radial temperature gradient at the junction of the hot zone and the adiabatic zone. The cell adjacent to the solid

liquid interface has complex dynamics depending on the location of the interface relative to the furnace configuration, thermal conductivities of crystal, melt and the ampoule, and the thermal and solutal Rayleigh numbers. The flow range from minimal convection to steady cellular and multi-cellular convection to periodic convection to quasiperiodic to intense turbulent mixing. The convection in binary alloys with destabilizing solutal effect on convection (for which the rejected solute is lighter than the solvent, e.g. Pb-Sn and Pb-Bi) usually undergoes rapid transition to intense turbulent convection from steady cellular/multicellular convection. In alloys with stabilizing solutal effect (the rejected solute is heavier than the solvent, e.g. Al-Cu and Sn-Cd) the convection evolves through a distinct set of bifurcations and the intensity of mixing is relatively smaller. For peritectic alloys (Sn-Cd, Pb-Bi) a two phase oscillating microstructure may develop in response to the oscillating segregation profile. For single phase alloys a large lateral disorder in cell spacing may occur in low convective systems (Al-Cu) compared to high convective system (Pb-Bi). The numerical calculations are found to agree well with experiments done with the systems mentioned.

9:50 AM

SINGLE AND MULTIPHASE FLOWS GENERATED BY ROTATING ELECTROMAGNETIC FIELDS: APPLICATION TO THE CRYSTALLIZATION OF THIXOTROPIC ALUMINUM ALLOYS SLURRIES AND COMPOSITES: *Charles Noël Vivés*[1]; [1]Université d'Avignon, Laboratoire de Magnétohydridynamique, 33, Rue Pasteur, Idem, Avignon, Vaucluse F84000 France

The elaboration of thixotropic alloys slurries and metal matrix composites requires a very vigorous stirring, often produced by rotating electromagnetic fields. In the case of relatively small section ingots of light metals (aluminum or magnesium alloys), the free surface deepness can reach several meters, and becomes then unacceptable. Indeed, a deep vortex is responsible for an undesirable entrapment of gas and oxides into the bulk of the melt during the mixing and solidification processes. This circumstance leads to porosity in casting and, in the case of composites, inhibits wetting at the slurry-particle interface to the detriment of bonding properties. The main goal of this experimental work was to overcome these specific and serious drawbacks through the use of two three-phase motor stators able to produce either single or contra-rotating flows. In this investigation, the working fluids were mercury and aluminum alloys. The approach was to measure separately the electromagnetic parameters, namely the magnetic field and current density components as well as the phase angle between these periodic vectors, and next to obtain the electromagnetic force field from these parts. Then, these results were connected with the experimentally determined free surface shape, velocity field and turbulence. Satisfactory results concerning the microstructure of solidified aluminum alloys slurries and aluminum alloy matrix composites (homogeneity, crystal shape, grain size, fraction of primary solid, and distribution of SiC particles) were obtained.

10:10 AM BREAK

10:30 AM

A PIV-BASED PHYSICAL MODEL FOR OSCILLATING GRAVITY INDUCED CONVECTION: M. Higgins[1]; B. R. Ramaprian[1]; *Ben Q. Li*[1]; [1]Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99163 USA

In designing metals processing systems for space applications, oscillation convective flows induced by transient g-jitter or residual gravity field must be considered. This paper presents a ground based experimental study of oscillating convection, with an intention to simulate some of basic g-jitter effects. Towards this end, a physical model is developed. The model uses water as working fluid and oscillating wall temperature is applied The oscillating convective flows in the model are visualized using a laser-based PIV system. With the model, various parameters affecting flows can be studied. Measured results for convective flows will be presented and compared with those obtained from a finite element model.

10:50 AM

GRAVITATION EFFECTS ON MACROSEGREGATION - EXPERIMENTS AND COMPUTATIONAL MODELING: *Jose Leon-Torres*[1]; Doru M. Stefanescu[1]; Subhayu Sen[2]; Peter A. Curreri[3]; [1]University of Alabama, Solidification Laboratory, College of Engineering, P.O. Box 870202, Tuscaloosa, AL 35487 USA; [2]USRA, NASA Marshall Space Flight Center, ES75, Huntsville, AL 35812 USA; [3]NASA Marshall Space Flight Center, ES75, Huntsville, AL 35812 USA

Experiments were performed under terrestrial gravity (1g) and during parabolic flights (10-2 g) to study the solidification and macrosegregation patterns of Al-Cu alloys. Alloys having 2% and 5% Cu were solidified against a chill at two different cooling rates. Microscopic and Electron Microprobe characterization was used to produce microstructural and macrosegregation maps. In all cases positive segregation occurred next to the chill because shrinkage flow, as expected. This positive segregation was higher in the low-g samples, apparently because of the higher heat transfer coefficient. A 2-D computational model was used to explain the experimental results. The continuum formulation was employed to describe the macroscopic transports of mass, energy, and momentum, associated with the solidification phenomena, for a two-phase system. The model considers that liquid flow is driven by thermal and solutal buoyancy, and by solidification shrinkage. The solidification event was divided into two stages. In the first one, the liquid containing freely moving equiaxed grains was described through the relative viscosity concept. In the second stage, when a fixed dendritic network was formed after dendritic coherency, the mushy zone was treated as a porous medium. The macrosegregation maps and the cooling curves obtained during experiments were used for validation of the solidification and segregation model. The model can explain the solidification and macrosegregation patterns and the differences between low- and high-gravity results.

11:10 AM

VISUALIZATION OF DYNAMIC BEHAVIOR OF IMMISCIBLE LIQUIDS DURING SOLIDIFICATION OF MONOTECTIC SYSTEMS: *J. B. Andrews*[1]; L. J. Hayes[1]; L. J. Little[1]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., 1150 10th Ave. S., BEC 254, Birmingham, AL 35294 USA

This presentation will cover results obtained from the microgravity glovebox investigation Wetting Characteristics of Immiscibles, which flew aboard the space shuttle Columbia during the USMP-4 flight in November and December of 1997. In this investigation, twelve compositions within the transparent succinonitrile-glycerine immiscible system were studied. The main intent of the investigation was to directly observe the events which lead to gross phase separation during microgravity processing of many immiscible alloys. One theory proposes this separation occurs when one of the immiscible liquid phases perfectly wets the walls of the container in which the alloys are processed. The wetting characteristics were varied in this investigation by varying the sample compositions. Perfect wetting of the container walls by the minor liquid phase was observed in several cases. In addition, flow was observed in some of the sample cells which appeared to be associated with the coalescence of droplets. This flow was particularly interesting in cases where the two immiscible liquids were present in roughly equal proportions. Video demonstrating the wetting behavior and the flows observed during this microgravity glovebox investigation will be shown as part of this presentation.

11:30 AM

THE EFFECT OF FLUID FLOW ON A SEDIMENTING PARTICLE NEAR A SOLIDIFYING INTERFACE: *Anthony Michael John Davis*[1]; [1]University of Alabama, Dept. of Math., 345 Gordon Palmer Hall, P.O. Box 870350, Tuscaloosa, AL 35487-0350 USA

The doping of binary substances with a small volume fraction of second phase inclusions is used in the solidification processing of MMC's to improve the material properties of the final cast. The presence in the melt of a solid particle of different conductivity causes modification to the temperature and solute concentration fields which together displace the interface through distances assumed here to be small enough for linearization. The determination of the interface profile from the compatibility condition is somewhat complicated. The interface displacement also contributes to the fluid flow in the melt and, in particular, to the velocity of sedimantation of the particle.

GENERAL ABSTRACTS: Session 7 - Issues in Materials Science: Thin Films & Temperature Sensing

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

Wednesday AM Room: 12
March 3, 1999 Location: Convention Center

Session Chairs: Dr. Mark Palmer, Virginia Commonwealth University, Dept. of Mech. Eng., Richmond, VA 23284-3015 USA; Elliot Schwartz, The Gillette Company, South Boston, MA 02127-1096 USA

8:30 AM

A NOVEL TECHNIQUE FOR NON-CONTACT TEMPERATURE MEASUREMENT OF SEMICONDUCTOR WAFERS: *Assaf Thon*[1]; Evgeny D. Glazman[1]; [1]3T, Theradion Industrial Park, Misgav 20179 Israel

Single wafer equipment semiconductor manufacturing performs a variety of thermal deposition and annealing processes. The operating temperature range is typically between 350°C to 1200°C and the pressure varies between 10-5 Torr to near atmospheric pressure. A firm window of working parameters characterizing every specific process should be maintained within a pre-set and usually small tolerance. While much effort is invested in widening the window of parameters, it is still necessary to keep the temperature error as small as some fractions of a percent of the set point. The trends towards smaller device dimensions (0.25 micron and below) and bigger wafer size (300 mm) impose clear standards on the equipment performance metrics. In this work we represent results of a novel technique for precise and accurate measurement of semiconductor wafers temperature. The technique combines multispectral passive and active methods. The true emissivity of the wafer is calculated dynamically at high rat. An accuracy of better than 1% was demonstrated for Si wafers with varied emissivity between 0.2 to 0.9, at the temperature range of 400°C to 850°C. Current work is now being done on increasing the accuracy to better than 0.5% for all emissivity range and up to 1200°C.

8:55 AM

CARBON FIBER EPOXY-MATRIX COMPOSITE FOR TEMPERATURE SENSING: Shuokai Wang[1]; *Deborah D. L. Chung*[1]; [1]State University of New York at Buffalo, Mechanical and Aerospace Engineering, 608 Furnas Hall, Amherst, NY 14260 USA

A polymer (epoxy)-matrix composite with the top two laminae of continuous carbon fibers in a crossply configuration was found to be a temperature sensor. Each junction between crossply fiber tow groups of adjacent laminae is a sensor, while the fiber groups serve as electrical leads. A junction array provided by two crossply laminae allows sensing of the temperature distribution. The contact electrical resistivity of a junction decreases reversibly upon heating (whether using light or hot plate to heat), due to the activation energy involved in the jump of electron across the junction. The contact resistivity decreases with increasing pressure during composite fabrication, due to the increase in pressure exerted by fibers of one lamina on those to the other lamina. The absolute value of the fractional change in contact resistivity per degree C increases with increasing pressure during composite fabrication, due to decrease in composite thickness, increase in fiber volume fraction and consequent increases in interlaminar stress and activation energy. A junction between unidirectional fiber tow groups of adjacent laminae is much less effective for temperature sensing, due to the absence of interlaminar stress.

9:20 AM

THERMODYNAMIC STABILITY OF SELECTED TERNARY COMPOUNDS IN THE Cu-In-Se SYSTEM BY A SOLID ELECTROLYTE EMF TECHNIQUE: *Pankajavalli Nagarajan*[1]; Muhsin Ider[1]; Chih-hung Chang[1]; Timothy J. Anderson[1]; [1]University of Florida, Dept. of Chemical Engineering, P.O. Box 116005, Gainesville, FL 32611 USA

Polycrystalline thin films of $CuInSe_2$ are a promising material for high efficiency photovoltaic solar cells in low-cost power generation applications. The four ternary compounds $CuInSe_2$, $Cu_2In_4Se_7$, $CuIn_3Se_5$ and $CuIn_5Se_8$ were reported to be contained in the Cu_2Se-In_2Se_3 pseudobinary system and can be important in the synthesis of high efficiency cells. The phase equilibria and thermochemistry of the Cu-In-Se system, however, is not well established. In particular, ambiguity exists about the location of the phase boundary between the chalcopyrite(α) and sphalerite(δ) phases of $CuInSe_2$. In addition, no thermodynamic data are available on the stability of these ternary phases, except the standard entropy and enthalpy of formation of $CuInSe_2$. The EMF of the following galvanic cells were measured: $W|In(l),In_2O_3(s)||YSZ||In_2O_3(s)$, $Cu_2Se(s)$, $Cu(s)$, $CuInSe_2(\alpha$ or $\delta)|C|W$ (I) $W|In(l),In_2O_3(s)||YSZ||In_2O_3(s)$, $Cu_2In_4Se_7(s)$, $CuInSe_2(\alpha$ or $\delta)$, InSe(s or l)$|C|W$ (II) $W|In(l),In_2O_3(s)||YSZ||In_2O_3(s)$, $CuIn_5Se_8(s)$, $CuIn_3Se_5(s)$, InSe(l)$|C|W$ (III). Measurements on the three cells were made using a 15 mol% yttria stabilized zirconia solid electrolyte in the temperature ranges 949 to 1150, 868 to 1179 and 977 to 1145K respectively. Combining literature data for the standard Gibbs energies of formation of Cu_2Se, InSe and $CuIn_3Se_5$ with the standard Gibbs energy changes determined from the above cells, the standard Gibbs energy of formation of $CuInSe_2$, $Cu_2In_4Se_7$ and $CuIn_5Se_8$ were determined to be $\Delta G°f\ CuInSe_2\ (\alpha) \pm 0.51$ (kJ mol^{-1}) = - 35.60 - 0.1718 T(K) $\Delta G°f\ CuInSe_2\ (\delta) \pm 0.18$ (kJ mol^{-1}) = - 13.91 - 0.19296 T(K) $\Delta G°f\ Cu_2In_4Se_7(s) \pm 1.94$ (kJ mol^{-1}) = - 596.88 - 0.04972 T(K) $\Delta G°f\ CuIn_5Se_8(s) \pm 1.97$ (kJ mol^{-1}) = - 1086.56 + 0.31051 T(K). From the above results, the α to δ phase transition temperature and the enthalpy of transition for $CuInSe_2$ were determined to be 1025K (from cell I), 1050K (from cell II) and 21.7 kJ mol^{-1} respectively. These results have been included in an optimization of the Cu_2Se-In_2Se_3 pseudobinary phase diagram. The implications for processing $CuInSe_2$ solar cells will be discussed.

9:45 AM

THE METALLURGY OF ARTIFICIAL DIAMOND FILM PLANARIZATION: *Khershed P. Cooper*[1]; James E. Butler[2]; [1]Naval Research Laboratory, Materials Science & Technology, Code 6321, 4555 Overlook Ave., SW, Washington, D.C. 20375-5343 USA; [2]Naval Research Laboratory, Chemistry Division, Code 6174, 4555 Overlook Ave., SW, Washington, D.C. 20375 USA

Potential applications of artificial diamond films are in cutting tools, thermal management, electronic devices and optical windows. To be functional, the rough faceted growth surface of the diamond film has to be planarized. One method of accomplishing this is thermochemical planarization. In our experiments, we held the diamond film in intimate contact with a flat piece of iron at a temperature below the Fe-C eutectic. As carbon atoms from the diamond diffused into the iron, the growth facets were removed and planarization occurred. We studied the effect of applied load, hold time and temperature on the planarization process. While the effect of load was linear, the influence of time and temperature appeared parabolic. Examination of the diamond/iron interface showed that as carbon diffusion proceeded, thin graphite and cementite films formed between the diamond and the iron. After planarization, the diamond film retained its structure, but the dissolution of carbon into the iron resulted in transformation products such as very finely spaced pearlite. The presence of cementite along the grain boundaries deep into the iron piece showed that carbon diffused to a greater length along grain boundaries. But due to the coarsened grain structure, the grain boundaries were too few to accelerate the diffusion process. Quantification of the faceted growth surface and estimations of the diffusion rates in Fe-C alloys helped understand the process kinetics.

10:10 AM
DEVELOPMENT OF ROUGHNESS IN ULTRATHIN ELECTRODEPOSITED TRILAYER FILMS: Rob Renner[1]; Bo Dou[1]; *Knona Liddell*[1]; [1]Washington State University, Chemical Engineering Dept., P.O. Box 642710, Pullman, WA 99164-2710 USA

Layered Co/Cu/Co and Ni/Cu/Ni films were electrodeposited onto a vapor deposited Cu seed layer on Si using the stepped potential, single bath technique and an unstirred aqueous electrolyte. In separate experiments, the thickness of each layer was varied between 2 and 20 nm. Atomic force microscopy was used to measure both the RMS peak height and the areal peak density; the substrate, single Co or Ni layers, and bi- and tri-layer samples were examined. The Co-Cu and Ni-Cu systems show qualitatively similar behavior. Increased peak height is linearly correlated with decreased peak density for the thinnest films, but neither height nor density is directly related to film thickness.

10:35 AM
MODELING ORIENTED NUCLEATION AND GRAIN GROWTH IN COPPER ELECTROCOATINGS: *O. B. Girin*[1]; [1]State Metallurgical Academy of Ukraine, Dept. of Physics, Prospekt Gagarina 4, Dnipropetrovak 32063 Ukraine

A thermodynamic and a mathematical model of texture formation in copper electrocoatings is discussed. The respective contributions of the grain boundary energy, the surface energy and the bulk energy to the driving force of texture formation in the coatings at the nucleation stage and using the grain growth of the major component are addressed. Estimates of shape anisotropy are given for texture nuclei and oriented grains in relation to anisotropy of electrocoating properties. Optimum process conditions are cited that allow the most developed texture and the most desired properties of copper electrocoatings.

11:00 AM
SOME ENGINEERING APPLICATIONS OF MATERIALS SCIENCE; SENSORS FOR HYDROGEN OR OXYGEN: David R. Morris, Dept. of Chemical Engineering, University of New Brunswick, Fredericton, NB E3B 5A3 Canada

This paper describes the exploitation of the properties of two materials for the development of chemical sensors: 1. The per fluorinated ionomer, Nafion H is a hydrogen ion conductor. This material property is utilized for the construction of a solid state electrochemical sensor for monitoring hydrogen in pipeline steel and for monitoring hydrogen or oxygen in high temperature water. The uptake of hydrogen into the pipeline steel at ambient temperature is strongly promoted by the presence of hydrogen sulphide. The sensor for application too high temperature (to ~500 K) water is a non-isothermal electrochemical cell with the reference electrode (Fe^{II}, Fe^{III} sulphate hydrates) at ambient temperature. Nafion is in the form of small diameter tube. The sensor voltage is a function of the dissolved hydrogen (or oxygen) concentration in the water and a function of the temperature at the sensing electrode. 2. The electrical resistance of palladium at a fixed temperature is a function of the concentration of hydrogen. This material property is utilized for the construction of a sensor for monitoring hydrogen (or deuterium) in high temperature (to ~570 K) water. Measurements of the electrical resistance of a palladium wire in hydrogen/inert gas mixtures and in hydrogen/water solutions permit determination of the Henry Law constant for hydrogen in water. Values of the Henry Law constant so determined are lower than values published in the literature, which were determined by equilibrium experiments.

GENERAL RECYCLING OF MATERIALS: Recycling of Aluminum and other Light Metals

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizers: Ilaria Accorsi, Chrysler Corporation, Product Quality, Toledo, OH 43606 USA; Isrun Bohlinger, Technical University of Berlin, Institute of Metallic Materials, Berlin D-10623 Germany

Wednesday AM Room: 1A
March 3, 1999 Location: Convention Center

Session Chairs: Gerrit H. Nijhof, Hoogovens , R & D, IJmuiden The Netherlands; Ray D. Peterson, IMCO Recycling, Irving , TX 75039 USA; Benji Maruyama, US Air Force, Research Lab., Wright Petterson AFB, OH 45433 USA

8:30 AM OPENING REMARKS

8:35 AM INVITED PAPER
UPGRADING OF NON-FERROUS METAL SCRAP, PARTICULARLY ALUMINIUM, FOR RECYCLING PURPOSES.: *Gerrit Nijhof*[1]; [1]Nijhof Consultancy, Heemsteedse Dreef 92, 2102 KN Heemstede The Netherlands

After the consumer phase of most products it is often difficult to separate the different metals present, particularly non-ferrous metals and stainless steels. A mixing of metal scrap will occur. When this mixture is directly melted a metal is produced with high amounts of impurities. These metals are suitable for application as casting alloys only. To prevent the mixing of scrap, separation at the source is required. Recently several techniques for separation of non-ferrous metals have become available. The following techniques will be discussed: 1. Eddy Current separation; to separate non-ferrous metals from a mixture of waste; 2. Fluid bed separation; to separate light and heavy metals; 3. Separation by image analysis; to separate cast and wrought alloys. Results of different recent experiments, using these techniques will be presented. However, when a separation of scrap is not possible a removal of undesired elements must be performed. Techniques, developed on laboratory scale have been presented at previous Light Metals Conferences and will be shortly reviewed.

9:00 AM
A SMALL-SCALE RECYCLING PROCESS FOR NON-FERROUS ALLOYS: *Richard Johnson*[1]; [1]TWI, Electron Beam, Friction & Forge Processes Dept., Abington Hall, Abington, Cambridge CB2 6AL UK

Large firms can invest in furnaces to recycle aluminium alloy scrap back into the production process, but smaller firms are less able to do so on economic grounds. They therefore obtain very low returns on their scrap arisings, as they are essentially paying for the transport of air and only some metal to the recyclers. Friction extrusion is one of a stable of friction technologies developed by TWI, which consolidates scrap directly into solid product. By generating the necessary heating locally by friction, the energy consumption of the process is very low, whilst the consolidation of the material is effected in the solid phase rather than by melting. This means there are minimal material losses from oxidation, and little fume generation when dealing with scrap contaminated by machining fluids, etc. The paper will review the process limitations and advantages, including the environmental benefits, and its applicability to a range of aluminium alloys and other non-ferrous materials.

9:25 AM
COLOR SORTING ALUMINUM ALLOY SCRAP FOR RECYCLING: *Rebecca K. Wyss*[1]; Paul B. Schultz[1]; [1]ALCOA, Technical Center, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

The ability to recycle and reuse scrapped materials increases in importance as the use of aluminum alloys in automotive and other applications continue to expand. Color sorting is a new process which has the capability to sort mixed alloy scrap so that it can be recycled into new wrought alloys. Aluminum manufacturers can use sorted scrap to make wrought alloys and replace high cost primary metal, currently used to produce wrought alloys, with lower cost metal units. Ultimately, the energy savings and economic and environmental benefits would be significant, substantially increasing the value of aluminum. The color sorting process involves chemically etching similar appearing scrap to color the scrap according to its chemical composition. The color differences are sufficient to allow optical recognition systems to automatically identify and separate scrap by alloy family for recycling. The distinct appearance of alloys from each of the 2xxx, 3xxx, 5xxx, 6xxx, and 7xxx alloy groups, which have been etched using commercially acceptable chemical treatments, will be presented.

9:50 AM BREAK

10:05 AM
DECOATING OF ALUMINIUM PRODUCTS AND THE ENVIRONMENT: *Richard Joseph Evans*[1]; Graham Guest[1]; Willis Bateman[1]; [1]Stein Atkinson Stordy, Ltd., Aluminium Division, Midland House, Ounsdale Rd., Wombourne, Wolverhampton WV5 8BY UK

This paper focuses on the environmental aspects of decoating aluminium products. Over the past ten years, Stein Atkinson Stordy have built up a considerable knowledge of the decoating process and its environmental criteria. The basic environmental standards applicable to decoating are outlined for both the European and American markets and their major differences highlighted. Types of scrap and their associated coating are discussed, together with typical emissions encountered and suitable methods of their environmental control. Aspects of plant design and operation, with typical field results are presented to reinforce the theoretical aspects of the body of the paper.

10:30 AM
BATH RECOVERY FROM CARBON DUST BY FROTH FLOTATION: *Juan J. del Campo*[1]; Mario Menéndez[2]; Juan M. Menéndez[2]; [1]Universidad de Oviedo, Dept. of Mats. Sci. and Eng. Metall., Avda. Manuel Llaneza 75, Gijon, Principado de Asturias 33208 Spain; [2]Universidad de Oviedo, Mining, Exploration and Operation, Escuela de Minas de Oviedo, Independencia 13, Oviedo, Principado de Asturias 33004 Spain

Carbon dust produced in the operation of aluminum electrolysis pots is a hazardous waste. It contains valuable fluorides and coke. There is a potential interest in the separation of both fractions in order to returnthe cryolite bath into the pots. A froth flotation process is proposed. The degree of liberation of carbon together with the operating conditions of the flotation plant are studied. Industrial experience at INESPAL METAL,S.A. with recycled bath is also presented.

10:55 AM INVITED PAPER
IMPROVING EFFICIENCY AND EMISSION REDUCTION - SOURCE PROCEDURES IN SECONDARY ALUMINUM INDUSTRY: *Ina Ollenschlaeger*[1]; [1]VAW Aluminium AG, Forschung und Entwicklung, Entwicklungszentrum Recycling, Georg-von-Boeselager-Str. 25, Bonn D-53117 Germany

Aluminum is used in many different products: from thin foil in packaging material over automotive components up to building applications. The various scraps of these products and their wide range of non-metallic components like paper, lacquers, and oils may cause a high level of emissions, especially dioxins. To avoid this output into the atmosphere, secondary processes like waste gas purification techniques are required which generate themselves contaminated materials and in any case cost. Another way to reduce emissions as dioxins and others and minimize energy input is by regulating source procedures like (in case of rotary drum furnace) variations in burner and charging technology, waste gas guidance, and process parameters. The experiments have been carried out in the recycling center, which is part of VAW Aluminium AG, department for research and development. As test equipment a rotary furnace in pilot scale (500 kg) was used and the emission, energy and mass balance were determined.

11:20 AM CLOSING REMARKS

HIGH TEMPERATURE COATINGS III: Ceramic and Intermetallic Coatings

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, Center for Laser Applications, Tullahoma, TN 37388 USA

Wednesday AM Room: 19
March 3, 1999 Location: Convention Center

Session Chairs: Michael J. McNallon, University of Illinois at Chicago, CME Dept., Chicago, IL 60607 USA; Robert J. Hanrahan, Los Alamos National Laboratory, Mats. Sci. and Tech. Div., Los Alamos, NM 87545 USA; Janet M. Hampikian, Georgia Institute of Technology, Dept. of Mats. Sci. and Eng., Atlanta, GA 30332-0245 USA

8:30 AM
THE EFFECT OF REACTIVE ELEMENT ADDITIONS ON THE OXIDATION OF BERYLLIUM MODIFIED NiAl: *R. J. Hanrahan*[1]; D. J. Thoma[1]; [1]Los Alamos National Laboratory, Mats. Sci. & Tech. Division, MST-6, TA 3 MS G770, Los Alamos, NM 87545 USA

Be may be solutionally substituted for Al in NiAl at up to 5-7 a/o. In our previous studies we have determined that at levels as low as 1 a/o Be that a protective oxide layer formed which consists of the spinel $BeAl_2O_4$. The usual transient alumina phases have never been observed to form under any exposure conditions tested. The oxide is extremely smooth and adherent however we still observe void formation at the metal-oxide interface and under cyclic oxidation or exposure above 1100°C the scale spalls in a manner similar to binary NiAl. We have therefore added several different reactive elements (Y, Zr, various lanthanides) to arc-cast ingots of Ni-48Al-2Be to determine whether these additions might further improve the oxide stability relative to the ternary alloy. Cyclic and static oxidation tests were conducted at 1000 and 1200°C in moist air.

8:50 AM
DEVELOPMENT OF HIGH TEMPERATURE SULFIDATION RESISTANT Fe-Al WELD OVERLAY COATINGS: *S. W. Banovic*[1]; J. N. DuPont[1]; P. F. Tortorelli[2]; A. R. Marder[1]; [1]Lehigh University, Whitaker Lab, 5 East Packer Ave., Bethlehem, PA 18015 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Fe-Al alloys, with low aluminum contents, are being investigated for use as corrosion protective weld overlay coatings. Previous research has demonstrated that compositions in the intermetallic regime have excellent high temperature sulfidation resistance. However, their low strength at high temperatures and lack of weldability limit their usage. Therefore, research has been initiated to study compositions in the disordered region which have lower amounts of aluminum. The high temperature sulfidation behavior of alloys with aluminum contents of 5-12.5 wt% Al was investigated using thermogravimetric techniques. These compositions are located near the crack/no crack boundary previously found during weldability studies. Samples were isothermally held in a reducing

gas (H_2-H_2S-Ar) at temperatures ranging from 500-700ϒC. Light optical and scanning electron microscopy were used to analyze the corrosion scales. Compositions of these reaction products were determined using electron probe microanalysis (EPMA). Depending upon the temperature, composition of the gas, and aluminum content of the alloy, various surface scales were observed to form. Those that developed an aluminum oxide scale were relatively protective and had very low weight gain. Samples that formed iron sulfide scales were generally less protective than those previously mentioned, but had lower weight gains than carbon steel and 309 stainless steel. These results indicate good promise for the practical application of Fe-Al overlay coatings in reducing environments.

9:10 AM

THE DEPOSITION OF CHROMIA, CHROMIA/YTTRIA, AND SILICA COATINGS VIA COMBUSTION CVD: Amanda E. Alexiou[1]; *Janet M. Hampikian*[1]; [1]Georgia Institute of Technology, School of Mats. Sci. and Eng., Bunger Henry Bldg., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

Combustion chemical vapor deposition was utilized to deposit chromia, 2 wt.percent yttria/chromia, and silica thin films onto Ni-20Cr. Amorphous phases of all three films were produced, as well as crystalline phases of both the chromia and chromia/yttria films. The phase generated depended on deposition temperature, which was varied between 220, for amorphous phase, and 500ϒC, for crystalline phase in the chromia films and from 700 to 800ϒC for the amorphous silica films. The films were characterized using SEM, TEM, EDS, and x-ray diffraction spectroscopy. The ability of the films to provide oxidation protection at 800ϒC was quantified using thermogravimetric analysis (TGA). All three types of coatings were deposited for 30 minutes and, in addition, the chromia films were also deposited for 15 and 7.5 minutes. The effects of film type and thickness were compared in the oxidation studies. All coatings reduced the oxidation kinetics of Ni-20Cr.

9:30 AM

NUMERICAL SIMULATIONS OF HVOF JET/SUBSTRATE INTERACTION: *S. Eidelman*[1]; X. Yang[1]; [1]Science Applications International Corporation, 1710 Goodridge Dr., McLean, VA 22102 USA

HVOF coatings of micro and nanoscale materials can present a problem when significant particle deceleration can occur in the stagnation region of jet/substrate interaction. Thus, very small particle will slow down and be diverted by the flow in the stagnation region. The material cannot be coated if it is composed of unagglomerated nanosized particles, because nanosize particles closely follow the streamlines of the carrying gas. This will result in powder loss and reduced coating efficiency and quality. We use comprehensive numerical simulation to examine the possible regimes for the coatings deposition considering the full range of system parameters for the HVOF jet/substrate interaction. We also explore the effect of nano-agglomerates density, size, and shape on coating efficiency, which can suggest the goals for materials preprocessing optimization. Rapid analysis of the parametric space using numerical simulations will shorten the time scale for new coating development and allow better understanding of the parameters that control coating efficiency and quality.

9:50 AM

HARDENING AND MICROSTRUCTURE OF ALUMINUM METAL SURFACE BY COMPLEX ALLOYING: *M. Okutomi*[1]; A. Obara[1]; K. Tsukamoto[1]; [1]Electrotechnical Laboratory, Opto-electronic Division, 1-1-4 Umeono, Tsukuba, Ibaraki 305 Japan

Hardening and microstructure of aluminum metal surface with CO_2 laser alloying assisted ultrasonic wave vibration technique have been investigated. A mixture of Al and Ti powder in an organic flux was painted onto the metal substrate followed by laser irradiation. To achieve uniform fine dendritic structure and to eliminate pores within the alloyed layer, laser irradiation was supplemented with ultrasonic vibration. Evaluation of the microstructure, hardness and tribological properties of the alloyed surface have been conducted. The alloyed surface consisted of dendrite structures of Ti_3Al and Ti-Al solid solution. The Ti_3Al dendrite appear to adhere effectively to other aluminum based phases thereby providing hardnesses for the alloyed surface in excess of 600 kg/mm^2. In addition, the wear resistance of the alloyed surface was about 1/7 time than that of the bare aluminum surface.

10:10 AM BREAK

10:25 AM

REACTION FORMED COATINGS FOR SiC FIBERS IN CERAMIC MATRIX COMPOSITES: *Michael J. McNallan*[1]; Daniel Ersoy[1]; Yury Gogotsi[2]; [1]University of Illinois at Chicago, CME Dept., M/C 246, Rm 3083 ERF, 842 W. Taylor St., Chicago, IL 60607 USA; [2]University of Illinois at Chicago, Mechanical Engineering, M/C 251, 842 W. Taylor St., Chicago, IL 60607 USA

A layer of compliant material is usually deposited on fibers for ceramic matrix composites to permit toughening by fiber pull out during failure. Chemical vapor deposition or sol gel techniques have usually been used to produce these coatings. These processes are expensive and sometimes fail to produce uniform, coherent coatings. Carbon films can be formed on SiC by selective chlorination of the silicon at high temperature and atmospheric pressure. The process is low cost and amenable to most SiC based fibers and whiskers. These films are generally of graphitic structure and can be further reacted to produce other coatings with better oxidation resistance.

10:45 AM

DEVELOPMENT OF A DIFFUSION BARRIER LAYER FOR HIGH-TEMPERATURE MOLYBDENUM ELECTRODES: *C. Suryanarayana*[1]; D. A. Buell[2]; D. Williamson[2]; J. J. Moore[1]; J. Disam[3]; [1]Colorado School of Mines, Advanced Coatings and Surface Engineering, Metall. and Mats. Eng., Golden, CO 80401-1887 USA; [2]Colorado School of Mines, Dept. of Physics, Golden, CO 80401 USA; [3]Schott Glaswerke, Mainz D-55014 Germany

Molybdenum electrodes are used in glass melting furnaces. To improve their oxidation resistance, especially at high temperatures, and also to match the coefficient of thermal expansion of molybdenum, application of a thin coating of a functionally graded $MoSi_2$+SiC layer is suggested. But, the problem with this coating is that silicon and carbon from the coating diffuse into the molybdenum substrate forming carbides and subsilicides resulting in the degradation and consequently losing the effectiveness of the coating. A diffusion barrier coating layer is being developed to overcome this problem. Reactive sputtering of an $MoSi_2$+1.96 mol SiC target in a nitrogen atmosphere produced an amorphous Mo-Si-C-N layer. This layer has been characterized for its structure and chemistry by a combination of techniques such as X-ray diffraction, differential thermal analysis, scanning electron microscopy, X-ray photon electron spectroscopy, and Auger electron spectroscopy. Annealing of this amorphous film at different temperatures was conducted to study the thermal stability of the amorphous layer and also to study its crystallization behavior. The results obtained on the microstructure, chemistry and the thermal stability of the diffusion barrier layer will be presented.

11:05 AM

PROTECTION OF HEATSTRESSED HYPERSONIC AIRCRAFT STRUCTURES MADE OF REFRACTORY MATERIALS BY NOVEL OXIDATION RESISTANT COATINGS: *V. Terentieva*[1]; [1]Moscow State Aviation Institute, Volokolamskoye Shosse 4, GSP, Moscow 125871 Russia

The problems connected with the theory and practice of creation reliable protective coatings for heatstressed elements of structures working under high speed enthalpy oxygen containing gas flows are considered. The main failure sources of coatings under these conditions are revealed and their evaluation criteria are determined. Hazard in commencing an oxidation reaction of the base material under coating is connected with density of open pores and cracks, and partial pressure of the oxidizer. The presence of technological and working defects makes worse the probability character of catastrophic destruction of coating and moves it to a region of much lower value of heat transfer. For the conditions under discussion a physico-chemical model of coating realized in practice for silicide type coating is offered. It provides much faster self-healing of micro-and macrodefects in the coating, blocking of oxidizer supply to pores and cracks by suppression on diffusion in gaseous phase and instantaneous formation of protective oxide film on the working

surface and continuous restoration of scale in erosion entrainment process.

11:25 AM
PREVENTION OF HYDROGEN PERMEATION ON TiAl BY ION IMPLANTATION: Y. Matsumoto[1]; Yao-Can Zhu[1]; Yasuo Suzuki[1]; *Nobuya Iwamoto*[1]; [1]Ion Engineering Research Institute Corporation, 2-8-1,Tsuda-yamate, Hirakata, Osaka 573-0128 Japan

Lightweight intermetallic TiAl is an important alloy for aerospace materials to raise the thrust-to-weight ratio. It has a problem with respect to its resistance to oxidation and hydrogen permeation, especially in the case of hydrogen or methane-burning environment. We have studied the effects of adding several elements, such as Cr, Nb, V and Mn by ion implantation, to prevent hydrogen embrittlement of TiAl at high temperature.

11:45 AM
SILICON CARBIDE COATING ON UO_2 PELLET BY A COMBUSTION REACTION: *Bong Goo Kim*[1]; Y. Choi[2]; Y. W. Lee[1]; D. S. Sohn[1]; [1]Korea Atomic Energy Research Institute, Capsule Development and Utilization, P.O. Box 105, Yusung, Taejeon 305-600 Korea; [2]Sunmoon University, Asan Korea

Multi-layer deposition of silicon carbide and pyrolytic carbon on UO_2 pellet was prepared by using a combustion reaction between carbon and silicon layers. The pyrolytic carbon and silicon layers were deposited by thermal decomposition of propane at 1250ϒC and plasma-enhanced chemical vapor deposition (PE-CVD) of silane at 500ϒC, respectively, followed by a combustion reaction between the two layers. Microstructural observation of the layer with SEM showed that an inner layer existed following the surface contour of the pellet and an outer layer had small amount of fine pores inside. Chemical analysis with EDX and AES revealed that the inner and outer layers were pyrolytic carbon and silicon carbide, respectively. From the TEM observation, the silicon carbide formed during the combustion reaction was fine crystalline beta-silicon carbide. Temperature distribution of the specimen during the combustion reaction was estimated by empirical and numerical methods.

12:05 PM
SUPERHARDNESS EFFECTS IN THE Si_3N_4/TiN CERAMIC NANO-MULTILAYER FILMS: *J. Xu*[1]; Mingyuan Gu[1]; Geyang Li[1]; [1]Shanghai Jiao-Tong University, State Key Lab of MMCs, Shanghai 200030 China

The polycrystalline Si_3N_4/TiN ceramic nano-multilayer films have been synthesized on Si substrates by a reactive magnetron sputtering technique. The primary objective of this research is to investigate the effects of modulation ratio and modulation period on the microhardness and to elucidate the hardening mechanisms of the synthesized nano-multilayer films. The results showed that the hardness of Si_3N_4/TiN nano-multilayers is affected not only by modulation period, but also by modulation ratio. The hardness reaches its maximum value when modulation period equals a critical value L0, which is about 12 nm with a modulation ratio of 3/1. The maximum hardness value is about 40% higher than the value calculated from the rule of mixtures. The hardness of nano-multilayer thin films was found to decrease rapidly with increasing or decreasing modulation period from the point of L0. The microstructure and the internal stress state of the nano-multilayer films have been investigated using XRD and TEM. Based on experimental results, the mechanism of the superhardness in this system was proposed.

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Structure-Property Relationship and Reliability II

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 Corp, San Jose, CA 95134 USA; Ephraim Suhir, Bell Labs., Murray Hill, NJ 07974 USA

Wednesday AM Room: 17A
March 3, 1999 Location: Convention Center

Session Chairs: M. Weiser, Johnson Matthey Electronics; S. Jin, Lucent Technologies

8:30 AM INVITED PAPER
RELIABILITY OF SOLDER JOINTS ON SEVERAL ALTERNATIVE PCB SURFACE FINISHES: *Z. Mei*[1]; F. Hua[1]; J. Glazer[1]; [1]Hewlett Packard Company, Electronic Assembly Development Center, 1501 Page Mill Rd., MS 5L-C, Palo Alto, CA 94304 USA

Electroless Ni coated with immersion Au is being used as an alternative metal finish to Sn-Pb on printed circuit board (PCB). Electroless Ni/electroless Pd/immersion Au is a new metal finish that is being introduced in PCB industry. Mechanical reliability of PBGA (plastic ball grid array) attached on the two metal finishes was studied in both as-received and aged conditions. Weak solder joints on Ni/Au were observed, resulted from a brittle fracture at the interface between Ni-Sn intermetallic compound and electroless Ni. After aging, the solder joint becomes even weaker and more brittle, resulted from the deposition of Au-Sn intermetallic compound at the interface between solder and Ni-Sn compound. The Ni/Pd/Au finish provided an improved solder joint reliability in some conditions, which is probably related to the Sn-Pd intermetallic compound formation. Some results on immersion Ag, immersion Sn, and electroless Pd on Cu or Ni may be presented at the conference, if data are available by then.

9:00 AM
MANUFACTURING FEASIBILITY AND RELIABILITY OF LOW MELTING SOLDERS: *F. Hua*[1]; Z. Mei[1]; J. Glazer[1]; [1]Hewlett-Packard Company, Electronic Assembly Development Center, 1501 Page Mill Rd., Palo Alto, CA 94304 USA

The applications of low melting, Sn-Bi and Sn-Bi-Pb based solders in electronic products were studied with the emphasis on manufacturing related issues and solder joint reliability. The melting temperatures and basic mechanical properties were determined as an initial screening assessment of viable candidate for further prototype development. Prototype products are printed circuit boards (PCBs) with organic coated Cu surface finishes, soldered with components with different lead frame metals, Sn-Pb on Alloy 42, Sn-Pb on Cu, Pd/Ni on Cu, Au on Ni-Cu. Visual inspection of solder joint, push and pull of component leads, shock, vibration or prototype production boards were conducted. An environmental thermal cycling chamber was used to test the thermal mechanical reliability of the solder joints. The fracture surface and cross section of solder joints were analyzed for failure mode and bonding intermetallic compounds. It is concluded that low temperature solders, along with correct selections of soldering flux and component lead surface finishes, could be viable solutions for electronic assembly.

9:25 AM INVITED PAPER

HIGH MILEAGE SOLDER JOINT RELIABILITY OF ALTERNATOR POWER DIODE ASSEMBLY: S. C. White[1]; E. L. Lutz[1]; *T. Y. Pan*[2]; H. D. Blair[2]; J. M. Nicholson[2]; [1]Visteon Powertrain Control Systems, Textile and McKean Rds., Ypsilanti, MI 48197 USA; [2]Ford Motor Company, Ford Research Laboratory, 20,000 Rotunda Dr., P.O. Box 2053, Bldg. R, M/D 3135, Dearborn, MI 48121-2053 USA

The reliability of the solder joints at the power diode leads to the rectifier in automotive alternators are required to last at least 10 years / 150,000 miles. The service environment of the alternator and the heat generation of the diodes themselves provide a great challenge to the solder joint reliability. The ambient service temperature of the alternator can get to 175°C and the internal diode can run as hot as 205°C. The solder joints have been and are assembled by a wave soldering process using a Pb-free Sn-Ag eutectic solder since the late 80's, and constitutes one of the very first Pb-free solder assemblies in mass production (5.5 million parts a year) in the industry. A recent introduction of a new diode, from a different supplier, was required to avoid a projected supply interruption and a significant cost increase. However, the new diode rectifier assemblies were shown to have poor lead wire solder joint thermal cycle reliability. It was discovered that the high thermal dissipation of the new diodes, normally a desirable feature, resulted in poor solder joint reliability. A cross-organizational team, utilizing advanced analytical tools, made a patent-pending rectifier leadframe design change which resulted in a dramatic increase in solder joint thermal cycle fatigue life while maintaining best-in-class rectifier package dimensions. The team achieved first-pass prototype success while using existing production facilities and still met aggressive launch timing.

9:55 AM

EFFECTIVE MICROALLOYING FOR THE IMPROVEMENT OF THERMAL FATIGUE AND CREEP RESISTANCE IN EUTECTIC Sn-Pb SOLDERS: N. Wade[2]; T. Akuzawa[3]; S. Yamada[2]; D. Sugiyama[2]; I-S. Kim[1]; *K. Miyahara*[1]; [1]Nagoya University, Dept. of Molecular Design and Eng., Nagoya 464-01 Japan; [2]TOPY Industrial, Ltd., Technical R&D Lab., Toyohashi Japan; [3]Nippon Filler Metals, Chiba Japan

This study aims to improve the thermal fatigue and creep resistance of Sn-Pb eutectic solder by the addition of small amount of effective elements, such as Sb, Ag, Cu and In, which are selected by preliminary experiments and statistical analysis. Thermal fatigue test was performed under the heat cycling of 233 to 393K. Creep tests were conducted at the stress and temperature range of 5 to 15 MPa and 313 to 378K. The above microalloying improved significantly the thermal fatigue life and creep life. For instance, the fatigue life for 50% crack generation was improved by twice and the creep life at 10 MPa and 353K by ten times. Transmission electron microscope observation indicated that the excellent properties came from the precipitation hardening by an appropriate combined addition of the microalloying elements.

10:20 AM BREAK

10:30 AM INVITED PAPER

ROLE AND RESPONSE OF Sn-Pb MICROSTRUCTURE IN CONSTITUTIVE MODEL-BASED PREDICTIONS OF SOLDER JOINT THERMAL MECHANICAL FATIGUE: *P. T. Vianco*[1]; S. N. Burchett[1]; M. K. Nielsen[1]; [1]Sandia National Laboratory, P.O. Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA

The capability to measure the extent of thermal mechanical fatigue in solder is an important milestone towards predicting the reliability of solder interconnects in electronics. Theoretical and experimental research has resulted in the development of a constitutive model for Sn-Pb solder, which when coupled with a finite element code, provides such an avenue for quantifying microstructural damage to Sn-Pb solder during thermal mechanical fatigue. The constitutive model based on two-term, power-series expression, each term explicitly representing the contributions of time-dependent and time-independent plastic deformation on the performance of the solder under thermal mechanical loading. An important response by the model is its description of the evolution of the solder microstructure during the course of thermal mechanical fatigue processes. Specifically, the model/code describes both spatial as well as temporal changes to the Pb-rich phase particle distribution in the Sn-Pb alloy that comprises the joint; the extent of such changes are believed to be the precursor to crack initiation and eventually, crack propagation and failure of the solder joint. The role of the Pb-rich phase particle size characterizing the starting microstructure of through-hole solder joints has been investigated. The spatial distribution of particle sizes in the range of $3x10^{-6}$ mm^2 to $20x10^{-6}$ mm^2, as determined from experimental studies, represented the starting microstructure input for the model. The Pb-rich phase size was distributed non-homogeneously in the solder joint; the effects of these distributions on fatigue damage and likely initiation sites of cracks will be described. The resulting microstructural development, as predicted by the model, will be evaluated and compared with available experimental observations. The sensitivity of thermal mechanical fatigue damage to the materials and joint geometries, as ameliorated or aggravated through the solder microstructure, will also be documented. [1]Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Dept. of Energy under contract DE-AC04-94AL85000.

11:00 AM

THE INFLUENCE OF TEMPERATURE ON FATIGUE-CREEP INTERACTIONS IN A LEAD-TIN SOLDER ALLOY: *W. J. Plumbridge*[1]; J. E. Moffatt[1]; [1]The Open University, Dept. of Mats. Eng., Milton Keynes MK7 6AA UK

Strain-controlled fatigue test, involving dwells at maximum strain limits, have been performed on bulk specimens of a eutectic lead-tin solder alloy at room temperature at 75°C. During continuous cycling, softening occurs at each temperature to around 10 and 15 percent respectively. The fatigue endurance is reduced by typically one third at the higher temperature. The incorporation of a dwell of 10 or 100s into the cycle causes a reduction in fatigue life in comparison to that observed during continuous cycling. Cycles containing balanced dwells at maximum and minimum strain limits are the most deleterious when lifetime debits of up to fivefold are observed at both temperatures. Unbalanced compression-only profiles tend to be the least damaging. The stress relaxation characteristics are similar in all dwells. Metallographic observations of sectioned specimens reveals surface and intergranular cracking which is accentuated at higher-strain ranges and temperature but not dramatically affected by the cycle shape. These findings are considered in terms of the behavior of other engineering alloys and potential lead-free replacement solders.

11:25 AM

EFFECT OF STRAIN RATE AND HOLD TIME ON THE ISOTHERMAL FATIGUE LIFE OF Sn-3.5Ag-X(X=Bi, Cu, In) SOLDER ALLOYS: *Yoshiharu Kariya*[1]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Mats Engineering, Shibaura 3-9, Minato-ku, Tokyo 108-8548 Japan

Strain rate and hold time are most important factors affecting fatigue life of solder alloys. However, no systematic studies have been carried out with respect to the influence of these parameters on fatigue properties of lead-free solders, while sufficient data are available for eutectic Sn-Pb solder. In our previous study, we clarified the effect of third element (Bi, Cu, and In) on the fatigue life of Sn-3.5%Ag binary alloy at moderately high strain rate and found that the fatigue life of Sn-3.5Ag binary alloy remarkably decreased with increasing content of bismuth though the addition of copper and indium slightly decreased the fatigue life of Sn-3.5% Ag alloy. In this paper, the effect of strain rate and tensile hold time on the isothermal fatigue life of bulk Sn-3.5%Ag-X(X=Bi, Cu and In) solder alloys have been studied with loading mode of sawtooth and trapezoid wave. Low strain rate is found to reduce the fatigue life of Sn-3.5%Ag-X. Increase in hold time at maximum strain also decreases the fatigue life of these ternary alloys, though holding for more than 2 minutes results in almost constant fatigue life. This should be ascribed to creep which occur during holding and low strain rate deformation. The result coincides well with the fact that applied stress has fully relaxed with 2 minutes even at room temperature. However, the fatigue life itself is found to be insensitive to hold time.

11:50 AM INVITED PAPER

CHARACTERIZING THE WEAK ORGANIC ACIDS USED IN LOW SOLIDS FLUXES: B. A. Smith[1]; *L. J. Turbini*[1]; [1]Georgia Insti-

tute of Technology, Dept. of Mats. Sci. Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

The elimination of chloroflurocarbons (CFC's) and other chlorinated cleaning solvents due to their long-term environmental impact has lead electronic assemblers to examine soldering fluxes that reduce or eliminate the need for post-solder cleaning. Today, low solids fluxes are replacing more traditional rosin-based and water-soluble fluxes because many of them can be used in a non-clean process. Most low solids fluxes use weak organic acids as active ingredient. It has been reported that some of these weak organic acids leave behind residues that are corrosive to copper. Surface Insulation Resistance (SIR) measurements of flux-processed comb patterns have been the main test method used to determine the corrosivity of flux residue. This test has been performed with test samples exposed to accelarated temperature and humidity conditions of 85°C and 85%RH and a 50V bias. Recent data on some weak organic acids suggests that they slowly disappear at this temperature and a lower test temperature of 65°C has been introduced into the new Bellco Standard. In Europe, this test is normally performed at 40°C and 93%RH. This paper will report on the application of SIR test to study the corrosive behavior of six carboxylic acids that are commonly used as the active ingredients in soldering fluxes. Coupons treated with equimolar solutions of the acids were either exposed to reflow-soldering conditions or wave soldered face-up to create partially heated residues. Both tests were run under two different accelerating conditions, the first being 85°C/85%RH for 7 days and the second being 40°C/93%RH for 20 days. This latter condition is being considered for inclusion in an ISO standard. At the end of the test period, both corrosion and SIR test samples were examined under a microscope and any residues or dendritic growth were documented. SEM and EDX characterization was also performed to determine the residue and dendrite composition.

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Twinning in Intermetallics

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

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

Wednesday AM Room: 17B
March 3, 1999 Location: Convention Center

Session Chairs: A. K. Vasudevan, Office of Naval Research, 800 N. Quincy St., Arlington, VA 22217-5660 USA; Terence E. Mitchell, Los Alamos National Laboratory, Center for Mats. Sci., Los Alamos, NM 87545 USA

8:30 AM INVITED PAPER

MICROMECHANISMS OF TWIN NUCLEATION IN TiAl: EFFECTS OF NEUTRON IRRADIATION: *Man H. Yoo*[1]; A. Hishinuma[2]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Japan Atomic Energy Research Institute, Dept. of Materials Science and Engineering, Tokai Research Establishment, Tokai, Ibaraki-ken 319-11 Japan

In Ti-47at%Al alloys of the duplex microstructure, consisting of equiaxial grains of the Ti_3Al and TiAl phases, an increase in tensile elongation at 873 K from 6% to 10% was reported after neutron irradiation to a dose of 1 x 10^{20} n-cm^{-2} (E > 1 MeV). This so-called radiation-induced ductility (RID) is attributed to the formation of effective twin embryos in the presence of interstitial-type Frank loops in γ-TiAl phase and the subsequent nucleation and growth of microtwins during post-irradiation plastic deformation. In this paper, micromechanics of defect clustering under irradiation and twin formation during deformation are analyzed. The results are given on the stability of Frank loops against unfaulting, the source operation of twinning partial dislocations, and the critical conditions for twin formation. The RID phenomenon will be elucidated from a viewpoint of the proposed micromechanisms and the TEM analyses of the irradiation-induced microstructure. [Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.]

9:05 AM

INTERFACE CONTROLLED DEFORMATION TWINNING IN TWO-PHASE TiAl WITH A LAMELLAR MICROSTRUCTURE: *Luke M. Hsiung*[1]; T. G. Nieh[1]; [1]Lawrence Livermore National Laboratory, Mats. Sci. and Tech., L-369, P.O. Box 808, Livermore, CA 94551-9900 USA

Mechanisms of deformation twinning in creep-deformed lamellar TiAl (γ) - Ti3Al (α2) have been investigated. Since the operation and multiplication of lattice dislocations within both //0032/ and α2 lamellae are very limited due to a refined lamellar microstructure (γ lamelae: 100 - 300 nm thick, α2 lamellae: 10 - 50 nm thick), the glide of preexisting interfacial dislocations (i.e. interfacial sliding) becomes a dominant deformation mode. During the gliding of interfacial dislocations along the lamellar interfaces, their mobility can be impeded by obstacles such as impinged lattice dislocations. This event increases as applied stress or strain increases and subsequently resulting in the pile-up of interfacial dislocations. When the alloys are crept at a high stress level, deformation twinning is found to become a predominant deformation mode with the deformation twins preferentially nucleating from lamellar interfaces. It is suggested that deformation twinning is a stress relaxation process for dissipating stress concentration at the head of each dislocation pile-up. A twinning formation mechanism driven by the pile-up of interfacial dislocations is accordingly proposed and verified. This work was performed under the auspices of the U.S. Department of Energy by LLNL under contract No. W-7405-Eng-48.

9:30 AM INVITED PAPER

DEFORMATION TWINNING IN A HfV2 + Nb - BASED LAVES PHASE ALLOY: Yoshisato Kimura[1]; *David E. Luzzi*[1]; David P. Pope[1]; [1]University of Pennsylvania, Dept. of Mats. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Single phase HfV_2, like other cubic C15 Laves phases, shows almost no plasticity at low temperatures. Nb additions are known to improve the ability of this material to deform by mechanical twinning, however single phase material remains extremely brittle. Two-phase alloys consisting of the C15 HfV_2 + Nb phase and a bcc (V, Nb) solid solution exhibit considerable ductility (3% plastic strain at ambient temperature), even though the alloy has the brittle C15 matrix. To investigate the deformation behavior of this two phase alloy, compression tests were conducted at low temperatures, down to 4.2K. The 0.2% flow stress drops dramatically at around 77K - it is nearly 500 MPa lower at 77K than it is at 300K - then rises rapidly at temperatures below 77K. No such anomaly is seen in samples with less than 20% volume fraction of C15. Transmission electron microscopy revealed that substantial mechanical twinning takes place in the C15 phase at room temperature and at 77K, but not at 4.2K. This lack of twinning at 4.2K suggests that mechanical twinning in the C15 is both a stress- and temperature-driven phenomenon. We believe that the cause of the drop in flow stress at 77K is an increased ease of mechanical twinning in the C15 phase of the two phase alloy.

10:05 AM BREAK

10:15 AM INVITED PAPER

TRANSMISSION OF TWINNING AND SLIP AT LAMELLAR INTERFACES IN Ti-Al: *Peter M. Hazzledine*[1]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45387 USA

Twinning is an important plastic mechanism in the gamma phase of lamellar Ti-Al at all temperatures. It may contribute both to soft mode and to hard mode deformation. Lamellar Ti-Al is an interface strengthened material in which deformation must be propagated across domain boundaries in the gamma phase during soft mode deformation and across

both gamma-gamma and alpha-gamma lamellar interfaces during hard mode deformation. The domain boundaries and the gamma-gamma lamellar boundaries may or may not themselves be twin interfaces. This paper examines the mechanisms of deformation transmission e.g. a hard mode twin in one lamella triggering slip in the neighboring lamella and attempts to calculate the contribution made to the Hall-Petch strengthening by such mechanisms.

10:50 AM INVITED PAPER

TWINNING IN $ReSi_{2-x}$: *Terence E. Mitchell*[1]; Amit Misra[1]; [1]Los Alamos National Laboratory, Center for Materials Science, MS-K765, P.O. Box 1663, Los Alamos, NM 87545 USA

$ReSi_{2-x}$ is usually referred to as rhenium disilicide but it actually has a stoichiometry of $ReSi_{1.75}$. Also it is usually described as having the tetragonal $MoSi_2$ C11b structure but in fact it exhibits a number of incommensurate and commensurate structures which are based on the C11b structure. Structure A has an incommensurate periodicity of 4.14a along the a axis. It has a small orthorhombic distortion (b/a~1.005) accompanied by twinning on the (110) plane and a small monoclinic distortion ($b\sim90.2_o$ accompanied by twinning on the (001) plane. Structure B is also incommensurate but has a larger monoclinic angle of 107_o; B appears to result from a shear transformation of A, giving rise to (001) twins. Prolonged annealing at high temperatures results in structure C which is commensurate and has planes parallel to, and exactly 4 times the spacing of, the(101) planes of the underlying C11b structure. Structure C is also twinned. The various structures are apparently caused by attempts to accommodate the Si structural vacancies. The relationship between these structures and the Nowotny "chimney-ladder" structures which have the general formula M_nSi_{2n-m} and are based on the $TiSi_2$ structure will be discussed.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Flow and Fracture

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. 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

Wednesday AM Room: 8
March 3, 1999 Location: Convention Center

Session Chairs: Michael V. Nathal, Nasa Lewis Research Center, Cleveland, OH MS 49-3 USA; Gilbert Henaff, LMPM-ENSMA, Teleport 2, Futuroscope Cedex F-86960 France

8:30 AM INVITED PAPER

ASSURING RELIABILITY OF GAMMA TITANIUM ALUMINIDES IN LONG-TERM SERVICE: *James M. Larsen*[1]; Andrew H. Rosenberger[1]; Kezhong Li[2]; *Reji John*[2]; David C. Maxwell[2]; *W. J. Porter*[2]; [1]Air Force Research Laboratory, Materials & Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA; [2]University of Dayton Research Institute, 1031 Irving Ave., Dayton, OH 45419-0128 USA

Gamma TiAl alloys offer the potential for major reductions in weight of turbine engine components, if higher-density materials can be replaced without sacrificing long-term reliability in service. To assess this potential, an overview is presented of the structural capabilities available from gamma titanium aluminide alloys. Emphasis is given to effects of material defects, mechanistic aspects of fatigue damage evolution, and the roles of high-temperature, time-dependent deformation and environmental effects. Although gamma alloys exhibit excellent fatigue strengths, in many applications the material's resistance to service-induced damage is a critical concern. In turbine engines, such damage may result from impacts by foreign objects in the flow path. In addition, numerous components in turbine engines are subject to both low- and high-cycle fatigue. To predict the range of behavior that may be expected for gamma titanium aluminides requires consideration of damage tolerance, specifically addressing the potential for growth of small fatigue cracks that may form under a variety of circumstances. Approaches for life prediction in these materials are discussed, emphasizing the roles of fatigue crack initiation and growth. In addition, an attempt is made to assess the strengths and limitations of these materials with respect to application requirements and to suggest avenues for improvements in the balance of mechanical properties.

9:00 AM

THREE CHARACTERISTIC PHASES OF FATIGUE CRACK GROWTH IN A TiAl BASED ALLOY: *Ze-Wen Huang*[1]; Paul Bowen[2]; [1]Birmingham University, IRC Centre in Materials, Edgbaston, Birmingham, West Midlands B15 2TT UK; [2]Birmingham University, School of Metallurgy and Materials, Edgbaston, Birmingham, West Midlands B15 2TT UK

The fatigue crack growth behaviour of a g-TiAl based Ti-48Al-2Mn-2Nb alloy has been studied at room temperature in air. Three distinct phases of fatigue crack growth were found commonly for the fully lamellar microstructures of 550 micrometer colony size. Fatigue crack initiation and early growth near threshold, as the first phase, proceeded at a very slow growth rate along a mode I crack direction. There is no crack deflection, and no intact ligaments are left in the crack wake during this growth. The second phase is characterised by intermediate growth rates and a non-linear variation of log da/dN with log DK, and this gives a wide range of crack growth rates and behaviour quite different from that observed in conventional alloys. Now there may be some crack deflection and small ligaments can be found in the crack wake. These two regimes account for ~ 95% of the total number of cycles to failure while the crack propagates a relatively short distance. The third phase is typically demonstrated to be a regime of catastrophic propagation. Most of the fatigue crack area observed derives from this very fast crack growth process, where a higher degree of crack deflection, branching and unbroken ligaments can be found. Extensive SEM examination has been carried out to correlate this mechanical behaviour with fractographic features. The mechanisms governing these three distinct crack growth phases will be discussed in terms of the underlying microstructures.

9:20 AM INVITED PAPER

THE VARIABILITY OF TENSILE PROPERTIES IN CAST GAMMA TITANIUM ALUMINIDES: Rafael Raban[1]; *Tresa M. Pollock*[1]; [1]Carnegie Mellon University, MSE Dept., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The use of investment cast gamma titanium aluminides in aircraft engine applications requires that some degree of tensile ductility be achieved reproducibly. Factors influencing the variability of tensile properties in several cast TiAl alloys have been investigated. Alloys include Ti-48Al-2Cr-2Nb, Ti-46.5Al-2Cr-2Nb, Ti-47Al-2Cr-2Nb+0.5at%B and Ti-45Al-2Cr-2Nb+0.9at%B. The variability of room temperature yield strength and tensile ductility within individual cast plates will first be discussed. Additionally, variations in properties across a range of casting conditions will be presented along with associated changes in microstructure for each alloy. Fractographic observations, common features of the failure process and factors which may reduce variability will be discussed.

9:50 AM

MICROSTRUCTURAL STRENGTHENING OF FULLY LAMELLAR Ti-Al ALLOYS: Peter M. Hazzledine[1]; *Dennis M. Dimiduk*[1]; [1]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433 USA

Fully lamellar Ti-Al alloys with fine lamellae have exceptional yield strengths at low temperatures while retaining attractive high temperature properties. The four most significant microstructural parameters

governing these mechanical properties are the average lamellar thickness, the distribution of lamellar thicknesses, the volume fraction of alpha 2 (or the number fraction of gamma/alpha 2 interfaces) and the grain size. A predictive theory of the Hall-Petch strengthening would include these parameters and take account of the extreme plastic anisotropy of the constituent lamellar grains. In addition, to describe recent very high strength alloys, the theory must include the effects of strength saturation and which become important in lamellae < 20 nm thick. This paper reports progress towards such a theory and compares it with experimental measurements.

10:10 AM

DYNAMIC TENSILE PROPERTIES OF Ti-47Al-2Mn-2Nb ALLOY: *Dongliang Lin*[1]; Wang Yu[1]; [1]Shanghai Jiao Tong University, Institute of Mats. Sci. & Eng., 1954 Haushan Rd., Shanghai 200030 PR China

Room-temperature tensile properties of polycrystal Ti-47Al-2Mn-2Nb alloy with near lamellar (NL) microstructure were investigated at the strain rates between 10-5 and 1000s-1 using conventional testing machine and a self-designed Split-Hopkinson tensile bar setup with a rotating disk. It is found that tensile ductility varies within a narrow range with the strain rate while dynamic strengths (sd) of the alloy are obviously higher than static strengths (ss). There exists linear relationship between ss and the logarithm of the strain rate (ln) while linear relationship exists between sd and the strain rate itself ( ). Fractography analysis indicates that the alloy fractures in a mixed mode of predominant transgranular cleavage and minor intergranular failure under static and dynamic strain rates. Environmental effect is excluded from the main cause for the room-temperature brittleness of the investigated alloy.

10:30 AM INVITED PAPER

THE CONTRASTING ROLE OF MICROSTRUCTURE IN INFLUENCING THE GROWTH OF LARGE AND SMALL FATIGUE CRACK IN TiAl ALLOYS: *Robert O. Ritchie*[1]; J. J. Kruzic[1]; J. P. Campbell[1]; [1]University of California, Berkeley, Dept. of Mats Sci and Mineral Eng, 463 Evans Hall #1760, Berkeley, CA 94720-1760 USA

The initiation and growth behavior of small (<500 μm) surface cracks in a γ-based TiAl alloy, of composition Ti-47Al-2Nb-2Cr-0.2B (at.%), has been studied in both duplex (average grain size ~150 μm) microstructures, with results compared to the corresponding behavior of long (>3 mm) though-thickness cracks. Small crack experiments were carried out in four-point bending with cracks both initiated naturally and from created defects; large-crack tests were performed with compact-tension samples. Results indicated that whereas the lamellar microstructures displayed far superior "large crack" fatigue-crack growth properties, growth-rate data from the small-crack experiments were similar for the two microstructures. Such results are interpreted in terms of the suppression of extrinsic toughening, in this alloy from crack closure and uncracked (shear) ligament bridging, with cracks of limited size. In light of the similarity in intrinsic fatigue-crack growth resistance, the higher crack-initiation res istance and the reduced extent of scatter for the finer-scale duplex structure, this microstructure is preferred to the tougher lamellar structure for most fatigue-crack applications.

11:00 AM

HIGH TEMPERATURE CRACK GROWTH IN A GAMMA TiAl ALLOY UNDER CONSTANT-CREEP-LOADING CONDITIONS: *Kumar Jata*[1]; *Young-Won (Y-W.) Kim*[2]; [1]Air Force Research Laboratory, AFRL/MLLM, Bldg. 655, Wright-Patterson AFB, OH 45433 USA; [2]UES, Mats. Development & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Crack growth behavior of a gamma titanium aluminide alloy, K5 (Ti-46Al-2Cr-3Nb-0.2W-0.2Si), was investigated under constant-loading conditions in a nearly fully-lamellar (NFL) microstructural form at 650 and 800ϒC. The NFL microstructure consisted of 400 μm size lamellar grains containing a small volume fraction of finely (5-20 μm) dispersed gamma grains. Both crack opening displacement (COD) and crack extension were measured as functions of holding time and apparent stress intensity factor. Results show that the COD changes with time are closely related to the creep deformation previously measured on the same material. Cracking occurred following the creep deformation/damage in the plastic zone and ensued at stress intensities much higher than that for fracture initiation toughness. At 650ϒC, crack extension occurred through microcracking (lamellar separation) and then fracture of the translamellar ligaments. At 800ϒC, the major crack propagates primarily along the boundary (lamellar and grain) areas, frequently resulting in dimpled fracture surfaces which may be caused by void formation. Analysis will be made on the complex relationships between the crack-tip area deformation (both instantaneous and creep), crack propagation, microstructure, and boundary weakening.

11:20 AM INVITED PAPER

FATIGUE CRACK GROWTH THRESHOLDS OF TiAl ALLOYS: *Kwai S. Chan*[1]; [1]Southwest Research Institute, Mats. and Structures, 6220 Culebra Rd., San Antonio, TX 78238 USA

The growth behavior of fatigue cracks in TiAl alloys have been shown to depend on the crack size. Most of the naturally nucleated small fatigue cracks have been found to arrest after nucleation. Only a few do propagate at stress intensity ranges below the large crack threshold. The objective of this paper is to review the current understanding of this small crack behavior and to explore the validity of the concept of a growth threshold for small cracks, defined in terms of the applied stress range and the crack length at which crack arrest occurs, for treating the growth/no growth of small cracks. Crack growth threshold values for small cracks in TiAl alloys are presented and compared against the large crack value. The possible application of a size-dependent threshold for predicting of fatigue crack growth in TiAl alloys is discussed and evaluated against laboratory data.

11:50 AM

INFLUENCE OF FOREIGN OBJECT DAMAGE ON THE ELEVATED TEMPERATURE FATIGUE RESPONSE OF GAMMA TITANIUM ALUMINIDE ALLOYS: *Trevor S. Harding*[1]; J. Wayne Jones[1]; Paul S. Steif[2]; Volus McKenna[2]; James M. Larsen[3]; Andrew H. Rosenberger[3]; [1]University of Michigan, Dept. of Mats. Sci. and Eng., 2105 H.H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA; [2]Carnegie Mellon University, Mechanical Engineering, Scaife Hall, Pittsburgh, PA 15213 USA; [3]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH 45433 USA

The high specific stiffness and strength of gamma TiAl alloys has made them leading candidates for use in weight-critical components including turbine blades. The effects of service induced damage events, such as foreign object damage (FOD), on the fatigue behavior of gamma TiAl is of great importance for transition of this class of materials into gas turbine engines. In the present study simulated FOD impact tests were conducted on three cast gamma TiAl alloys, Ti-47.9Al-2.0Cr-1.9Nb, Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si and Ti-47Al-2Nb-0.8vol.%TiB2. Impact sites were subsequently characterized in terms of types and severity of damage present on the specimen surface. Fatigue failure stress was determined using an incremental step loading fatigue test conducted in air at 600°C. Correlations between severity of impact damage and fatigue failure stress will be discussed in terms of a threshold-based model. Fractographic analysis was used to reveal the mechanisms of initial damage and crack propagation, as well as, the shape and size of internal damage. These results are then correlated with the threshold-based approximation of fatigue failure stress. Further modeling and analysis of the role of near-threshold and small crack growth from impact damage sites in terms of estimated lifetime will be discussed.

12:10 PM

MIXED MODE CRACK GROWTH IN GAMMA TiAl: *Reji John*[1]; W. John Porter[1]; Andy Rosenberger[2]; [1]University of Dayton Research Institute, 300 College Park, Dayton, OH 45322 USA; [2]AFRL/MLLN, Wright-Patterson AFB, Dayton, OH 45433 USA

Gamma TiAl alloys are under consideration for use as engine blade materials in advanced aerospace engines. In these components, stage I crack growth can be expected to occur under mixed mode (mode I + mode II) loading at locations such as the root of the blade. The "brittle" behavior of Gamma TiAl at low temperatures (steep da/dN versus ΔK) necessitates characterization of the near-threshold behavior under service loading conditions. Hence, a program was initiated to investigate the effect of mixed mode loading on the near-threshold behavior of a Gamma TiAl alloy (identified as 395). A centrally notched disk specimen was used to conduct the tests at room and elevated temperatures.

Unique tests were also conducted using specimens with large (approx. 5mm), centrally located grains to characterize the interlamellar growth. This presentation will discuss the mode mixity effects on the interlamellar and intralamellar crack growth behavior of Gamma TiAl.

LONG TERM STABILITY OF HIGH TEMPERATURE MATERIALS: Stability of Ni-base Alloys - I

Sponsored by: Structural Materials Division, High Temperature Alloys Committee, Physical Metallurgy Committee
Program Organizers: Gerhard E. Fuchs, Lockheed Martin Corporation, Schenectady, NY 12301-1072 USA; Kathryn A. Dannemann, Southwest Research Institute, San Antonio, TX 7828-0510 USA; Todd C. Deragon, Special Metals Corporation, New Hartford, NY 13413-5392 USA

Wednesday AM Room: 9
March 3, 1999 Location: Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Mats. Sci. and Eng., Gainesville, FL 32611-6400 USA

8:30 AM KEYNOTE
THE STABILITY OF SUPERALLOYS: *S. T. Wlodek*[1]; [1]Gamma Prime Consultants, 17810 Pueblo Vista Lane, San Diego, CA 92127-1272 USA

Any structure that is exposed to temperatures, different from those at which that structure was previous equilibrated, will change so as to achieve the form that is stable at that temperature. A change in the structure sensitive properties of that material will then result. This review summarizes such reactions for the superalloy family of alloys, used in gas turbines. The superalloy family of compositions, whether based on nickel, cobalt or nickel-iron systems, is not immune to such property changes. Indeed, due to their high service temperatures, these alloys usually exhibit large changes in their structure sensitive properties during normal service. Properties that can be so affected include: tensile strength and ductility, creep, rupture, rupture ductility and all modes of fatigue, including crack growth rates. These changes in properties can be major. Nevertheless, the design community continues to design on the basis of as-heat treated properties. A better appreciation of the importance of structural stability remains one area through which further improvements in superalloy properties can still be achieved. This paper reviews the structural changes that can occur in all of the components of a superalloy. These include the precipitation of unwanted topological-closed packed phases and other intermetallics, change in carbides and boride structures, and changes in the amount and morphology of gamma prime (γ') and similar strengthening precipitates. The treatments of the approaches that have been developed to predict, or control such structural changes is included, and where possible, the effect of such changes have on properties is documented. The available data on the long time stability of superalloys is summarized in tabular form, listing the published studies on stability, nature of the precipitation reactions, and documented effects on properties for each commercial alloy. The morphology of major structural changes is illustrated. If the structural changes that an alloy undergoes in service, particularly the size and amount of γ' and γ", are characterized, such data can be used to estimate the service temperature that the engine component encountered. The techniques for estimating service conditions, from such measurements, so useful in failure analysis, are presented.

9:10 AM INVITED PAPER
LONG-TERM MICROSTRUCTURAL STABILITY OF SINGLE CRYSTAL SUPERALLOYS: *W. S. Walston*[1]; [1]GE Aircraft Engines, One Neumann Way, Cincinnati, OH USA

Long-term microstructural stability is a key parameter in the development and application of single crystal superalloys to gas turbine engines. The formation of topologically close packed (TCP) phases occurs to some degree in most single crystal superalloys. The effect of these phases on properties will be discussed. In addition, the effects of time and temperature on the formation of TCP phases will be shown for recent single crystal superalloy, including René N6. Another form of microstructural instability has recently been observed in high refractory content single crystal superalloys. This instability, termed SRZ, is a cellular phase transformation that can occur either beneath coatings or in the alloy substrate. The occurrence and effects of this instability in various alloys will be discussed. The third form of microstructural instability observed in several single crystal superalloys is the conversion of the γ matrix with γ' precipitates to a microstructure with a continuous γ' phase. Several examples of this behavior will be shown. The relationship between this form of instability, rafting and creep rupture properties will also be discussed.

9:40 AM
THERMAL STABILITY OF HIGH TEMPERATURE STRUCTURAL ALLOYS: *R. K. Rasefske*[1]; A. Castagna[1]; C. E. Jordan[1]; [1]Lockheed Martin, P.O. Box 1072, Schenectady, NY 12301-1072 USA

High temperature structural alloys are under evaluation for numerous applications which require long term operation at elevated temperatures. The effect of elevated temperature exposure on the microstructure and mechanical properties of a number of alloys was characterized. Iron-based alloys (330 stainless steel, 800H and MA 956) and nickel-based alloys (Hastelloy X, Haynes 230, 718 and MA 754) were evaluated for room temperature tensile and impact toughness properties after exposure at 750°C for 10,000 hours. Of the iron-based alloys evaluated, Alloy 800H showed the greatest degree of primary carbide coarsening and a corresponding reduction in ductility and toughness as compared to the unexposed condition. Within the group of nickel-based alloys tested, Alloy 718 showed the most dramatic change in structure as it formed delta phase during the long time exposure at 750°C with significant reductions in strength, ductility, and toughness. Haynes 230 and Hastelloy X showed significant carbide precipitation and a resulting reduction in ductility and toughness. Haynes 230 was also evaluated for room temperature tensile and impact toughness properties after 10,000 hours of exposure at 480°, 590°, 850°, 950° and 1050°C. Microstructural evaluation showed that extensive precipitation of $M_{23}C_6$ carbides occurred at 590°C. At 750°C the precipitates of $M_{23}C_6$ carbides coarsened, and primary M_6C carbides were found to be W and Mo rich, and contain little Cr. For exposures above 850°C, the M_6C carbides changed morphology from blocky to a more irregular type shape. Mechanical testing of the thermally exposed Haynes 230 specimens indicated relatively small reductions in ultimate tensile strength and yield strength over the range of exposure temperatures studied, however, large reductions in impact strength were evident after exposure at temperatures of 750°C and above.

10:00 AM
THE EFFECT OF LONG TERM ISOTHERMAL EXPOSURE ON THE MICROSTRUCTURE AND PROPERTIES OF INCONEL® ALLOY 783: *Sarwan Mannan*[1]; John deBarbadillo[1]; Stanley Gregory[1]; [1]Inco Alloys International, Inc., 3200 Riverside Dr., Huntington, WV 25705-1771 USA

Recently developed INCONEL® alloy 783 (nominal composition of Ni-34Co-26Fe-5.4Al-3Nb-3Cr) is precipitation strengthened by Ni3Al-type Gamma Prime and NiAl-type Beta phases. Due to its low coefficient of thermal expansion, high strength, and good oxidation resistance, alloy 783 was designed for use in aircraft gas turbine components such as rings, casings, shrouds, and seals and has been considered for use in a number of other critical industrial turbine components. In this study, commercially produced alloys 783, 718 and 909 were annealed and aged using recommended heat treatments. The materials were then isothermally exposed at 1100°F (593°C) for times up to 10,000 hours. After every 1000 hours of exposure, specimens were subjected to room temperature tensile (RTT) and high temperature tensile (HTT) testing. The microstructure of as-produced and exposed materials was characterized using optical microscopy, scanning electron microscopy and transmission electron microscopy. Variation in tensile properties with iso-

thermal exposure time was correlated with the microstructure. Further, exposed specimens were HTT tested in air and vacuum to evaluate the effect of test environment. For alloy 783, creep crack growth tests were carried out in the as-produced and isothermally exposed conditions to determine the effect of exposure on the crack growth. INCONEL® is a registered trademark of Inco group of companies.

10:20 AM BREAK

10:40 AM INVITED PAPER
ACCELERATED HIGH TEMPERATURE PERFORMANCE EVALUATION FOR ALLOY OPTIMIZATION AND REMAINING LIFE ASSESSMENT: *David A. Woodford*[1]; [1]Materials Performance Analysis, 1707 Garden St., Santa Barbara, CA 93101 USA

Creep strength and fracture resistance are two properties that are critical in the selection and optimization of high temperature alloys. These same properties may be progressively impaired by service exposure and are, therefore, key to the assessment of remaining life of high temperature components. In recent years, a methodology based on accelerated measurements of these properties using, respectively, a self-programmed variable stress creep test (stress relaxation test), and a constant displacement rate notched tensile test, has been developed. The methodology, referred to as Design for Performance, decouples these two properties. Changes in creep strength due to thermal exposure are determined principally by microstructural evolution, whereas changes in fracture resistance may be dominated by environmental interactions. Examples of the approach applied to alloy optimization and to component remaining life prediction are drawn from ferritic steels, and wrought and cast nickel based alloys, including monocrystals.

11:10 AM INVITED PAPER
CALCULATED PHASE EQUILIBRIA AND ITS USE IN PREDICTING LONG THERMAL STABILITY: *Nigel Saunders*[1]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey GU2 5YG UK

Knowledge of the stable equilibrium state of a material is directly applicable to issues of long term thermal stability as it describes the state which the material ultimately tries to achieve. It is particularly useful in circumstances where kinetics enables equilibrium, or near equilibrium, to be reached. The CALPHAD route provides an excellent method by which phase equilibria can be predicted in complex alloys (ref.1). Using this approach it is then further possible to predict critical temperatures and driving forces for various transformations which can be used in kinetic equations so that rates of transformations can be predicted (ref.1). The present paper describes the current state of the art in predicting phase equilibria via the CALPHAD method for a number of material types, including Fe- and Ni-based alloys, and gives examples of its use in the area of long term thermal stability. Reference 1. N. Saunders and A. P. Miodownik, "CALPHAD - A Comprehensive Guide", Elsevier, Science, N.Y., 1998

11:40 AM
THE EFFECT OF LONG-TERM THERMAL EXPOSURE ON THE MECHANICAL PROPERTIES OF FOUR MODERN HIGH-TEMPERATURE NICKEL-BASE ALLOYS: *R. Rodger Seeley*[1]; S. Krishna Srivastava[1]; [1]Haynes International, Inc., Eng. & Tech., 1020 West Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

The effect of long-term thermal exposures on the mechanical properties and microstructures of four modern nickel-base superalloys have been studied. The HAYNES® 230®, HR-120®, 556™, and 242® alloys were exposed at temperatures up to 980ϒC for times up to 20,000 hoours. The mechanical properties evaluated are tensile strength, ductility, Charpy V-notch impact energy and elastic-plastic fracture toughness. The microstructures and fracture characteristics of the materials are discussed. Long-term thermal exposures generally resulted in higher strength and lower ductility at room temperature. Fatigue at room temperature and moderately high temperatures is also effected. Low toughness values were accompanied by intergranular fracture characteristics as determined by optical and scanning electron microscopy. The fracture toughness of this material appears to be closely related to the ductility. Even for the severest exposure conditions, most high-temperature alloys still retain useable toughness.

MANUFACTURING ISSUES IN RAPID THERMAL PROCESSING: Session I

Sponsored by: Electronic, Magnetic and Photonic Materials Division, Thin Films and Interfocus Committee
Program Organizers: N.M. Ravindra, New Jersey Institution of Technology, Dept. of Phys., Newark, NJ 07102 USA; Daniel F. Downey, Varian Ion-Implant System, Gloucester, MA 01930 USA; Anthony T. Fiory, Lucent Technologies, Bell Labs., Room 1D468, Murray Hill, NJ 07974-0636 USA; Steven D. Marcus, AST Elektronik USA Inc., Tempe, AZ 85284 USA; B. Sopori, National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA

Wednesday AM Room: 14A
March 3, 1999 Location: Convention Center

Session Chair: Bhushan L. Sopori, National Renewable Energy Laboratory, Golden, CO 80401 USA

8:30 AM INVITED PAPER
ULTRA-SHALLOW JUNCTION FORMATION BY RAPID THERMAL ANNEALING OF ION-IMPLANTED DOPANTS: *Aditya Agarwal*[1]; [1]Eaton Semiconductor Equipment Operations, 55 Cherry Hill Dr., Beverly, MA 01915 USA

This paper reviews recent progress in the understanding of dopant diffusion and activation, and discusses emerging strategies for formation of the extremely shallow, low-resistivity junctions required for future CMOS IC device technology generations. Shallow junction formation requires an integrated process consisting of ion implantation(I/I) and rapid thermal annealing (RTA). Process optimization therefore requires the contribution of the III and RTA parameters to be understood both separately and together. A short list of these junction formation process parameters includes: I/I -dopant species, energy and dose, as well as preamorphization implant parameters (if any); RTA-ramp-up rate, temperature control and uniformity, time and ambient. The interdependence of these parameters is exhibited by enhanced diffusion phenomena such as transient- and boron-enhanced diffusion (TED and BED). Other examples of the interdependence of the junction formation process parameters include the tradeoff between implant dose and annealing temperature, and the role of preamorphization in increasing dopant activation. These phenomena will be illustrated with experimental data. A RTA strategy, which has emerged in the last couple of years, is spike annealing, characterized by very high ramp-rates and soak times of the order of only tens of milliseconds. Spike anneals are capable of further reducing sheet resistance by allowing higher temperature anneals while limiting the overall thermal budget for diffusion. Experimental data from several sources will be compared. While the primary method for achieving ultra-shallow junctions in the foreseeable future (down to 0.13 micron technology node) appears to be RTA of ultra low energy implanted dopants, much progress has recently been made with alternative technologies such as dopant introduction by plasma immersion ion implantation (Pill), or dopant activation by laser thermal processing (LTP). The progress and limitations of these technologies will be discussed.

9:00 AM INVITED PAPER
EFFECTS OF "FAST" RAPID THERMAL ANNEALS ON SUB-KEV BORON AND BF_2 ION IMPLANTS: *Daniel F. Downey*[1]; Judy W. Chow[1]; Adam F. Bertuch[1]; Steven D. Marcus[2]; [1]Varion Ion Implant Systems, Gloucester, MA 01930 USA; [2]STEAG AST Elektronik Inc., Tempe, AZ 85284 USA

The effects of "fast" ramp-rates and spike anneals are investigated for 0.25, 0.5 and 1.0 keV "B" and for 1.1 and 2.2. keV BF_2. Below an implant energy threshold where no extended defects occur, fast ramp-rates become important in minimizing the thermal diffusion component and reducing the junction depth. Above this implant energy threshold, TED minimizes the advantages of these fast ramp-rates. Annealing

in a low and controlled 02 ppm in N_2 ambient further reduces diffusion by inimizing/eliminating oxygen related enhanced diffusion effects, while simultaneously optimizing anneal reproducibility and across-the-wafer uniformity. This paper identifies these implant and anneal conditions/ thresholds where diffusion is solely governed by thermal diffusion; and demonstrates how fast ramp-rates and spike anneals can be implemented to minimize diffusion in a controlled, reproducible and uniform 200 mm wafer process.

9:30 AM

THERMAL ACTIVATION OF SHALLOW BORON IMPLANTS: *A. T. Fiory*[1]; K. K. Bourdelle[2]; [1]Bell Laboratories, Lucent Technologies Inc, Murray Hill, NJ 07974 USA; [2]Bell Laboratories, Lucent Technologies, Inc., Orlando, FL 32819 USA

Boron implanted into n-type Si with dose in the 10^{15} cm^{-2} range and with energies from 500 eV to 5 keV was activated by annealing in nominally pure N2 and in N2 with small admixtures of 02. Various temperature-vs-time heating cycles were examined. The lowest thermal budget used heating rates up to 150ϒC/sec, cooling rates up to 800°C/sec, and minimal dwell time at the maximum temperature. Dopant activation was characterized by sheet electrical measurements. Surface oxidation was characterized by film thickness ellipsometry. Defined p-n junction depths were inferred from analysis of electrical measurements and secondary ion mass spectroscopy profiles. Fractions of activated dopant increase with boron diffusion from the implanted region, Surface oxide serves to retard dopant loss to the ambient for high-temperature anneals.

9:50 AM INVITED PAPER

SECONDARY DEFECT PROFILE RELATED TO LOW ENERGY IMPLANTED BORON MEASURED UP TO 3.5 UM DEPTH INTO Si-SUBSTRATES: *L. Soliman*[1]; M. Benzohra[2]; P. Martin[1]; K. Katata[1]; F. Boussaid[1]; A. Martinez[3]; M. Ketata[1]; [1]LEMI, Universite de Rouen, 76821 Monti-Saint-Aigman Cedex France; [2]LAAS-CNRS, 7 AV, Colonel Roche, Toulouse Cedex 310077 France; [3]INSA, Complexe Scientifique de Rangueil, Cedex 31400 France

Low energy implantation is the most promising option for ultra shallow junction formation in the next generation of silicon BICMOS technology. Among the dopants that have to be implanted, boron is the most problematic because of its low stopping, power and its tendency to undergo transient enhanced diffusion and clustering during thermal activation. This paper reports an experimental contribution with the help of secondary defect profiles to our understanding of low energy B implants in crystalline silicon. Shallow p^+n junctions were formed by low energy B Implantation 10^{15} cm^{-2} at 3 keV - into a reference or preamorphized with germanium - 10^{15} cm^{-2} at 30 KeV - n-type crystalline silicon. Rapid Thermal Annealing (RTA) for 15 sec at 950ϒC was then used. Secondary defect profiles induced by this process are measured with isothermal transient capacitance in association with Deep Level Transient Spectroscopy (DLTS). Relatively high concentrations of electrically active defects have been obtained up to 3.5 um into the crystalline silicon bulk. The relation of these defects with boron is discussed. The result is in agreement with boron transient enhanced diffusion in Si-substrate as E.J.H. Collart has reported it with back samples S.I.M.S. measurements.

10:20 AM BREAK

10:40 AM INVITED PAPER

ULTRA SHALLOW JUNCTION FORMATION OF SOURCE/ DRAIN EXTENSIONS FOR 100 NM TO 180 NM TECHNOLOGIES: A PRODUCTION WORTHY PROCESS: *Steven D. Marcus*[1]; Daniel F. Downey[2]; Wilftied Lerch[3]; Judy W. Chow[2]; Adam F. Bertuch[2]; [1]STEAG AST Elektronik, Tempe, AZ 85294 USA; [2]STEAG AST Elektronik, Gambh, Dorristadt Germany; [3]Varian Ion Implant Systmes, Gloucester, MA 01930 USA

The successful formation of S/D extensions that satisfy the National Technology Roadmap for Semiconductors (NTRS) for sub 180nm technologies has been previously reported. However, to successfully implement this process into production, precise and repeatable control of a number of variables is paramount. The substrate; the low energy implant process; the RTA temperature, temperature overshoot, anneal time, uniformity and ambient control all must be taken into consideration. The repeatability of 0.25 to 1 keV B^{11} 1.1 to 2.2 keV BF_2 and 0.5 to 2 keV As+ implants to a dose of le15/cm^2 using the Varian VIISta-80 and VIISion-80 PLUS will be demonstrated. The STEAG AST Elektronik AST3OOO rapid thermal processor will be used to demonstrate the post implant anneal repeatability. Ramp up and ramp down rates, spike vs. l0s dwell times and oxygen concentrations have the most influence on the final junction parameters. Repeatability of<1% 1sigma will be demonstrated for average Rs, junction depth and machine specific parameters for 50 and 250ϒC/s ramp up rates in conjunction with either .l0s or spike (0s) anneals. 1000ϒC (l0s) and 1050ϒC (0s) anneals will be used to gauge the performance.

11:10 AM

INFLUENCE OF VAPOR PHASE CLEANING AND RAPID THERMAL OXIDATION AND NITRIDATION ON THE SILICON - SILCONDIOXIDE INTERLACE: *N. Sacher*[1]; B. Froeschle[1]; F. Glowacki[1]; [1]STEAG AST Elektronik, Gambh, Daimierstrasse 10, Dornstadt D-89160 Germany

For 0.18 UM technology and beyond the properties of the Si/SiO_2 interface becomes more and more important Therefore a Vapor Phase Cleaning Module (VPC) is integrated in a gate Oxide Cluster Tool to ensure a good quality of the interface. As pre-oxidation cleaning different types of vapor phase cleaning (VPC) are performed in an STEAG AST Vapor Phase Cleaning Module and compared to standard wet cleaning. The oxidation, performed in a STEAG AST RTP Module, was carried out in pure oxygen 0_2 or in nitric oxide (NO). Also some oxides has been nitrided by a NO-anneal, which follows directly the oxidation. The nitrogen incorporation is investigated using Secondary Ion Mass Spectroscopy (SIMS). The roughness after the cleaning is analyzed by Atomic Force Microscopy.

11:30 AM INVITED PAPER

THIN SiO_2 FILMS GROWN FOR BRIEF OXIDATION TIMES: *A. T. Fiory*[1]; [1]Bell Laboratories, Lucent Technologies, Inc., Murray Hill, NJ 07974 USA

Bulk p -type wafers prepared with HF and RCA cleaning were oxidized in an atmospheric pressure 0_2 ambient in an incandescent-lamp processor. Minimal effective oxidation times of several seconds were obtained by rapidly heating wafers at rates up to 150ϒC/sec and then turning off lamp power just as the desired peak temperature is approached. Cooling rates vary up to about 80ϒC/sec. Films thicknesses obtained by this method increase from about 16 A for peak temperature of 1000ϒC to about 22 A for peak temperature of 1100ϒC. Oxidation process uniformity under 1% at one standard deviation over 1 5Oum wafers, equivalent to 2.5ϒC temperature variation, was obtained by optimizing relative power ratios to the lamps.

MATERIALS PROCESSING FUNDAMENTALS: Synthesis & Processing

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

Program Organizers: W. D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Huimin Liu, UES, Inc., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Laboratory, P.O. Box 2008 Bldg. 4508, Oak Ridge, TN 37831-6083 USA

Wednesday AM Room: 5A
March 3, 1999 Location: Convention Center

Session Chairs: Huimin Liu, UES Software, Inc., Annapolis, MD 21401 USA; Samim Anghaie, University of Florida, INSPI, Gainesville, FL 32611-6502 USA

8:30 AM

ELECTRICALLY POWERED PROCESSING OF NiCrAlY ALLOY POWDER: *J. M. Doh*[1]; S. K. Hur[2]; J. R. Groza[3]; [1]Korea Institue of Science and Technology, Division of Metals, P.O. Box 131, Cheongryang, Seoul 130-773 Korea; [2]Changwon National University, Dept. of Industrial Engineering, Changwon, 641-773 Korea; [3]University of California at Davis, Chemical Engineering & Materials Science Dept., Davis, CA 95616 USA

In the last decades, numerous field assisted sintering processes have been developed. Common to all these processes are particles surface phenomena related either to the electrical breakdown of surface oxides or local melting of dielectric oxide films with higher resistance. Among them, field activated sintering technique (FAST) has been recognized as very efficient in providing a quick densification. This process involves an intensive electrical discharge pulse at modest mechanical pressure application associated with resistive heating of the powder compact. Compared with other field assisted sintering processes, this process may benefit from a cleaning step due to the discharge pulse application. It is assumed that the gas molecules adsorbed on the particle surfaces and surface oxides are removed through ionization or excitation. However, the specific effect of the pulsed current application is largely unknown. The purpose of the present work is to examine the sintering characteristics of NiCrAlY and to investigate field effects on powder surface chemistry and microstructure of NiCrAlY under air, vacuum, and nitrogen atmosphere when the pulsed current (alternative current) is used for the densification of NiCrAlY powders.

8:55 AM

CaCO3 AS A SOURCE OF CALCIUM FOR ELECTROCHEMICAL SYNTHESIS OF Pb-Ca ALLOYS: Derek J. Fray[1]; *Evguenia B. Freidina*[1]; [1]University of Cambridge, Materials Science and Metallurgy, Pembroke St., Cambridge, Cambridgeshire CB2 3QZ UK

We have investigated a new electrochemical process for preparing lead-calcium alloys. Calcium metal, reduced from a molten chloride mixture, forms an alloy with liquid lead cathode. The addition of calcium carbonate to conventional CaCl2-NaCl electrolyte enables us to avoid chlorine production on the anode and replace it with oxygen and carbon dioxide. Study of the ternary system CaCl2-NaCl-CaCO3 has shown a range of melt compositions, where the electrolysis can be held below 650Ỿ. In order to prevent the side reaction of carbon deposition CO3(2-) + 4e(-)=C(s) + 3O(2-) on the cathode we have separated the anolyte from the catholyte with alumina diaphragm. The factors, which affect the level of impurities and the current efficiency of the process, are discussed.

9:20 AM

REDUCTION OF SULFUR DIOXIDE BY CALCIUM SULFIDE TO PRODUCE ELEMENTAL SULFUR: *Byung-Su Kim*[1]; Hong Yong Sohn[1]; [1]University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112-0114 USA

The recovery of elemental sulfur from sulfur dioxide has important implications for coal-burning power plants and nonferrous metal smelters. The thermodynamic analyses showed calcium sulfide to be suitable for recovering elemental sulfur from sulfur dioxide. At the temperatures of 973 to 1153K under the sulfur dioxide partial pressures of 5~60 kpa elemental sulfur from sulfur dioxide was recovered by calcium sulfide. The kinetics of this reaction were measured at the same conditions using a thermogravimetric analysis technique in the absence and presence of vanadium catalyst. The reactivity of regenerated calcium sulfide was also investigated. At 1073K under the sulfur dioxide partial pressure of 25.8 kpa, 50% of the original calcium sulfide in the absence, and 60% in the presence, of the vanadium catalyst was converted to calcium sulfate in an hour. The effect of the vanadium was lower in the regenerated calcium sulfide. At 1073K under the sulfur dioxide partial pressure of 25.8 kpa in the absence of the van adium catalyst, 55% of the regenerated calcium sulfide were converted to calcium sulfate in an hour even after three cycles. A "pore blocking" model was found to fit the reaction rate reasonably well.

9:45 AM

HIGH TEMPERATURE INTERACTIONS BETWEEN SILICON NITRIDE AND TITANIUM CARBIDE: *Hurman Rauf Eric*[1]; Nedret Can[2]; [1]Delft University of Technology, Faculty of Applied Earth Sciences, Mijnbouwstraat 120, Delft 2628 RX The Netherlands; [2]University of the Witwatersrand, School of Process and Materials Engineering, Private Bag 3, Johannesburg, Gauteng WITS 2050 South Africa

Kinetics and mechanism of the chemical interactions between silicon nitride and titanium carbide were investigated by isothermally reacting samples under nitrogen and argon atmospheres at temperatures between 1600//068 and 1700°C. The powder compact initially contained 20, 30, and 40 volume per cent TiC. Kinetic analyses were performed by measuring weight losses and determining the composition of titanium carbonitride ($TiC_{1-x}N_x$) at predetermined time intervals. It was found that the degree and rate of reactions increased with increasing temperature and TiC content under both nitrogen and argon atmospheres. Silicon nitride in the presence of TiC was stable under nitrogen atmosphere and the reactions were confined to silicon nitride-TiC and TiC-gas(nitrogen) interfaces. At both interfaces reactions occur by simultaneous atomic diffusion of nitrogen towards the center of the particle and desorption of carbon in the opposite direction resulting in formation of titanium carbonitrides of varying compositions. The desorbed carbon diffuses to particle surface and reacts with silicon nitride and forms SiC. This reaction proceeds by the formation of an intermediate layer of liquid silicon. Reaction products under nitrogen were β-SiC, titanium carbonitride and nitrogen gas. The maximum value of "x" in the titanium carbide formula was found to be 0.67. Silicon nitride was unstable under argon atmosphere in the presence of TiC and dissociated completely into liquid Si and nitrogen within approximately four hours of reaction time, depending upon temperature. The reaction mechanism at the initial stages under argon atmosphere is similar to the one under nitrogen. However, considerable amount of silicon nitride dissociates within the first half hour. Liquid Si aggressively attacks both titanium carbonitride and partially reacted TiC particles through cracks and pores and diffuses into the interior parts of the particle forming liquid solutions. After this stage, reactions under argon atmosphere proceeds as can be predicted from the ternary Si-Ti-C system.

10:10 AM BREAK

10:20 AM

PROCESSING AND TESTING OF PSEUDO-TERNARY CARBIDE FUELS FOR HIGH TEMPERATURE SPACE NUCLEAR REACTORS: *Travis Warren Knight*[1]; Samim Anghaie[1]; [1]University of Florida, INSPI, P.O. Box 116502, Gainesville, FL 32611-6502 USA

The development of various processing techniques for pseudo-ternary carbide nuclear fuels—namely (U, Zr, Nb)C and (U, Zr, Ta) C—is presented. Pseudo-ternary carbide samples of low uranium content

(<1g/cm3) have been investigated for the their improved thermochemical stability for application in high temperature space nuclear reactors and propulsion systems. The high melting points of pseudo-ternary carbide fuels (typically >3200K) provide for a higher service temperature and a greater specific impulse (Isp) than previous nuclear thermal propulsion designs. Techniques have been developed for the processing of pseudo-ternary carbides with efforts directed toward the development of net-shape fabrication of fuel elements. Preliminary results show that these processing techniques could be used in the fabrication of new, innovative core designs such as the square lattice or "honeycomb" design. The paper discusses the development of processing techniques for pseudo-ternary carbides and plans for hot hydrogen testing and the development of net-shape processing of pseudo-ternary carbide fuel elements.

10:45 AM

SYNTHESIS OF STABILIZED POTASSIUM FERRATE AND ITS APPLICATION IN WATER TREATMENT: *N. Neveux*[1]; N. Kanari[1]; N. Aubertin[1]; O. Evrard[1]; [1]LEM, Associated to CNRS UMR 7569, Mineral Processing and Environmental Engineering, ENSG, INPL, Vandceuvre BP 40, 54501 France;[2]Universite Henri Poincare Nancy I, Laboratoire de chimie du solide Minerale, Associated to CNRS UMR 7555, Vandceuvre BP 40, 54501 France

For the first time, dehydrated ferrous sulfate was successfully used for the diy synthesis of potassium ferrate. Moreover, ferrate ion is stabilized as a solid solution where hexavalent iron is partially substituted by S^{VI} leading to a sulfato-ferrate having the following formula K2(Fe,S)04. This substance has an efficient life time of about six months. Ferrate ion is a powerful oxidant (F^{o} = 2.2 V), a bactericidal agent and possesses coagulant, flocculant properties. Its reduction generates nascent oxygen, hydroxyl groups and $Fe(OH)_3$. Alkali metal ferrate is probably the future global agent for water treatment. Applications of this material in the field of urban and industrial effluents' treatment have been investigated. Heavy metals' removal of cations such as Ag, Cu, Cd, Mn, Ni, Pb, Zn, was successful. Results of sulfato-ferrate use for cyanide's destruction, decrease of COD, soil remediation, discoloration of pulp and paper effluents will be summarized.

11:10 AM

SYNTHESIS OF AL_2O_3-WC COMPOSITE POWDER FOR CUTTING TOOLS BY SHS PROCESS: *J. Zhang*[1]; J. J. Lee[1]; C. W. Won[1]; S. S. Cho[1]; B. S. Chun[1]; [1]Chungnam National University, Rapidly Solidified Materials Research Center (RASOM), Taejon 305-764 Korea

Al_20_3.WC Composite powder for cutting tools was synthesized by self-propagating high-temperature synthesis using Al powder as a reducing agent. WC, W2C, and A203 were concurrently formed in WO3-Al-C system. It was found that the complete reaction was achieved with excessive addition of carbon and appropriate processing parameters such as degree of dilution, particle size of aluminum, compaction pressure of green pellets and carbon source. The final product which was leached by 50% HNO3/HF diluted solution was consisted of M203-55 wt% WC having 2-3um of mean particle size.

11:35 AM

PREPARATION OF HIGH PURITY Si POWDER FOR ELECTRONIC DEVICE BY SHS PROCESS: *J. H. Lee*[1]; [1]Chungnam National University, Rapidly Solidfied Materials Research Center (RASOM), Taejon 305-764 Korea

High-purity Si powder for electronic devices especially raw material for wafer was prepared by the self-propagating high-temperature synthesis from a mixture of SiO_2 and Mg. The MgO in the product was leached with dilute HCl solution. The complete reduction of SiO_2 required excess magnesium than the stoichiometric mole ratio. The product silicon had a purity of 99.988% which was higher than that of the reactant SiO_2. This is because the impurities were either volatilized at the high temperature generated during the rapid exothermic reaction or dissolved into the HC1 solution during leaching.

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

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

Wednesday AM Room: 14B
March 3, 1999 Location: Convention Center

Session Chairs: Vijay Gupta, UCLA, Dept. of Mech. & Aero. Eng., Los Angeles, CA 90095-1597 USA; Sia Nemat-Nasser, UCSD, Center of Excellence for Adv. Mat., San Diego, CA 92093-0416 USA

8:30 AM INVITED PAPER

STRUCTURE AND PROPERTIES OF METAL/CERAMIC INTERFACES: *Manfred Ruhle*[1]; [1]Max-Planck-Institut fur Metallforschung, Seestr. 92, Stuggart D-70174 Germany

Properties of metal/ceramic interfaces determine the properties of bulk composites which are made of different constituents. Therefore, it is crucial to understand the correlation between structure and properties of those interfaces. Model studies were performed for diffusion-bonded materials where two single crystalline specimens were bonded. The atomic structure of the interface can best be revealed by transmission electron microscopy (TEM) techniques. The chemical composition of the interface and the regions near the interface can be determined by analytical electron microscopy (AEM) techniques. Information on bonding across the interface can be obtained by studies of the energy-loss near-edge structure. Mechanical four-point bending tests were done. The fracture energy is determined as a function of the diffusion-bonding conditions, different orientations and chemical composition at the interface. Results for Nb/Al2O3 and Cu/Al2O3 will be reported and compared with models existing in the literature.

9:00 AM

NANO-SCALED INTERFACIAL DEBONDING IN MULTILAYED ALUMINUM OXIDE/GOLD MATERIALS (EXPERIMENT AND THEORY): *Scott X. Mao*[1]; [1]The University of Calgary, Dept. of Mech. Eng., 2500 University Dr. NW, Calgary, Alberta T2N 1N4 Canada

To understand the interfacial cracking in thin ceramic/metal layered material is key important in the thin film and coating technology. Insitu observation on interface crack growth of microlayered aluminum oxide/gold material has been carried out. It has been found that voids exist along the interface and act as debond sources for interfacial cracking. When main crack front approaching these interfacial voids, high peak stress generated by the constrained metal layer in the front of the main crack is then acting on the voids and debonds occur. From the observation on the debonds surface by atomic force microscope AFM, we found that the debonds propagate by dislocation slip steps. The width of each slip step is 1 micro meter and height is ~10 nano meters. Based on the AFM observation, a super dislocation model in metal/ceramic layered material has been proposed to describe the crack tip stress, strain energy release rate. The crack tip blunting and toughening due to dislocation shielding from interface crack has been analyzed. The meal length scale (thickness) effect on the interfacial cracking and toughening has been found.

9:20 AM INVITED PAPER

SCRATCH OF POLYMER COATINGS WHICH ADHERES TO THE INDENTER: *James C. M. Li*[1]; [1]University of Rochester, Materials Science, College of Arts, Sciences and Eng., Rochester, NY 14627 USA

The load displacement relations for a spherical indenter which adheres to a film coating over a substrate is studied including the area of contact as a function of the normal load, film thickness and the interfacial energy between the film and the indenter. This indenter is then used to scratch such a film coating so that the relation between the horizontal force required as a function of normal load and the scratching speed can be understood. In the elastic or anelastic region, there are no scratch marks left afterwards. But when the normal load exceeds a critical value, the film is damaged with a possible de-adhesion of the film from its substrate. Since the scratch test is thermodynamically unstable, it bifurcates into stick/slip stages which will be described. Work supported by NSF through DMR 9623808 monitored by Dr. Bruce MacDonald. An Industrial Fellowship award from Eastman Kodak Company and the NSF Institute for Mechanics and Materials directed by Marc Meyers is gratefully acknowledged also.

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QUANTITATIVE CHARACTERIZATION OF SCRATCH AND MAR BEHAVIOR OF POLYMERIC COATING SURFACES: *Li Lin*[1]; Gregory S. Blackman[2]; Robert R. Matheson[1]; [1]Dupont Company, Marshall R&D Laboratory, 3401 Grays Ferry Ave, Philadelphia, PA 19146 USA; [2]Dupont Company, CR&D, Experimental Station, E323-110B, Wilmington, DE 19880 USA

Scratch and mar damage of automotive topcoats is of increasing concern to the automobile industry and its customers. To better characterize and understand scratch and mar behavior, we developed a microscratch technique which measures normal and tangential force with a few micronewtons precision, and penetration depth with a few nanometers precision as function of time or position during a scratch experiment. With this technique, scratch and mar resistance is evaluated based on plastic and fracture thresholds. This technique allowed us to prepare well defined scratches in terms of damage type (plastic or fracture) and size. A statistic survey was conducted relating these damages to visual appearance. In addition, loading rate, scratch rate, aging and other parameters that may effect scratch and mar resistance of polymer coatings were also examined.

10:10 AM BREAK

10:20 AM INVITED PAPER

MEASUREMENT OF THE INTERFACE CRACK VELOCITY AND THE BRITTLE-TO-DUCTILE TRANSITION TEMPERATURE : *Vijay Gupta*[1]; Michael O'Brien[1]; Jianxin Wu[1]; [1]University of California, Dept. of Mechanical and Aerospace Engineering, 38-137E, Engg. IV Bldg., Los Angeles, CA 90095-1597 USA

A technique to measure the interface crack velocity has been developed with a view to study the brittle-to-ductile transition phenomenon in bimaterial interface systems involving metallic coatings. The experimental strategy involved generating a line crack of approximately 100 μ/m in extent at an interface between a 1 μ/m-thick and 600 μ/m wide Nb strip and a (0001) surface of sapphire, using photolithography. A microcircuitry consisting of equi-spaced Al wires of 100 A thickness was deposited orthogonally on top of the Nb strip, including the areas beyond the crack tips. A compressive stress wave of approximately 20 ns duration was generated on the backside of the sapphire substrate by exfoliating a constrained Al film by a Nd:YAG laser pulse. The compressive stress wave upon reflecting into a tensile wave from the free surface of the coating led to the initiation of the interface crack. The critical stress pulse amplitude was obtained by recording the transient velocity of the sapphire's free surface, away from the crack tip region, by using an optical interferometer. Prior to wave loading, a small current was introduced in the Al wire mesh to establish a circuit such that the voltage drop across the reference resistor was proportional to the number of Al lines broken by the propagating crack front. Because the voltage-change occur rather quickly, it was impossible only to record changes proportional to this voltage on a fast digitizer with a 5 ps rise-time. Thus, the recorded voltage-time record provided information only on the total duration of crack propagation. An independent measure of this parameter was also obtained by recording free surface velocity of the sapphire by an interferometer. To compute average crack velocity, the total crack advance during this time was obtained by viewing the shock-loaded specimens under a scanning electron microscope. The measured critical free surface velocity at crack initiation was used to calculate the energy flow at the crack tip (equal to the interface toughness) by using a simulation based on dynamic fracture. Thus, the present setup allows the measurements of both the interface crack velocity and the interface toughness. At ambient temperature, the interface crack velocity was found to approach the Raleigh wave speed of the stiffer material, which challenges all the presently available models of dynamic fracture as the energy release rate becomes zero at the measured crack speeds. The above apparatus is being used to measure the interface crack speeds and toughness as a function of temperature. This should lead to the determination of the brittle-to-ductile transition temperature, beside providing a means to understand the local inelastic processes that contribute to the plastic component of the measured interface toughness.

10:50 AM

MICROMECHANICS OF COMPRESSION FAILURE OF FIBER-REINFORCED COMPOSITES: *Jeffrey D. McGee*[1]; Sia Nemat-Nasser[1]; [1]University of California, San Diego, Center of Excellence for Advanced Material, 9500 Gilman Dr., San Diego, CA 92093-0416 USA

Micromechanics-based modeling of failure mechanisms in heterogeneous and anisotropic composites is considered, focusing on the phenomenon of kink-band formation in woven composites under biaxial compressive dynamic loads. Using a modified split-Hopkinson bar, samples of woven fiber-reinforced polymeric composites are subjected to biaxial compression, and the process of initiation and growth of kink bands is captured by high-speed photography. A micromechanics model is developed to describe this phenomenon by homogenizing a periodic composite with woven fibers into a homogenous but anisotropic effective material and then examining the process of growth of an array of interacting wing cracks within the effective anisotropic solid. The resulting properties' degradation and the failure mechanisms are studied.

11:10 AM

INFLUENCE OF FIBER COATING THICKNESS ON FRACTURE BEHAVIOR OF CONTINUOUS WOVEN NiCaLoN® FABRIC-REINFORCED SILICON-CARBIDE MATRIX CERAMIC COMPOSITES: James H. Miller[1]; Rick A Lowden[1]; *Peter K. Liaw*[2]; John D. Landes[3]; [1]Oak Ridge National Laboratory, P.O. Box 6423, Oak Ridge, TN 37831-6423 USA; [2]University of Tennessee, Materials Science and Engineering, 427B Dougherty Building, Knoxville, TN 37996-2200 USA; [3]University of Tennessee, Mechanical and Aerospace Engineering and Engineering Science, Perkins Hall, Knoxville, TN 37996 USA

Nicalon® plain-weave fiber fabric-reinforced silicon carbide (SiC) matrix composites with various pyrolytic carbon fiber/matrix interface coating thicknesses have been successfully fabricated by forced chemical vapor infiltration (FCVI) methods. The influence of the carbon interface coating thickness on the fracture behavior of these fiber fabric-reinforced SiC composites has been investigated. Experimental results indicate that fiber coating thickness significantly alters the fracture behavior of SiC composites. The fracture strength exhibits a maximum as the coating thickness increases. A theoretical model has been developed to simulate the fracture behavior in the SiC composites with varied carbon interface coatings. The model assumes that microcracking, which is due to low matrix toughness, continuously initiates and arrests. The model-predicted fracture behavior compares well with the experimental results. This research was performed in cooperation with the University of Tennessee, Knoxville, under contract 11X-SN191V with Lockheed-Martin Energy Research Corporation and is sponsored by the US Department of Energy, Assistant Secretary for Conservation and Renewable Energy, Office of Industrial Technology, Industrial Energy Division, under contract DE-AC05-84OR21400 with Lockheed Martin Energy Research Corporation.

11:30 AM

DEFORMATION AND FRACTURE OF A COMPOSITE REINFORCED WITH BONE-SHAPED SHORT FIBERS: *Yuntian T. Zhu*[1]; James A. Valdez[1]; Michael G. Stout[1]; Ning Shi[2]; Shujia Zhou[3]; Darryl P.

Butt[1]; Terry C. Lowe[1]; [1]Los Alamos National Laboratory, Materials Science and Technology, MS G 755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Los Alamos Neutron Science Center, MS H805, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Applied Physics Division, MS B-258, Los Alamos, NM 87545 USA

The fiber-matrix interface in short-fiber composites is a limiting factor in improving mechanical properties such as strength and toughness. A strong interface makes it difficult to relieve fiber stress concentration in front of an approaching crack, and results in fiber breakage, while a weak interface cannot effectively transfer load from matrix to fiber. We have found that bone-shaped short-fibers can overcome this problem by transferring load through the enlarged fiber ends while having a weak interface. It is also found that bone-shaped fibers can effectively bridge cracks and to stop crack propagation. This presentation will discuss the deformation and failure processes of bone-shaped-short-fiber reinforced composites, and explain why the bone-shaped short-fiber composites have both higher strength and toughness than conventional short-fiber composites.

MINIATURE STRUCTURES & COMPONENTS UNDER CYCLIC LOADING; FATIGUE & INTERNAL FRICTION: Session III

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee; Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee; ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: H. D. Merchant, Gould Electronics, Inc., Eastlake, OH 44095-4001 USA; Thomas R. Bieler, Michigan State University, Dept. of Mats. Sci. & Mech., East Lansing, MI 48824-1226 USA; James C. Earthman, University of California, Dept. of Chem. Eng. & Mats. Sci., Irvine, CA 92717-2535 USA; M. Wuttig, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20743-2115 USA

Wednesday AM Room: 11B
March 3, 1999 Location: Convention Center

Session Chair: Harish Merchant, Gould Electronics, Eastlake, OH 44095 USA

8:30 AM INVITED PAPER
ELASTIC CONSTANTS OF METALLIC SUPERLATTICES: *Ivan K. Schuller*[1]; M. Grimsditch[2]; [1]University of California, San Diego, Physics Dept. 0319, 9500 Gilman Dr., La Jolla, CA 92093-0319 USA; [2]Argonne National Labs, Materials Science Division, Argonne, IL 60439 USA

Metallic superlattices exhibit interesting elastic anomalies which are correlated with structural changes. Many of these findings are controversial and often contradictory. These include anomalies in the shear, biaxial, flexural and Young's modulii of a variety of metal/metal, metal/semiconductor and ceramic/ceramic superlattices. We will describe a series of experiments relating mechanical anomalies with structural and electronic properties. These will then be discussed in light of theoretical models which have been proposed to explain the anomalous elastic properties.

9:10 AM INVITED PAPER
FATIGUE OF COPPER/POLYIMIDE LAMINATE, COPPER/ADHESIVE/POLYIMIDE MULTI-LAYER CONSTRUCTION AND COPPER-BASED FLEXIBLE PRINTED CIRCUIT: *Harish D. Merchant*[1]; Sidney J. Clouser[1]; Duane B. Mahnke[2]; [1]Gould Electronics, 34929 Curtis Blvd., Eastlake, OH 44095 USA; [2]Rogers Corporation, 100 N. Dobson Rd., Chandler, AZ 85224-6196 USA

Copper is bonded to the dielectric film by plasma treatment of the film surface prior to copper deposition; alternately it is bonded through an intermediate adhesive layer between the polyimide substrate and the thin copper foil. The copper is present as a continuous layer or as an etched line pattern of a given linewidth and pitch. In the flexible circuit, the copper lines are symmetrically sandwiched between the polymeric adhesive and dielectric layers. During cyclic loading, the load transfer and the strain accommodation between layers and the residual stress fields within copper dramatically alter the fatigue behavior of copper which either "rides" on the polymeric substrate or is effectively shielded from fatigue damage. The net effect is an orders of magnitude enhancement of fatigue life. The strain-based fatigue is characterized in the flex and fold modes for a wide range of strain amplitudes. Alternately, for a given strain amplitude, the flexible circuit is characterized in a roll fatigue mode whereby one side of the copper surface is subjected to tension/tension and the other side to compression/compression type loadings. If the inter-layer delamination and the variations in layer thickness are controlled, the mean fatigue life as long as 100×10^6 cycles to failure and a statistical description of fatigue are possible. The tension/compression fold fatigue mode mimics the printer hinge motion and the tension/tension plus compression/compression roll fatigue mold mimics the disk drive motion.

9:40 AM INVITED PAPER
INFLUENCE OF MICROSTRUCTURE SIZE ON THE PLASTIC DEFORMATION KINETICS, FATIGUE CRACK GROWTH RATE AND LOW-CYCLE FATIGUE OF SOLDER JOINTS: *Hans Conrad*[1]; Z. Guo[1]; Y. Fahmy[1]; Di Yang[1]; [1]North Carolina State University, Dept. of Mats. Sci. and Eng., Advanced Materials & Electro-Effects Laboratory, Raleigh, NC 27695-7907 USA

This paper reviews the work by the present authors and that in the literature on the effects of microstructure size on the plastic deformation kinetics (PDK), fatigue crack growth rate (FCGR) and low-cycle fatigue (LCF) of near eutectic Pb-Sn solder joints. Variation in microstructure size were obtained by: (a) varying the cooling rate following reflow, (b) isothermal annealing and (c) thermo-mechanical cycling (TMC). The principal microstructure features considered are the mean colony size dc and the average eutectic phase size d(Pb-Sn) = (d(Pb + dSn))/2. A decrease in the as-reflowed microstructure size had the following effects at shear stresses $t(tou)/m(mu) < 2 \times 10^{(-3)}$: (a) decreased the flow stress, (b) decreased the FCGR and (c) increased the fatigue life. Opposite behavior tended to occur for $t(tou)/m(mu) > 2 \times 10^{(3)}$. The effects of phase coarsening by isothermal annealing or TMC wore more complex, but in general tended to be similar to those for the as-reflo wed microstructure size. Constitutive equations are prepared which predict behavior in reasonable agreement with experiment.

10:10 AM BREAK

10:20 AM INVITED PAPER
MECHANICAL SPECTROSCOPY IN THE STUDY OF ANELASTIC AND STRUCTURAL BEHAVIOUR OF NANOSTRUCTURED METALS: *Ennio Bonetti*[1]; [1]Dipartimento di Fisica dell'Universita di Bologna, Gruppo Struttura Della Materia, Viale Berti Pichat 6/2, Bologna I-40127 Italy

Nanostructured (n-) metals due to their unusual specific physical properties have been the subject of a lot of theoretical and experimental investigations in recent years. A well used approach to study this class of materials refers to a simple two-components structural model consisting of the crystallites with dimensions in the nanometer range and the interfaces or interfacial phase. The high interface (surface) to volume ratio and the detailed structure of the interfaces are responsible for some of the interesting and specific properties of these materials. The Mechanical Spectroscopy approach to study the mechanical properties in the anelastic and or viscoelastic regime of n-metals demonstrated to be a powerful structure sensitive probe up to now not yet extensively employed, detailed information on some specific parameters determining the mechanical behaviour and the structural stability of n-materials can be obtained through measurements of the real and imaginary components of the complex mechanical susceptibility. These include the short range interfaces dynamics, whether it is of relaxational or viscoelastic-nature as well as the occurrence of structural relaxation and or

phase transitions affecting the interfaces without appreciable grain growth. These last effects are driven by microstrain localization at the interfaces occurring when entering the nanometer regime. Some examples and recent results dealing with mechanical spectroscopy measurements on nanostructured Al and Ni will be presented. The anelastic behaviour of these materials will be discussed with particular reference to structural stability against grain growth relaxation mechanisms at the interfaces and mechanical behaviour at high homologous temperatures.

10:50 AM INVITED PAPER

CYCLIC DEFORMATION BEHAVIOUR OF ULTRAFINE GRAIN COPPER: *Sean R. Agnew*[1]; A. Yu Vinogradov[2]; J. R. Weertman[1]; R. Z. Valiev[3]; [1]Northwestern University, Evanston, IL 60208 USA; [2]Kanazawa University, Kanazawa 920 Japan; [3]Ufa State Aviation University, Ufa Russia

The metals processed by severe plastic deformation techniques have come to be known as ultrafine grain (UFG) or submicrocrystalline metals and have received much interest recently. One area of study that has produced contradictory results is fatigue performance, namely whether or not UFG Cu undergoes fatigue softening. Recently, through careful TEM observation, continued fatigue testing, and differential scanning calorimetry new answers have been obtained to explain previous contradictions. We can now present a clearer picture of UFG Cu's fatigue performance. A summary of the investigation including Coffin-Manson and S-N plots, static recrystallization kinetics, and failure behavior will be presented.

NANOSTRUCTURED HYBRID MATERIALS: Synthesis and Processing

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Physical Metallurgy Committee

Program Organizers: Gan-Moog Chow, National University of Singapore, Dept. of Mats. Sci., Kent Ridge, Singapore 117600 Yeukuang Hwu, Institute of Physics, Academia Sinica, Nankang, Taipei Taiwan; Sara Majetich, Carnegie Mellon University, Dept. of Phy., Pittsburgh, PA 15213 USA; Luz Martinez-Miranda, University of Maryland, Dept. of Mats.& Nuclear Eng., College Park, MD 20742-2115 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Wednesday AM Room: 16A
March 3, 1999 Location: Convention Center

Session Chair: Robert D. Shull, NIST, Dept. of Magnetic Mats., Gaithersburg, MD 20899 USA

8:30 AM INVITED PAPER

NANOCHANNEL GLASS ARRAYS: *Ronald Joseph Tonucci*[1]; [1]Naval Research Laboratory, Code 5611, 4555 Overlook Ave., Washington, D.C. 20375 USA

Nanochannel glass arrays are complex glass composites with features as small as 10 nanometers. The interface between composite elements must be tightly controlled to avoid diffusion at these small lateral dimensions. By carefully controlling process conditions, 2-dimensional arrays have been fabricated with packing densities as high as 100 billion elements per square centimeter. A number of glass compositions have been used to fabricate the arrays and the composite can be engineered to contain etchable glass materials. Upon removal of the etchable glass, an array of highly regular channels can be formed. The arrays are thermally stable to temperatures in excess of 600ϒC and under certain conditions can be modified to be thermally stable to temperatures as high as 1000ϒC. A variety of materials have been deposited into the channels of the array to create high aspect ratio nanowires. Materials include metals, polymers and semiconductors with diameters as small as 33 nm. The fabrication and characterizat ion of these nanostructured hybrid materials will be discussed along with several applications.

9:00 AM

SELF-ASSEMBLED MAGNETIC NANOPARTICLE ARRAYS: *Sara A. Majetich*[1]; Yan Jin[1]; Cindi L. Dennis[1]; [1]Carnegie Mellon University, Dept. of Physics, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Ordered arrays of magnetic nanoparticles are prepared by two methods: the synthesis of a nonmagnetic liquid crystals which are solidified and transformed to a ferrimagnetic iron oxide phase, and the self-assembly of monodisperse ferromagnetic cobalt particles with uniform nonmagnetic coatings. Compared to other self-assembling systems, magnetic nanoparticles have an additional magnetostatic force, which can be either attractive or repulsive. This favors the formation of magnetically aligned chains of magnetic dipoles, rather than two- or three-dimensional structures. Both of the synthetic routes overcome this problem by making the magnetic forces weak relative to other interactions during the self-assembly process. Transmission electron microscopy (TEM) is used to characterize the degree of ordering of the nanoparticle arrays. The magnetization directions of individual particles and direct evidence of interparticle coupling are obtained from the Foucault method of Lorentz microscopy. Particle interactions ar e also characterized by magnetic force microscopy (MFM), showing the transition between chain-forming and layer-forming behavior.

9:20 AM BREAK

9:50 AM INVITED PAPER

MECHANICAL PROPERTIES OF THERMAL SPRAY NANO HYBRID COATINGS: *Peter Strutt*[1]; [1]Inframat Corporation, 20 Washington Ave., Suite 106, North Haven, CT 06473 USA

A methodology has been developed to deposit nanostructured coatings of hard ceramic materials by DC plasma thermal spraying. One of these nanocoating materials is alumina-13 wt% titania, which is deposited on steel substrates using a Metco 9MB plasma-arc gun. In examining sliding wear behavior, comparative studies have been made with coatings produced using conventional Metco 130 alumina-13 wt% titania powder feed material. The studies show that the properties of deposited coatings depend on the morphology and chemical composition of the agglomerated nanoparticle feed powder. When the thermal spray fed powder consists of hollow shell agglomerated nanoparticles, the wear resistance of the coatings is only slightly better (40%) than that of coatings produced with conventional Metco 130 powder. However, using solid nanoparticle agglomerated Al2O3/TiO2 powder there is a 200% wear resistance improvement over the conventional counterpart. Interestingly, the addition of nanostructured yttria-stabilized zirconia (n-Y SZ) to the powder feed is significant, for example, a 5 wt % addition of n-YSZ results in a fourfold reduction in weight loss during sliding wear.

10:20 AM

NONEQUILIBRIUM ALLOYS AND COMPOSITES IN NANOSTRUCTURED TWO-PHASE ALLOYS: *Evan Ma*[1]; J. H. He[1]; H. W. Sheng[1]; P. J. Schilling[2]; [1]The Johns Hopkins University, Dept. of Mats. Sci. and Eng., Baltimore, MD 21218 USA; [2]Louisiana State University, Center for Advanced Microstructures and Devices, Baton Rouge, LA 70803 USA

Using the Cu-Fe system as a model system, we discuss the formation of highly nonequilibrium alloys and composites in nanophase powder mixtures prepared by mechanical milling. Even though the system exhibits positive heat of mixing and negligible solid solubility at ambient temperature in equilibrium, atomic-level alloying between Cu and Fe occurs in nanophase mixtures of Cu and Fe produced by milling, forming either a single phase supersaturated solid solution, or a two-phase microstructure comprising both fcc and bcc solution phases. Extended X-ray Absorption Fine Structure (EXAFS) and X-ray Absorption Near-Edge Structure (XANES) techniques have been used to monitor changes in local environments and determine the compositions and volume fractions of the coexisting phases. Molecular Dynamics (MD) simulations have been used to elucidate atom-level structural evolution at the interfaces between the two nanostructured constituent elements with and without the presence of plastic deformation. Based on the findings from

experiments and simulations, the alloy formation and two-phase coexistence are explained in terms of the dynamics, energetics and heterogeneity of the nonequilibrium defects and external forcing events. The alloyed nanocrystalline solid solution powders have been used as precursors to obtain uniform two-phase nanocomposites through in situ phase decomposition during hot consolidation into bulk samples. The mechanical properties of the fully dense two-phase nanocomposites obtained are explained in terms of, in addition to the effects of phase volume fractions and grain sizes, the topological arrangements of the two-phase microstructure and effectiveness of the interphase interfaces as slip transmission barriers.

10:40 AM

PROCESSING OF Ni-SiO2 NANOCOMPOSITES USING SEVERE PLASTIC TORSIONAL STRAINING: *Y. T. Zhu*[1]; I. V. Alexandrov[2]; A. V. Korznikov[2]; T. C. Lowe[1]; R. Z. Valiev[2]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Division, MG G755, Los Alamos, NM 97545 USA; [2]Ufa State Aviation Technical University, Institute of Physics of Advanced Materials, K. Marksa 12, Ufa 450000 Russia

Severe Plastic Torsional Straining (SPTS) is a technique to induce large plastic shear deformation under high stress. It has been widely used to refine the grain size of monolithic metals and alloys, as well as to consolidate both micrometer-sized and nanometer-sized powders. This presentation will report the processing of Ni-SiO2 nanocomposites by SPTS consolidation of Ni-SiO2 powder mixtures. It is shown that the SPTS process can simultaneously consolidate the Ni-SiO2 powders and refine the grain size. The SPTS-consolidated nanocomposites is >98% dense. Microstructures, mechanical properties and thermal stability of the SPTS consolidated nanocomposites will be discussed.

11:00 AM

NANOCOMPOSITE MATERIALS PREPARED BY AEROSOL SYNTHESIS USING QUANTUM DOTS AS BUILDING BLOCKS: *J. M. Nedeljkovic*[1]; D. P. Uskokovic[1]; [1]Vinca Institute of Nuclear Science, Belgrade & Institute of Technical Sciences of the Serbian Academy of Sciences and Arts, Belgrade Yugoslavia

Spherical non-agglomerated nanocomposite particles and coatings of complex composition and controlled phase content, suitable for direct application or fabrication of high tech materials can be prepared by aerosol synthesis using ultrasonic spray pyrolysis of quantum dots as building blocks. Microscopic techniques with different level of resolution, such as scanning electron microscopy, high resolution field emission scanning electron microscopy, atomic force microscopy and scanning tunnelling microscopy were used. The methodology provides general procedure for the rational design of novel and potentially useful nanocomposite materials.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Hard Coatings

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

Wednesday AM Room: 7B
March 3, 1999 Location: Convention Center

Session Chairs: Douglas H. Lowndes, Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN 37831-6056 USA; Sun Kyu Kim, University of Ulsan, School of Mats. and Met. Eng., Mugeodong, Ulsan 680-749 Korea

8:30 AM INVITED PAPER

SURFACE DESIGN TOWARD WEAR-RESISTANT, SELF-LUBRICATING COATING: *Kazuhisa Myoshi*[1]; [1]National Aeronautics and Space Administration, Lewis Research Center, Cleveland, OH 44135 USA

High tribological reliability is of crucial importance in operating the many interacting surfaces that are in relative motion in mechanical systems. Improving the tribological functionality of materials such as achieving low friction and good wear-resistance is an aim of this investigation. According to the adhesion theory of metallic friction (mu = sA/W) and the relation between the coefficient of friction and the total surface energy in the real area of contact (mu ~ gamma.A), reducing friction requires minimizing the shear strength of the interface s, the real area of contact A, the total surface energy in the real area of contact gamma.A, and the plowing or cutting contribution (where W is the normal load). Reducing wear generally requires minimizing these factors while maximizing the hardness, strength, and toughness of bulk materials. Toward this end, surface engineering studies were used to describe several tribological coatings, including carbon-based and boron nitride coatings. Surface and bulk material properties, which determine the adhesion, friction, and wear behavior of interacting materials, are described. The primary emphasis is on the nature and character of these coatings, especially their surface chemistry, atomic bonding state, and microstructure. In addition, the reduction of friction and wear by thin oxide, carbon-based, and boron-based films that are formed on metal and ceramic surfaces is stated.

8:55 AM INVITED PAPER

CATHODIC-ARC CARBON FILMS AS PROTECTIVE OVERCOATS FOR DISK DRIVE APPLICATIONS: *W. Fong*[1]; D. Bogy[1]; C. S. Bhatia[2]; [1]University of California, Dept. of Mechanical Engineering, Berkeley, CA 94720 USA; [2]IBM SSD, 5600 Cottle Rd., San Jose, CA 95193 USA

Hydrogenated (CHx) and nitrogenated (CNx) carbon films ranging in thicknesses 7 nm and up are commonly used as protective overcoats on media found in disk drives today. However, as a real densities continue to increase, thinner overcoats (< 7 nm) will be needed to reduce the magnetic spacing between the head and disk. At these thicknesses, film properties such as surface coverage, corrosion protection, and wear durability affect the overall tribological performance of the head-disk interface. Cathodic-arc carbon films have been investigated in this paper as an alternative to CHx and CNx overcoats and results from nano-indentation tests, corrosion tests, and a series of tribological tests are summarized.

9:20 AM

CHARACTERIZATION OF TiN/CN_X MULTILAYERS DEPOSITED BY DC MAGNETRON SPUTTERING: *Y. H. Chen*[1]; [1]Northwestern University, Dept. of Materials Science and Engineering, Evanston, IL 60208 USA

Titanium nitride - carbon nitride multilayers (TiN/CNx) with TiN (111) crystalline orientation have been studied in order to obtain hard coatings with good tribological properties. Thin CNx layers were deposited to interrupt the growth of TiN in order to suppress the TiN columnar structure. The films were deposited by reactive DC magnetron sputtering on Si (100) substrates at room temperature. Deposition pressure as low as 2.5 mTorr in an argon-25% nitrogen mixture were determined to be the best deposition conditions for obtaining TiN (111) texture. Bias voltage was applied on the substrate to enhance ion bombardment of the growing film. The thickness ratio of TiN/CNx layers varies from 1 to 10 keeping CNx thickness equal to 0.75 nm. Low-angle x-ray diffraction showed good interface between layers. The internal stress measured by the substrate curvature is directly related to the layer thickness ratio, i.e., smaller thickness ratio gives smaller internal stress. The surface roughness relates to the thickness ratio in the same way. Multilayers show better tribological performance than monolithic TiN thin films.

9:35 AM

13C NMR SPECTROSCOPY OF AMORPHOUS HYDROGENATED BORON CARBIDE AND AMORPHOUS HYDROGENATED CARBON NITRIDE: *Janet Braddock-Wilking*[1]; Joseph LaManna[1]; Shu-Han Lin[1]; Bernard J. Feldman[1]; [1]University of Missouri-St. Louis, Departments of Chemistry and Physics, Center for Molecular Electronics, St. Louis, MO 63121 USA

We report the 13C NMR spectrum of amorphous hydrogenated boron carbide and amorphous hydrogenated carbon nitride. Both alloys are grown in an rf CVD plasma reactor. The amorphous hydrogenated boron carbide spectrum is dominated by one line at 15 ppm. We interpret this line as due to carbon bound in boron carbide icosahedra, because polycrystalline boron carbide with boron carbide icosahedra as the unit cell gives a very similar NMR spectrum. The amorphous hydrogenated carbon nitride spectrum consists of two broad lines centered at 20 ppm and 130 ppm and two sharp lines at 131 ppm and 164 ppm. The broad line at 130 ppm and sharp line at 131 ppm are due to sp2 carbon in an amorphous matrix and crystalline environment, respectively. The broad line at 20 ppm is possibly due to sp2 carbon bound to a nitrogen atom in an amorphous matrix. The sharp line at 164 ppm is not understood, but is probably related to the sharp 164 ppm line previously observed in the NMR spectrum of amorphous carbon nitride grown by magnetron sputtering.

9:50 AM

STUDY OF CN_X FILMS DEPOSITED BY ICP-CVD FROM N_2/CCl_4/H_2 PRECURSORS: Marie-Paule Delplancke-Ogletree[1]; *Jiri Bulir*[1]; [1]Université Libre de Bruxelles, Metallurgy - Electrochemistry, CP 165, 50 Avenue F.D. Roosevelt, Brussels 1050 Belgium

Thin CN_x films were deposited on silicon substrate by inductively coupled r.f. plasma chemical vapor deposition (ICP-CVD). Gas mixture of N_2 /CCl_4 /H_2 was used for the deposition. Influence of deposition parameters on film quality and plasma properties was studied. Nature of gaseous species, electron temperature an plasma density were obtained by optical emission spectroscopy and Langmuir probe measurements. Pressure, ratio of H_2 to CCl_4, substrate temperature and injected r.f. power were varied in the following ranges: p=10-200 Pa, H_2 /CCl_4=1-10, T_s =100-400 C, and P=50-300W. Film composition was studied by AES before and after in-situ Ar ion sputtering. The N/C ratio varied in the range of 0.5-0.7. Chlorine contamination of the deposits was detected but could be reduced by optimizing the deposition conditions. Decrease of both, Nitrogen and Chlorine concentrations was observed after the surface was sputter cleaned. Fourier transform infrared spectroscopy (FTIR) was used for determination of chemical bonding. Presence of Hydrogen was confirmed by N-H stretching vibrational band at 3350 cm^{-1} and weak C-H stretching band. A group assigned to C=C and C=N was detected around 1650 cm^{-1} as well as a nitril CN group at 2220 cm^{-1}. Surface morphology of the films was studied by atomic force microscopy (AFM).

10:05 AM

INVESTIGATION OF MECHANICAL PROPERTIES OF Ti(C,N) AND TiN THIN FILMS DEPOSITED ON CUTTING TOOLS: *Erich Lugscheider*[1]; Cyrus Barimani[1]; Markus Lake[1]; [1]Aachen University of Technology, Materials Science Institute, Aachen Germany

Increasing demands on production processes in terms of performance, reliability and environmental compatibility shape the specification profile for modern wear resistant coating systems. The cutting process is determined by a complex kind of straining. Due to this fact, the cutting tools should possess complement properties, like hardness and toughness. These complement requirements are fulfilled by PVD coated cutting tools. Especially specific cutting operations like high speed cutting (HSC) or cutting without coolants necessitate PVD coated cutting tools. The performance and tool lifetime of PVD coated cutting tools is mainly influenced by the mechanical properties like hardness, residual stress or bond strength of coating and substrate. For the present work three different substrate materials, high speed steel, cemented carbides and cermets, were coated with Ti(C,N) and TiN. The deposition processes were carried out with the Cathodic Arc Ion Plating Process by varying the layer thickness. Depending on different layer thicknesses, the mechanical properties like hardness, residual stress or bond strength were investigated. To determine the hardness a nanoindenter was used. The bond strength was investigated by the scratch test and a hardness test called Rockwell C test. To investigate the residual stress a stripe bending test was used. This paper describes the experimental techniques and the results of the mechanical film properties in consideration of different film thicknesses.

10:20 AM INVITED PAPER

OVERVIEW-HIGH DEPOSITION RATE CERAMIC-METALLIC COATINGS BY ELECTRON BEAM-PHYSICAL VAPOR DEPOSITION (EB-PVD) PROCESS: *Doug Wolf*[1]; J. Singh[1]; [1]The Pennsylvania State University, Applied Research Laboratory, State College, PA 16804 USA

Chromium, titanium carbide (TiC) and partially yttria stabilized zirconia (YSZ) coatings deposited by energy electron beam-physical vapor deposition (EB-PVD) will be presented along with their potential applications. Interest in replacing chromium electroplating process has sparked the use of EB-PVD technology for the repair of many components including landing gears due to relatively high deposition rate (100-150 m/minute with an evaporation rate ~ 10-15 Kg/hour), dense coatings, precise composition control, columnar and poly-crystalline microstructure, ad low contamination. This paper discusses the microstructure, wear behavior and friction coefficients of chromium coatings produced by EB-PVD and will be compared with thermal sprayed WC-Co coatings. In addition, the surface morphology, microstructure and texturing of TiC films produced by reactive ion beam assisted EB-PVD will be discussed for wear resistant applications including cutting tool industry. YSZ has also been grown by EB-PVD resulting in a columnar microstructure for thermal barrier applications (TBC). Microstructural evolution of various ceramic and metallic coatings developed by the EB-PVD process will be presented.

10:45 AM BREAK

11:00 AM INVITED

TRIBOCHEMICAL POLISHING OF SILICON NITRIDE AND CARBIDE: *T. E. Fischer*[1]; V. Muratov[1]; Z. Zhu[1]; [1]Stevens Institute of Technology, Hoboken NJ 07030

Tribochemical polishing is the removal of material by a chemical reaction that is stimulated by friction. Scratch-free surfaces with a roughness R_a = 0.5 nm are routinely obtained on silicon nitride. The chemical and mechanical anisotropy of silicon carbide decrease the polishing ability, resulting in surfaces with a roughness R_a = 1.2 nm. The mechanisms by which friction accelerates the chemical reaction have been studied by measuring the reaction rate as a function of temperature, reagent concentration, normal force and sliding speed. In all cases, it is found that the reaction rate is proportional to the friction coefficient above a threshold that depends on the reactive fluid. The specific temperature-dependence is small, showing that the activation energy for the reaction is reduced from 120 to less than 10 kJ/mole by friction. Two tribochemical mechanisms have been identified: the growth of a

surface layer that is removed by friction operates at low sliding speeds only and a direct stimulation of the reaction is observed in all other conditions.

11:25 AM INVITED
CURRENT ISSUES IN TOTAL HIP ARTHROPLASTY: THE HEAD-STEM INTERFACE: *Ramakrishna Venugopalan*[1]; Linda C. Lucas[1]; [1]Department of Biomedical Engineering, Univeristy of Alabama at Birmingham, 1075 13th Street South, Birmingham, AL 35294-4440

Modular connections commonly exist at the head-stem region in total hip arthroplasties (THAs) and allow for adaptation of the metaphyseal-diaphseal ômis-matchö of the anatomy. Modularity takes advantage of the material properties of the different combinations of materials specifically suited for the functional needs of the various implant components and is thus a very cost-effective solution to the difficult demands of THA. However, modularity also places increased demands on manufacturer and surgeon alike in terms of design, manufacture, and assembly issues. This talk will address specific concerns regarding electrochemical-mechanical interactions in mixed-metal modular head-stem connections in THAs. The effect of a nitrogen diffusion-hardening process on the titanium alloy component with reference to improving the resistance of the head-stem interface to electrochemical-mechanical interactions will also be presented and analyzed.

11:50 AM
APPLICATION OF ELECTRON SPECTROSCOPY FOR THE ENHANCEMENT OF METASTABLE CERAMIC FILMS: *R. Cremer*[1]; M. Witthaunt[1]; D. Neuschutz[1]; [1]Rheinishch-Westfalische Technische Hochschule Aachen, Lehrstuhl fur Theoretische, Aachen D-52056 Germany

Metastable ceramic coatings are gaining increased attention in industrial applications. One of the most familiar is the wear resistant (Ti,Al)N coatings which is extensively used for the protection of cutting tools. Another example of ternary metastable films with superior performance in comparison to the stable constituent binaries is Al-O-N. The high chemical and thermal stability of Al-O-N makes this film an attractive candidate for diffusion barriers in gas turbines. Due to the non-equilibrium conditions of physical vapor deposition, this technique is exceptionally suitable for the deposition of metastable films. The presented paper will focus on the particularities of physical vapor deposition and characterization of metastable ternary coatings. As many metastable solution phases do not form crystalline phases, special attention will be paid to the analysis of the binding states of the components by Auger-and Photoelectron spectroscopy. It will be shown that the analysis of binding states and electronic structure is a powerful tool for the determination of the properties of metastable solution phases and their enhancement for technical application.

12:05 PM
DEVELOPMENT OF HARDFACING CONSUMABLE MATERIAL FROM IRON CARBIDE: *Brajendra Mishra*[1]; David L. Olson[1]; David Fazzina[1]; [1]Colorado School of Mines, Dept. of Metallurgical and Materials Engineering, Center for Welding, Joining, and Coatings Research, Golden, CO 80401 USA

A novel hardfacing consumable material has been development using commercial grade iron carbide and ferro-titanium. The process of forming titanium carbide particles in a martensic matrix from these reactants, passing through the welding arc into the weld deposit, is inexpensive. The product phases provided high hardness and superior wear properties. With optimal composition and processing conditions, weld deposits have been produced which show notable levels of wear resistance. The flux coated arc welding with shielding gas was used for this purpose. Penetration in weld-deposits was achieved by enhancing the exothermically assisted reactions leading to the proper formation of weld beads for hardfacing. The weld bead size and morphology was improved by switching from an argon-carbon dioxide gas to a nitrogen shielding gas. The optimum microstructure consisted of carbides which are imbedded in a martensite matrix with some retained austenite. Key considerations in further optimization of the weld microstructure included reducing the free silicon by the addition of molybdenum and the addition of aluminum to reduce the amount of oxygen. It has been demonstrated that excess oxygen acts to reduce the amount of carbon and titanium available to react and form titanium carbide.

12:20 PM
STRESSES GENERATED ON ALUMINUM AS THE FUNCTION OF APPLIES POTENTIAL AND pH IN PRESENCE OF CHLORIDE: *S. E. Benjamin*[1]; F. A. Khalid[2]; [1]Colorado School of Mines, Dept. of Metallurgy and Materials Engineering, Golden, CO 80401 USA; [2]Gulam Ishaq Khan Institute of Engineering Science & Technology, Faculty of Metallurgy and Materials Engineering, Topi Pakistan

The resistance of the oxide film on aluminum is imperiled by the stresses generated during growth of the oxide. The present studies focus upon the stresses generated on aluminum as the function of the applied potential in sulfuric acid solutions of different strengths containing chloride ions. The resulting current depicts the formation/dissolution of the oxide which is controlled both by the pH and the chloride anion. The magnitude and direction of stresses generated during the anodic oxidation process are measured by the beam deflection technique. The results have been interpreted in terms of the formation and annihilation of the anion (O2-) and the cation (Al3+) vacancies. Reduction in the aluminum ion vacancies (VAl3+) results in the compressive stress deflection whereas, tensile stress deflection is introduced due to an increase in the oxygen ion vacancies (VO2-).

SYNTHESIS OF LIGHTWEIGHT METALS III: Titanium - III

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. Centre, Farnborough, Hampshire GUI14OLX UK; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Neqev Israel; P. G. McCormick, University of W. Australia, Res. Centre for Adv. Min. & Mat. Proc., Nedlands, W.A. 6907 Australia

Wednesday AM Room: 10
March 3, 1999 Location: Convention Center

Session Chairs: J. Ma, Beijing Institute of Aeronautical Materials (BIAM), Beijing 100095 China; P. G. Partridge, DERA, Struct. Mats. Centre, Farnbourough, Hans GU14 6TD UK

8:30 AM INVITED PAPER
OVERVIEW OF TITANIUM GOLF CLUBS - PART I: *Jimin Ma*[1]; Chenggong Li[1]; F.H. (Sam) Froes[3]; [1]Beijing Institute of Aeroautical Materials (BIAM), P.O. Box 81, Beijing 100095 China; [3]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Titanium is ideally suited for many golf club applications including woods and putters. The case for titanium irons is less clear. This paper will discusss the development of new titanium concepts for irons including tungsten weighting to lower the center of gravity of the head.

8:50 AM INVITED PAPER
OVERVIEW OF TITANIUM GOLF CLUBS - PART II: *Jimin Ma*[1]; Chenggong Li[1]; F.H. (Sam) Froes[3]; [1]Beijing Institute of Aeroautical Materials (BIAM), P.O. Box 81, Beijing 100095 China; [3]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Titanium is ideally suited for many golf club applications including woods and putters. The case for titanium irons is less clear. This paper will discusss the development of new titanium concepts for irons including tungsten weighting to lower the center of gravity of the head.

9:30 AM

CONSOLIDATION, MICROSTRUCTURE AND PROPERTIES OF TAPE-CAST Ti / SiC FIBRE COMPOSITES: *Z. X. Guo*[1]; C.M. Lobley[1]; Q. Kang[1]; [1]University of London, Dept. of Mats., Queen Mary and Westfield College, Mile End Rd., London E1 4NS UK

Light weight composite based continuous SiC fibres and titanium matrices offer very desirable combinations of high-temperature strength, stiffness, and creep resistance, which are ideal for various components in the future generation of gas and steam turbine engines. However, large-volume applications are currently hampered by the relatively high cost of the material. A great percentage of the cost is due to its manufacturing. A low-cost tape-casting processing technique has been under study at our laboratory. The process uses relatively large inexpensive matrix alloy powders mixed with an organic binder to form a slurry, which is then cast to form a thin uniform powder tape. Once dried, the tape is laid-up with filament-wound SiC fibres and consolidated in a vacuum hot press, with a burnout dwell at moderate temperatures to ensure removal of the organic components. This paper concentrates on the study of the consolidation process of tape-cast powder/fibre preforms. The effects of temperature, pressure and time on consolidation kinetics were evaluated. Microstructural examination of both partially and fully consolidated composites was carried out by means of optical, SEM and TEM analyses to identify matrix flow behavior, microstructural evolution, and integrity of the composites. Composites of very uniform fibre distribution with no fibre damage have been achieved. The as-processed composites were extensively tested under tension and four-point bending. The results show that the average strength of the composites reaches at least 92% of the law-of-mixtures value. This is comparable to values obtained on similar composites manufactured by alternative processes. However, a considerable cost saving and a high degree of product flexibility are realised from the tape-casting route.

9:50 AM

SYNTHESIS AND CHARACTERIZATION OF TITANIUM-BASED INTERMETALLIC / METALLIC LAMINATES: *C. B. Loader*[1]; S. J. Howard[1]; C. Malcolm Ward-Close[1]; [1]DERA Farnborough, Structural Mats. Centre, Room 2008, A7 Bldg., Farnborough, Hampshire GU14 OLX UK

Titanium aluminides are a potential replacement for the current aerospace alloys, at present however their low room temperature toughness and ductility limits their application. Introducing a ductile phase into the intermetallic matrix has been shown to increase the effective toughness of the composite material. Three methods were selected for the fabrication of titanium/titanium aluminide laminates; diffusion bonding, electron beam physical vapour deposition and vacuum plasma spraying. The resultant laminated composite materials were expected to process both high temperature creep resistance and low temperature ductility. Microstructural evaluation of the laminates was performed and mechanical property data was generated. Results show the strength and toughness variation obtained in multi-layered materials compared to monolithic materials.

10:10 AM BREAK

10:25 AM

IMPACT TOUGHNESS OF LAMINATES COMPOSED OF LAYERS OF TITANIUM-ALLOY AND TITANIUM-ALLOY MMC: *Y. Q. Zuo*[1]; D. Smith[1]; P. G. Partridge[2]; A. Wisbey[2]; [1]University of Bristol, Dept. of Mech. Eng., Queens Bldg., University Walk, Bristol, Avon BS8 1TR UK; [2]DERA, Farnborough, Structural Materials Centre, Farnbourough, Hants GU14 6TD UK

Instrumented impact toughness values in bend have been obtained for symmetrical 3-layer laminates composed of diffusion bonded IMI 834 Ti-alloy and Ti-6Al-4V alloy/continuous SiCr metal matrix composite (MMC) sheets. The MMC represented 50 volume % of the laminate and was present as either a single layer at the midplane (C-type laminate) or as two thinner layers at the surfaces of the laminate (S-type laminate). Control test pieces consisted of 100% IMI 834 and 100% MMC and notched and unnotched test pieces were used. The laminate microstructure was particularly effective in increasing the notched impact strength relative to both the IMI834 Ti-alloy and the MMC. The impact toughness could be predicted in terms of the work to initiate fracture and to propagate a crack in this system. The modulus value for the S-type laminate with only 50% MMC (192 GPa) was within about 4% of the value for the 100% MMC test piece. The laminate allows cost effective utilisation of the MMC and a lower density compared with monolithic Ti-alloy. It will be shown that the laminate has a unique overall combination of properties.

10:45 AM

HIGH-TEMPERATURE TRIBOLOGICAL BEHAVIOR OF Ti-V ALLOYS AND Ti-40Mo: *Xu Huang*[1]; Jimin Ma[1]; Chunxiao Cao[1]; Bao Wang[1]; Yang Gao[1]; Yaohe Zhou[2]; [1]Beijing Institute of Aeronautical Materials (BIAM), P.O. Box 81-15, Beijing 100095 China; [2]Northwestern Polytechnical University, Xi'an 710072 China

Generally combustion property of titanium alloy is related closely to its friction behavior. The variations of unlubricated sliding friction coefficients and wear rates vs temperature up to 900℃ for Ti-20V, Ti-40V and Ti-40Mo alloys were determined by pin-on-disc tribometer. The results show that the friction coefficients and wear rates decrease with increasing temperature, and decrease sharply at 700℃ for the Ti-V alloys and 800℃ for Ti-40Mo alloy, that the friction coefficient of Ti-40V is lower than that of Ti-20V. The morphology of friction surfaces and composition of oxide scales formed on the surfaces were examined by SEM-EDAX. The structures of transferred oxides from Ti-20V, Ti-40V were analyzed by XRD. It is suggested that the decrease of friction coefficients and wear rates of the Ti-V alloys and Ti-Mo alloy at elevated temperature is resulted from the lubrication due to the formation of soft, liquid V_2O_5 or MoO_3 oxide.

11:05 AM

THE PRODUCTION OF MAGNESIUM-TITANIUM ALLOYS BY MECHANICAL ALLOYING: *Simon B. Dodd*[1]; Susan Morris[1]; [1]DERA, Structural Materials Centre, Griffith Building (A7), DERA Farnborough, Hampshire GU14 0LX UK

In this paper the development of a mechanical alloying (MA) production technique for novel magnesium titanium binaries will be discussed. The aim of the work was to develop complete solid solution of titanium in a magnesium matrix for use as structural corrosion resistant lightweight alloys. The alloy range under investigation ranged from 0 to 20 wt% Ti. Previous work using physical vapour deposition (PVD) had demonstrated the potential of the alloy system for corrosion resistance and indicated that alloys under 20 wt% Ti provide a good balance between corrosion resistance and density. Traditionally the use of magnesium alloys, especially in aerospace applications, has been limited by the perceived poor corrosion resistance. The addition of titanium produces both a self-healing corrosion layer and reduced galvanic potential. The boiling point of magnesium is lower than the melting point of titanium and only novel processing routes such as MA and PVD can be successfully utilised to produce alloys of magnesium and titanium. This paper will describe the work carried out to develop an MA process route for solid solution Mg-Ti alloys. The influence of various process control agents on the synthesis of Mg-Ti solid solutions will be discussed. In addition, this paper will detail some of the material properties obtained from these novel materials.

11:25 AM

SYNTHESIS OF TITANIUM-VANADIUM MASTER ALLOYS BY MECHANOCHEMICAL PROCESSING: *E. G. Baburaj*[1]; Tri Widodo[1]; F .H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Titanium-vanadium master alloys are extensively used for the preparation of Ti-Al-V alloys. The present work is an attempt to produce the high melting Ti-V alloys in powder form by co-reduction of chlorides of Ti and V. Stoichiometric amounts of $TiCl_4$ and VCl_3 corresponding to the alloy composition $Ti_{50}V_{50}$ have been milled together with the reducing agents CaH_2 and Mg in a spex mill. The reaction product after leaching consisted of hydrides of Ti and V. It is not clear whether the hydrides have mutual solubility of one element in the hydride of the other. Further work on dehydrogenation and consolidation is in progress.

THE MARTIN E. GLICKSMAN SYMPOSIUM ON SOLIDIFICATION AND CRYSTAL GROWTH: Microgravity Processes

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 Mats. Sci. & Eng., Evanston, IL 60208 USA

Wednesday AM Room: 11A
March 3, 1999 Location: Convention Center

Session Chairs: Robert S. Sokolowski, IGC, 1875 Thomaston Ave., Waterberry, CT 06704 USA; Ed Winsa, NASA Lewis Research Center, Space Div., Cleveland, OH 44135 USA; Matthew Koss, R.P.I., Dept. of Mats. Sci. & Eng, Troy, NY 12180 USA

8:30 AM INTRODUCTION

8:45 AM
SOLIDIFICATION BEHAVIOR OF BINARY ORGANIC EUTECTICS AND MONOTECTICS; 1,2,4,5-TETRACHLOROBENZENE-M-AMINOPHENOL SYSTEM: *Uma Shanker Rai*[1]; [1]Banaras Hindu University, Chemistry Dept., Varanasi, UP India

With a view to study the solidification behaviour of binary organic eutectics and monotectics, phase-diagram, growth behaviour and microstructure of 1,2,4,5-tetrachlorobenzene and m-aminophenol system were studied.

9:15 AM
EVOLUTION OF SIDEBRANCH SPACINGS IN THREE-DIMENSIONAL DENDRITIC GROWTH: *Christoph Beckermann*[1]; Qiao Li[1]; [1]University of Iowa, Mechanical Engineering, 2412 EB, Iowa City, IA 52242 USA

The sidebranching behavior in three-dimensional dendritic growth is investigated through a detailed measurement of the arm spacings of succinonitrile (SCN) dendrites using images from the microgravity experiments of Glicksman and coworkers. The measurements show that the sidebranching evolution is divided into two regimes: an initial linear regime and a subsequent non-linear coarsening regime. A simple model, based on the Mullins-Sekerka linear stability theory, is developed to describe the initial sidebranching behavior and is found to be in excellent agreement with the experimental results. In the non-linear regime, the measured arm spacings are compared with previous coarsening theories. It is found that a careful distinction between arm spacings and curvatures is necessary to explain the measurements.

9:35 AM
EFFECT OF STABILIZING TEMPERATURE GRADIENTS ON THERMAL CONVECTION IN PHYSICAL VAPOR TRANSPORT OF HG2CL2: *J. W. Choi*[1]; M. H. Kwon[2]; S. K. Kwon[3]; G. T. Kim[1]; Martin E. Glicksman[4]; [1]Hannam University, Chemical Engineering Dept., 133 Ojung-Dong, Taejon 306-791 Korea; [2]SKC Company, R&D Center, Songgu, Chonan 330830 Korea; [3]Agency of Defense Development, Taejon 305600 Korea; [4]R. P. I., Materials Sci.& Eng. Dept., Troy, NY 12180-3590 USA

Mercurous chloride (Hg2Cl2) crystals hold promise for many acousto-optic and opto-electronic applications. This material is prepared in closed ampoules by the physical vapor transport (PVT) growth methods. Due to the temperature gradient between the source and the growing crystal region which is the driving force for mechanism of PVT, i.e., evaporation-condensation, the thermal buoyancy-driven convection may occur. The thermal boundary conditions established by imposing different temperature on sidewalls of the enclosure cause simultaneous horizontal and vertical convective flow in the PVT of Hg2Cl2. It is found that for the ratios of horizontal to vertical thermal Rayleigh numbers RaH /Ra = 1.5, the convective flow structure changes from multicellular to unicellular for the base parametric state of Ra = 2.79 x 104, Pr = 0.91, Le = 1.01, Pe = 4.60, Ar = 0.2 and Cv = 1.01. The vertical gradient tends to destabilize the flow that leads to oscillations. This paper will present the effect of stabilizing temperature gradients on thermal convection for various aspect ratios (widt h-to-transport length).

9:55 AM INVITED PAPER
A COMPARISON OF MORPHOLOGICAL INSTABILITY AND OTHER INTERFACIAL INSTABILITIES: *Ranga Narayanan*[1]; [1]University of Florida, Dept. of Chemical Engineering, Gainesville, FL 32611 USA

This is a review talk and will focus on the solidification instability of interface morphology. I shall compare this classical problem with another classical problem in fluid mechanics i.e., the Marangoni instability problem. There will be a discussion of interface morphology changes when convection occurs for solidification of pure substances and for solidification of a dilute binary alloy. I shall also relate some of the phenomena with the instabilites that occur during an electro-deposition process.

10:15 AM BREAK

10:35 AM INVITED PAPER
WHY WE WERE WRONG ABOUT THE EFFECT OF CONVECTION ON DENDRITIC GROWTH IN MICROGRAVITY: *Mathew Koss*[1]; [1]R. P. I., Materials Science and Engineering Dept., Troy, NY 12180 USA

The Isothermal Dendritic Growth Experiment (IDGE), a NASA sponsored series of Space Shuttle microgravity experiments, was designed and operated to grow and photograph dendrites, the ramified tree like morphology assumed by many liquid-solid solidification interfaces, in the absence of convective heat transfer. This data from this experiment forms a benchmark for fundamental tests of dendritic solidification theories. The first space flight of the IDGE in March of 1994 provided a rich data set from which we published many new results and conclusions. But, subsequent analysis, additional theoretical work, and the data from the second IDGE space flight experiment in March of 1996 showed that one of our first published conclusions was wrong. How and why we made, eventually recognized, and corrected this error is an interesting and important story for the scientific community in general, and NASA's microgravity science community in particular.

11:05 AM
THE INFLUENCE OF INTERFACE MORPHOLOGY ON HEAT TRANSPORT IN DENDRITIC SOLIDIFICATION: *Jeffrey C. LaCombe*[1]; *Matthew B. Koss*[1]; Douglas C. Corrigan[1]; Afina Lupulescu[1]; Laura Tennenhouse[1]; Martin E. Glicksman[1]; [1]Rensselaer Polytechnic Institute, Materials Science and Engineering, CII Building, Room 4219, Troy, NY 12180 USA

The method of moving heat sources is applied to the problem of dendritic growth in a quiescent, isothermal melt. By specifying various solid-liquid interface shapes in the model formulation, a clear understanding of the influence of the tip shape and the side-arm cruciform is obtained. The results of this study provide an explanation of several aspects of the microgravity-conducted Isothermal Dendritic Growth Experiment (IDGE). When corrections to Ivantsov's paraboloidal tip shape are incorporated into the transport solution to reflect the actual observed tip shape, enhanced agreement with the experimental data results. Additionally, these conclusions support earlier work by Schaefer (J. Cryst. Growth, vol. 43, p. 17) in suggesting that under the conditions of the IDGE experiment, the side-branch region of a dendrite can contribute significantly to the thermal field at the tip. Furthermore, the growth process is not sensitive to the details of the tip shape, provided the interface is largely paraboloidal. These suggest that the scatter in the IDGE data can be explained by stochastic variations in the side branch

structure, and the corresponding influence that this region has upon the transport processes at the tip. With these results, it is now reasonable to claim that the basic transport solution describing dendritic growth is correct, provided details such as container wall effects and dendrite self-interaction are accounted for. Until now, these conclusions have not been completely supported by quantitative evidence. Furthermore, these results suggest that the side branch region of a dendrite affects the transport portion of the dendritic growth problem as well as the scaling portion.

11:25 AM

UNDERCOOLING EFFECTS ON DROPLETS COARSENING IN THE Cu-Co SYSTEM: *Delin Li*[1]; Michael B Robinson[2]; Thomas J Rathz[3]; [1]NASA/MSFC NRC, Space Sciences Lab, Drop Facilities, ES75, Huntsville, AL 35812 USA; [2]NASA/MSFC, Space Sciences Lab, ES71, Huntsville , AL 35812 USA; [3]UAH, Materials Processing Lab, Huntsville, AL 35899 USA

The Cu-Co system is often chosen to study Ostwald ripening through aging treatment or liquid phase sintering, because of its low misfit strain and a similar mass density between Co and Cu. It has been shown that the growth of decomposed or sintered particles basically obeys the well-known LSW theory, though there are discrepancies about the coarsening rate and size distribution. Another attraction of the binary Cu-Co system is the potential thermodynamic tendency to immiscibility upon undercooling. This allows a study of phase separation process and droplet coarsening in a metallic liquid-liquid mixture where the amount of experimental data obtained to date is scarce. In this paper, liquid phase separation and droplet growth were investigated for a near-critical Cu50Co50 and an off-critical Cu68Co32 composition (atomic) by means of undercooling experiments. It was observed that liquid phase separation could occur at about 50 K below the miscibility gap, while the two resultant melts were further undercooled by larger than 300 K prior to solidification. The droplet growth behavior was found to exhibit three regimes: a power law growth, linear growth, and saturation stage. From microstructural examinations, it turned out that in addition to dendrites and droplet-like morphology reported previously, an interconnected structure was observed for the near-critical composition at intermediate undercooling. This interconnected structure played a crucial role in droplet coarsening at high undercooling.

11:45 AM

SINGLE ROLL CASTING OF ALUMINIUM ALLOY STRIP: *Daniel Liang*[1]; Wendy E. Borbidge[1]; Ross V. Allen[1]; [1]CSIRO, Materials Science and Technology, Private Bag 33, Clayton South MDC, Victoria 3169 Australia

Al-Si alloy strip has been produced by use of a single roll casting assembly with or without an additional deforming roll. The assembly has been configured to overcome defects such as porosity and surface ripples. The mechanisms for the defect formation were investigated as a function of operational parameters, strip thickness and deforming load. Internal microstructures were characterised for various solidification conditions. Results show that strips of 1 to 6mm thick with sound internal microstructures and satisfactory surface quality can be obtained by using the modified single roll casting assembly.

Institute of Metals Lecture and Robert F. Mehl Medalist
Time: 12:00 Noon
Room: 6E
Location: San Diego Convention Center

LMD Luncheon
Time: 12:00 Noon
Room: Marina Ballroom
Location: Marriott Hotel

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

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Thermodynamics & Phase Equilibria Committee

Program Organizers: Robert Schiffman, R.S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica, Bologna I-40126 Italy

Wednesday PM Room: 15B
March 3, 1999 Location: Convention Center

Session Chair: Mike Robinson, MSFC, MSFC, AL 35812

2:00 PM

INTRODUCTION TO CONTAINERLESS PROCESSING: *Michael Wargo*[1]; Shinichi Yoda[2]; Rainer Kuhl[3]; Philip Gregory[4]; [1]NASA Headquarters, Code UG, Washington, DC 20546 USA; [2]NASDA, Space Utilization Research Center, Sengen, 2-1-1, Tsukuba-shi, Ibaraki-ken 305 Japan; [3]DLR, Koenigswinterer Str. 522-524, Bonn, 53227 Germany; [4]Canadian Space Agency, 6767 Route de L'Aeroport, St-Hubert, Quebec J3Y 8Y9 Canada

Containerless processing is the most promising technology for enabling new types of material production through deep supercooling, taking highly precise thermophysical properties measurements, and making highly purified materials. The development of hardware related to this technology for International Space Station utilization has been supported independently by NASDA, DLR, and CSA, each providing facilities with different positioning technologies. Each positioning method has an advantage for some particular microgravity experiments. ISS is expected to be used collaboratively by all participating countries for the study of the microgravity sciences, and it is hoped that these containerless facilities will be able to completely cover all of the microgravity sciences requiring containerless processing with each method contributing best to a particular science study. The purpose of this workshop is as follows. Firstly, we will try to define the science envelope which could be covered by the containerless proce ssing methods in microgravity. Secondly, if there is a science area which is not covered by one of the existing containerless processing methods, we will try to determine a possible scenario by which it could be covered, which may involve modification of one of the existing containerless processing methods. Thirdly, we will provide recommendations to the participating agencies for any perceived short-comings or modifications of the containerless processing facilities in order for these facilities to cover the necessary studies contained within the defined science envelope. In general, the experimental facilities for ISS should be well matched for performing a particular science study in order to get fruitful experimental results necessary for the benefit of humankind.

2:20 PM

TEMPUS CONTAINERLESS PROCESSING FACILITY FOR SPACE STATION - PRELIMINARY DESIGN AND SPECIFICATION: *Joerg Piller*[1]; [1]Dornier GmbH, Daimler-Benz Aerospace, Friedrichshafen D-88039 Germany

The containerless processing facility TEMPUS was successfully used for electromagnetic levitation experiments during the MSL-1 Spacelab mission in 1997. Scientific goals have been the study of nucleation statistics and solidification speeds in undercooled melts, and the determination of thermophysical properties of the melt above and below the melting point. For future research in this promising field the accommodation of an Advanced TEMPUS facility on board of the International Space Station is under discussion. The results of a design study are presented to show that the well-proven technology of TEMPUS Spacelab can be transferred to a Space Station facility. A set of samples will be processed in experiment containers which will be exchanged together with coil system, windows, stimuli or other specific devices.

2:40 PM

ELECTROMAGNETIC LEVITATION IN MICROGRAVITY: EXPERIMENTS AND RESULTS OF THE TEMPUS TEAM: *Ivan Egry*[1]; [1]DLR, WB-RS, Institut fuer Raumsimulation, Cologne 551140 Germany

Electromagnetic levitation is a convenient tool for containerless processing of electrically conducting materials. In microgravity, only small stabilizing forces are needed; therefore, positioning and heating can be decoupled. The TEMPUS facility is a realization of an electromagnetic levitation facility designed to operate in microgravity. It allows to study properties and nucleation behavior of undercooled metallic melts. During recent Spacelab flights, experiments have been performed on a number of different materials. Thermophysical properties, such as specific heat, viscosity, surface tension, thermal expansion were measured, and the nucleation behavior as well as the growth velocity of the nucleated stable or metastable phases were studied. In this paper, an overview of the experimental approaches and the results obtained will be given.

3:00 PM

ELECTROSTATIC LEVITATION FURNACE (ELF) FOR THE JAPANESE EXPERIMENT MODULE (JEM): *Hiroki Karasawa*[1]; [1]NASDA, Space Utilization Research Center, Sengen, 2-1-1, Tsukuba-shi, Ibaraki-ken 305 Japan

The Electrostatic Levitation Furnace (ELF) for the Japanese Experiment Module (JEM) can treat many kinds of materials such as metals, semiconductors, and ceramics. The position of the experiment sample is controlled by the electrostatic force between the charged sample and eight independently controlled electrodes whose voltage are determined by the feedback control system using the sample positioning signals detected by 2-axis CCD cameras. The samples will be heated by four lasers. The lasers' power can be controlled independently, and it is possible to perform experiments under vacuum condition or some kind of gas atmosphere. ELF provides various observation capabilities such as a pyrometer, a thermal imaging system, and a video camera. The Preliminary Design Review (PDR) of ELF will be held in March 1999. Preliminary specifications and breadboard model test results will be presented.

3:20 PM BREAK

3:40 PM

EXPERIMENTS PERFORMED BY THE ELECTROSTATIC LEVITATION FURNACE (ELF): *Naokiyo Koshikawa*[1]; [1]NASDA, Space Utilization Research Center, Sengen, 2-1-1, Ibaraki-ken, Tsukuba-shi 305 Japan

Electrostatic levitation is a useful processing method which enables us to process any kind of materials including insulators such as ceramics. As an example, we will show you the preliminary result of the undercooling experiment of BiFeO3 in microgravity performed by using the Electrostatic Containerless Furnace by sounding rocket TR-IA#7. The capability of the Electrostatic Levitation Furnace for ISS is widespread, but the most characteristic point is "lowest turbulence" which gives us accurate physical properties measurements and larger undercooling.

4:00 PM

CONTAINERLESS RAPID SOLIDIFICATION FROM UNDERCOOLED MELT OF OXIDE MATERIALS : *Kazuhiko Kuribayashi*[1]; [1]The Institute of Space and Astronautical Science, 3-1-1 Yoshinodai, Sagamihara, Kanagawa Japan

Abstract not availale.

4:20 PM
OVERVIEW OF SPACE-DRUMS - A DYNAMICALLY CONTROLLED MULTI-BEAM ACOUSTIC LEVITATOR-POSITIONER: *Philip Gregory*[1]; [1]Canadian Space Agency, 6767 Route de l'Aéroport, St-Hubert, Québec J3Y 8Y9 Canada

Space-DRUMS is a new approach for positioning, manipulating, and shaping samples in both gravity and microgravity environments. It relies on the direct acoustic radiation force from many equivalently-positioned ultrasonic beams under dynamic feedback control to leviate or position large samples.

4:40 PM
SCIENCE TO BE PERFORMED ON SPACE-DRUMS: *Rodney Herring*[1]; [1]Canadian Space Agency, 6767 Route de l'Aéroport, Québec , St-Hubert 8Y9, J3Y Canada

Space-DRUMS is designed to support a full range of containerless material processes in a microgravity environment. Acoustic radiation forces will be used to position specimens during scientific investigations.

ALUMINA AND BAUXITE: Silica Chemistry in the Bayer Process

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

Wednesday PM Room: 6E
March 3, 1999 Location: Convention Center

Session Chair: Dr. Peter McIntosh, Kaiser Aluminum & Chemical Corporation, Pleasanton, CA USA

2:00 PM
THE KINETICS OF SEEDED CRYSTALLIZATION DESILICATION OF SYNTHETIC BAYER LIQUOR: *Jonas Addai-Mensah*[1]; Mark C. Barnes[1]; Andrea R. Gerson[1]; [1]University of South Australia, Ian Wark Research Institute, The Levels, Adelaide 5095 Australia

Crystal growth kinetics of pure sodalite, pure cancrinite and their phase mixtures resulting from seeded-desilication of synthetic Bayer liquor have been studied under isothermal, batch precipitation conditions. The temperatures and concentrations used were similar to those at which sodium aluminosilicate scale forms in alumina plant heart exchangers. Sodalite and cancrinite seeding resulted in faster desilication rate and the suppression of scale formation. An activation energy of 30 kj per mol and a second order dependence of the desilication rate on SiO2 relative supersaturation were estimated for sodalite crystal growth. For cancrinite, the rate depended on SiO2 relative supersaturation to the power of 3 with an activation energy of 80 kj per mol. A model for sodalite-cancrinite mixed-phase precipitation showed that the observed kinetics are simply a linear combination of those exhibited by pure crystal growth. It appears that theoretical modelling of sodalite and cancrinite may be achieved using 2 Dimensional Nucleation theories.

2:30 PM
REDUCING ALUMINA PRODUCTION COSTS: *Ronald F. Nunn*[1]; [1]The Winters Company; Tucson, Arizona USA

Alumina is an essential raw material for the production of aluminum in electrolytic cells. There are about 50 operating alumina plants worldwide producing more than 40 million tpy of metal grade alumina by the Bayer process. The cash cost of producing alumina in these plants varies from less than $100/tonne to more than$200/tonne for a number of different reasons. This large variation suggests considerable potential cost savings. Alumina is a worldwide commodity, as a result, very few alumina plants can consider themselves insulated from international competition. Over the next 10 years, about 9 million tonnes of additional alumina capacity is forecast to come on stream. A useful tool for worldwide alumina plant operating cost curve. The bulk of new capacity will no doubt be installed in the plants with low production costs at the bottom end of the cost curve. As a result, those plants at the high end of the curve today will be pushed even higher on the cost curve and potentially out of existence. This paper examines the factors influencing the cost of the major items involved in the cost of producing alumina, including bauxite, caustic soda, fuel, power, labot and maintenance. It then reviews methods for reducing production costs, including improved technology, which will improve a plant's position on the cost curve and make it more competitive.

3:00 PM
SODALITE-CANCRINITE PHASE TRANSFORMATION, KINETICS, AND SOLUBILITY IN BAYER LIQUOR: *Jonas Addai-Mensah*[1]; Mark C. Barnes[1]; Andrea R. Gerson[1]; [1]University of South Australia, Ian Wark Research Institute, The Levels, Adelaide 5095 Australia

The equilibrium solubility of pure sodalite and pure cancrinite crystals in systhetic, spent Bayer liquor has been determined over a range of temperatures and as a function of liquor concentration of NaOH and Al(OH)3. The solubility of both sodalite and cancrinite increased linearly with increasing temperature. For cancrinite, increasing the concentration of NaOH and Al(OH)3 increased solubility dramatically. The mechanism and kinetics of sodalite transformation to cancrinite was investigated under a variety of conditions. The transformation was observed to involve a solution-mediated mechanism with sodalite disolution and subsequent nucleation and growth of cancrinite crystals. Furthermore, the transformation of reaction rate was found to be first order with respect to the relative concentration of sodalite. Over the temperature range of 160-240ϒC, an activation energy of 133 kj per mol was estimated for the overall mechanism .

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4:00 PM
CHARACTERIZATION OF SYNTHETIC ALUMINA TRIHYDRATE CRYSTALS AND BAYER LIQUORS BY THERMAL ANALYSIS: *Jonas Addai-Mensah*[1]; [1]University of South Australia, Ian Wark Research Institute, The Levels, Adelaide 5095 Australia

Modulated and Standard Differential Scanning Calorimetry and Thermogravimetric analysis of several crystalline Al(OH)3, amorphous Al(OH)3 and Bayer liquors have been carried out. The analysis of reversing and non-reversing heat flows showed that the enthalpic events taking place during thermal decomposition are dependent upon particle formation history and structure. The changes in solids specific heat capacity were observed to be influenced by particle size, crystallinity and agglomerate porosity. For synthetic, supersaturated sodium and potassium aluminate liquors, the specific heat capacity decreased dramatically with decreasing concentration of uncoordinated, bulk water. Furthermore, caustic and caustic aluminate solutions made from NaOH appear to have a higher concentration of bulk water than equivalent solutions made from KOH. The difference between bulk water content of potassium and sodium liquors is believed to be associated with the extent of ion-pairing and water coordination abilities of solution Na+ and K+ ions.

4:30 PM
THE SYNTHESIS OF THE PURE Al2O3 LAYERS ON THE SINTERED CORUNDUM CUTTING TOOLS BY THE CVD METHOD: Andrzej Kwatera[1]; Wojciech Tomaszewski[1]; *Mariusz A. Wójcik*[1]; Julian Plewa[2]; Horst Altenburg[2]; [1]Academy of Mining and Metallurgy, Faculty of Ceramic and Materials Engineering, Av. Mickiewicza 30, A-3, Cracow 30-059 Poland; [2]Fachhochschule Munster, Fachbereich Chemieingenieurwesen, Stegerwaldstrasse 39, Steinfurt, Munster D-48565 Germany

The initial conditions of the synthesis of the pure and high density Al2O3 layers on the sintered corundum cutting tools by the CVD method was presented in this paper. The alumina metalorganic compounds were used in the investigations. Results shown that high density and well adhered Al2O3 layers were obtained on cutting tools substrates with the deposition rate of 3,5um/min. Obtained layers can be applicated

for the manufacture of the precise robot parts with exactly controlled chemical composition giving the cutting speed about 900m/min.

ALUMINUM ALLOYS FOR PACKAGING IV: 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

Wednesday PM Room: 3
March 3, 1999 Location: Convention Center

Session Chairs: Subodh K. Das, ARCO Aluminum, Inc., Louisville, KY USA; John E. Adams, Metal Container Corporation, St. Louis, MO 63127-1218 USA

2:00 PM
TEXTURE EVOLUTION AND CORRESPONDING FORMABILITY OF AA 3104 ALUMINUM ALLOYS DURING COLD ROLLING: *Xiang-Ming Cheng*[1]; Yansheng Liu[1]; James G. Morris[1]; [1]University of Kentucky, Light Metals Research Laboratories, Dept. of Chemical And Metallurgical Engineering, College of Engineering, 177 Anderson Hall, Lexington, KY 40506 USA

The deformation behavior of can body aluminum alloys is mainly determined by crystallographic texture. The original hot band texture and the cold rolling process have significant influences on the ultimate texture and thus on the formability. AA 3104 aluminum alloy has been used in this investigation. Before cold rolling the materials have been annealed at certain conditions in order to obtain a fully recrystallized microstructure. Samples at different cold rolling reductions have been prepared for cup testing and crystallographic texture determination. By comparing texture components and the corresponding earing results, a better understanding of the relation between crystallographic texture components and earing is expected. Several degrees of reduction have been employed, especially reductions near the critical point which leads to a change of earing from 90 degrees to 45 degrees. In addition, different heating rates of the hot band have been used to obtain different original textures for the important effect on texture evolution and the resulting ultimate earing behavior.

2:20 PM
CONSTITUTIONAL RELATIONSHIPS FOR THE HOT WORKING OF AA 3004 (Al-1.0 Mn-1.2Mg): *J. Belling*[1]; Hugh McQueen[1]; [1]Concordia University, Mechanical Engineering, Montreal H3G 1M8 Canada

Recrystallized plate of 3004 (0.96% Mn, 1.23%Mg, 0.37Fe) was subjected to hot torsion tests in the ranges 250° to 500°C and 0.1 to 10 s-1. The flow curves strain hardened to a broad peak and softened slightly towards a steady state regime. At higher temperature, T and lower strain rate E the maximum stress s and the softening were lower and the fracture strains were higher. The exponential law was found satisfactory, but the power law was not. The constants A, n, Q HW for the equation: A(sinh as)n = E exp (QHW/8.31 T) were derived for a ranging from 0.01 to .008 MPa -1. The constants differed slightly for longitudinal and transverse specimens. The use of a = 0.04 or 0.06 MPa -1 makes comparison with published data simpler; the agreements were reasonable given the variations in composition. Optical microscopic examination revealed elongated grains in which the subgrains became more clearly defined as T rose and E diminished. Clearly dynamic recovery provided good hot workability through a substructure with reduced strain energy which also inhibited recrystallization unless the product is heated to about 50° above the deformation temperature as previously published.

2:40 PM
INVESTIGATION OF LOW RESOLUTION TEXTURE ANALYSIS FOR ON LINE R- VALUE DETERMINATION OF CAN BODY MATERIALS: *Y. Liu*[1]; X. M. Cheng,[1]; Y.L. Liu[1]; C.S. Man[1]; J. G. Morris[1]; [1]University of Kentucky, Light Metals Research Laboratories, Dept. of Materials and Chemical Engineering, Lexington, KY 40506 USA

Aluminum alloys for can body making are produced by hot and cold rolling. The hot band or annealed hot band is the initial condition for subsequent cold rolling. Excellent deep drawability is mainly determined by the initial texture of the hot band if the cold reduction is constant. On line determination of the Lankford parameter (R-value) for the hot band is very important to industrial practice. In the present paper, hot bands of direct chill cast (DC) and strip cast (SC) AA3004 aluminum alloys were annealed at different temperatures in order to simulate the exit condition of the alloys from hot rolling mills. Textures were determined by the normal pole figure method and low resolution technique which determines several texture components by X-ray diffraction. The empirical equations between the texture components determined by the low resolution technique and other texture components are derived. The on line R-value determination is simulated and the reliability is discussed.

3:00 PM
THE INFLUENCE OF PRE-HEAT TREATMENT ON THE RECRYSTALLIZATION AND TEXTURE OF STRIP CAST AA3004 ALUMINUM ALLOY: *Y. L. Liu*[1]; J. Qiu[1]; G. Liao[1]; J.G. Morris[1]; [1]University of Kentucky, Light Metals Research Laboratories, 177 Anderson Hall, Lexington, KY 40506 USA

The strip cast process provides a significant advantage for producing packaging materials. However, the metallurgical and mechanical characteristics of the material produced by the strip cast process are quite different from material produced by the traditional DC casting process. Therefore, it is of industrial interest to carry out further research on the strip cast material. In this study, the packaging material, aluminum alloy AA3004, was industrially produced by the strip cast process. The hot band was subjected to various heat treatments. The evolution of the microstructure and texture during heat treatment was examined. The influence of heat treatment on the recrystallization behavior and texture behavior of the cold rolled sheets was invested. The effect of pre-existing texture on the evolution of texture during annealing of the cold rolled sheets is discussed.

3:20 PM BREAK

3:40 PM
DEFORMATION MICROSTRUCTURE AND SLIP PATTERN IN ALUMINUM: *N. Hansen*[1]; [1]Riso National Laboratory, Materials Research Dept., Rosklide DK-4000 Denmark

Aluminium and aluminium alloys have been deformed in tension, rolling and channel die compression over a large strain range and a temperature range from RT to 600ϒC. The structural evolution has been followed by applying semiautomatic and automatic SEM and TEM techniques and it has generally been observed both in single crystals and polycrystals that the structures are subdivided by deformation induced dislocation boundaries and high angle boundaries. These boundaries have different characteristics depending on materials and process parameters. Of importance is the crystallographic orientations of the crystals or the grains. This parameter has been studied for the different deformation modes and it has been found that links exist between microstructure and local crystallography and the macroscopic plastic behaviour. This has led to an analysis of the effect of the slip pattern on the microstructural evolution as a function of strain and temperature. Finally is discussed the effect of grain orientation and slip pattern on the dislocation density and the stored energy of relevance to the modelling of the mechanical and thermal behaviour of aluminium and aluminium alloys.

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THE KINETICS OF ISOTHERMAL β" AND β' FORMATION IN AlMgSi ALLOYS: *Jörgen van de Langkruis*[1]; Marcel S. Vossenberg[1]; Wilhelmus H. Kool[1]; Sybrand van der Zwaag[1]; [1]Delft University of

Technology, Laboratory of Materials Science, Rotterdamseweg 137, Delft, Zuid Holland 2628 AL The Netherlands

The mechanical properties during and after processing of AlMgSi alloys are strongly influenced by the state of the alloying elements in the alloy. Especially, the formation of Mg2Si β'-phase has an important effect on the extrudability and also on the mechanical properties after extrusion by binding solute Mg and Si. In this work DSC experiments on several commercial and laboratory AlMgSi grades are described. The DSC used is equipped with fast heating (500 K/min) and quenching (200 K/min) facilities. The samples were solutionised, precipitated to induce isothermal transformation into β' or β'', rapidly quenched and subjected to a DSC scan. The heat treatment cycle was entirely performed in the DSC, which strongly improves experimentation time and quality. The effect of the heat treatments on the size and location of the β' and β'' peaks was studied. For an AA6063 type alloy it was found that after precipitation at 458 K the size of the β'' peak decreased with precipitation time, which is correlated with the amount of β'' already precipitated. It was also found that with increasing precipitation time, using a scanning rate of 10 K/min, the location of the β''-peak shifted to lower temperatures, whereas with scanning rate 5 K/min only the peak size decreased and a peak shift was not observed. This is attributed to the retarding effect of nucleation on the transformation, which is significant at high scanning rates and low β'' contents. Isothermal β' and β'' phase transformation diagrams of some of the grades investigated are presented. Compositional effects were also observed: adding excess Mg, excess Si or Fe changes the size and location of the β' peak and tends to decrease the 50% β' transformation time. Also some precipitation peaks were observed, which do not occur in stoichiometric AlMgSi alloys.

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ODF ANALYSIS ON TEXTURE OF 3004 ALLOY SHEET: *Sachio Urayoshi*[1]; Ryou Shoji[1]; [1]The Furukawa Electric Co., Ltd., Material Engineering Section, 21-1 Kurome, Mikuni-cho, Sakai-gun, Fukui Prefecture 913-0048 Japan

The rolling texture of 3004 aluminum alloy was analyzed by examining its orientation distribution function (ODF). The aim was to study the difference texture resulting from two typical production methods for can body stock. First, the rolled materials were studied right after intermediate annealing; it was found that the material continuously annealed after being hot rolled (hereafter called HCAL) had a recrystallized texture with a high accumulation of the cube-orientation {001} <100>. However the material continuously annealed after being both hot rolled and cold rolled (hereafter called ICAL) had a lower cube-orientation accumulation than the HCAL material, and it showed a random recrystallized texture with little accumulation of any particular orientations. On the other hand, after final cold rolling, the HCAL material, with a high final reduction, exhibited a deviation in peak orientation density from the S orientation to the Brass orientation, and the ICAL material, with a low reduction, from the S orientation to the Cu orientation. In both cases, the peak orientation did not coincide with the S orientation which is characteristic of the rolling texture.

ALUMINUM REDUCTION TECHNOLOGY: Emerging Technologies

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, Ltd., Brisbane, Queensland 4001 Australia

Wednesday PM Room: 6F
March 3, 1999 Location: Convention Center

Session Chairs: Jay Bruggeman, Alcoa Aluminium Company of America, Chem. Sys. Div., Alcoa Center, PA 15069-0001 USA; Barry Welch, The University of Auckland, Dept. of Chem. & Mats. Eng., Auckland 1001 New Zealand

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PATHS FOR ALUMINIUM PRODUCTION IN THE NEW MILLENNIUM: *B. J. Welch*[1]; [1]Auckland University, Dept. of Chemical and Materials Engineering, Private Bag 92019, Auckland New Zealand

In the last two decades we have seen the demise of the much heralded ASP Chloride Process as an alternative Aluminium metal production path, a drying up of papers on carbo-thermal production options, but a steady stream of articles proposing the use of drained cathode technology (by a wettable titanium diboride coating) and others extolling the virtues and potential materials for inert anode technology. We also saw a rush of smelter technologies papers in the early 1980's claiming energy consumption achievable less than 12.5 kWh per kg. However the recent emphasis has been on high amperage, more cost efficient technologies. Current efficiencies in excess of 96% can be routinely obtained by new technologies and even aged technologies can be retrofitted to perform at 95%. The challenge then is to lower cell voltages and one of the key limitations for this is the need to maintain adequate superheat while avoiding of sludge formation and electrolyte concentration gradients. Electrochemical technology based on the new concepts being considered (drained wetted cathodes, inert anodes, and even chloride electrolytes) face exactly the same problems and challenges that the present technology does but they are even more acute and demanding. These challenges can be met however and undoubtedly the industry is faced with interesting times as it continues to strive to be competitive.

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INERT ELECTRODES IN ALUMINIUM ELECTROLYSIS CELLS: *H. Kvande*[1]; [1]Hydro Aluminium Metal Products, P.O. Box 80, Stabekk N-1321 Norway

Inert electrodes would mean a technological revolution of the Hall-Héroult process. The production costs for aluminium may be reduced by 15 to 20% by avoiding the consumable carbon anodes, and by lowering the energy consumption by use of a wettable or drained cathode. Environmentally, all $C0_2$ and CF_4 gas emissions from the cells would be eliminated. Considerable progress has been achieved with respect to the development of wettable cathodes on the basis of titanium diboride. However, little or no success has been reported for inert anodes, and the development of an inert anode material has appeared to be extremely difficult. In spite of its high research priority, it is possible that a viable technological solution for an inert anode may not be found in the foreseeable future. The present paper reviews the state-of-the-art of inert anodes and cathodes in aluminium electrolysis cells, and discusses their main economical, technological and environmental advantages. Bipolar cells would be the superior cell construction, but these still represent a great challenge to researchers in the field of materials science.

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A DYNAMIC INERT METAL ANODE: *J. N. Hryn*[1]; M. J. Pellin[2]; [1]Argonne National Laboratory, Energy Systems Division, 9700 S. Cass Ave., Bldg. 362, Argonne, IL 60439 USA; [2]Argonne National Laboratory, Mats. Sci. Division, 9700 S. Cass Ave., Bldg. 200, Argonne, IL 60439 USA

A new concept for a stable anode for aluminum electrowinning is described. The anode consists of a cup-shaped metal alloy container filled with a molten salt that contains dissolved aluminum. The metal alloy can be any of a number of alloys, but it must contain aluminum as a secondary alloying metal. A possible alloy composition is copper with 5 to 15 weight percent aluminum. In the presence of oxygen, aluminum on the metal anode's exterior surface forms a continuous alumina film that is thick enough to protect the anode from chemical attack by cryolite during electrolysis and thin enough to maintain electrical conductivity. However, the alumina film is soluble in cryolite, so it must be regenerated in situ. Film regeneration is achieved by the transport of aluminum metal from the anode's molten salt interior through the metal wall to the anode's exterior surface, where the transported aluminum oxidizes to alumina in the presence of evolving oxygen to maintain the protective alumina film. Periodic addition of aluminum metal to the anode's interior keeps the aluminum activity in the molten salt at the desired level. This concept for an inert anode is viable as long as the amount of aluminum produced at the cathode greatly exceeds the amount of aluminum required to maintain the anode's protective film.

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REDUCTIONS CONDITIONS ENCOUNTERED IN CRYOLITE BATHS: *J. A. Sekhar*[1]; J. Liu[1]; H. Deng[1]; [1]University of Cincinnati, Dept. of Materials Science and Engineering, International Center for Micropyretic, P.O. Box 210012, Cincinnati, OH 45221 USA

We have noted an unusual effect that several of the oxide and ferrite materials which are being considered for use as non-consumable anodes are reduced by the bath to the metallic state when held for even short durations in the bath. Conditions for such reduction are explored. In addition, when reduced to metal, the product retains the shape and approximate dimensions of the initial oxide form. The implications of this effect on the utility as a non consumable anode are explored.

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CHARACTERIZATION OF WETTABLE TiB2-CARBON MATERIAL COMPOSITE: *M. Dionne*[1]; A. Mirtchi[2]; G. L'Espérance[1]; [1]Centre for Characterization and Microscopy of Materials, Dept. of Metallurgical and Materials Engineering, Ecole Polytechnique de Montreal, P.O. Box 6079, Succ. Centreville, Montreal, Quebec H3C 3A7 Canada; [2]Alcan International, Ltd., Electrolysis, 1955, Mellon Blvd., Jonquiere, Quebec G7S 4K8 Canada

Since the continuous improvement of the conventional carbon cathode blocks over the years and the advantages of TiB_2 such as good wettability by liquid aluminum, relatively good electrical conductivity and wear resistance, it was an attractive idea to intimately mix those two giving birth to a wettable TiB_2-carbon material with totally new properties. In this presentation, the chemical stability of TiB_2 particles in molten aluminum and in the electrolytic bath is discussed. Results showing the characterization of TiB_2-carbon composite material after electrolysis and immersion tests are presented as well as results showing the dissolution of TiB_2 particles, by the electrolytic bath, along specific crystallographic planes. Furthermore, evidence that Al_2O_3 saturated electrolytic bath enhances dissolution of TiB_2 is presented.

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CATHODES WETTED BY ALUMINUM IMPROVE CURRENT EFFICIENCY: *W. Haupin*[1]; [1]2820 Seventh St. Rd., Lower Burrell, PA 15068 USA

The two major factors affecting current efficiency are bath chemistry and stability of the aluminum-bath interface. Bath chemistry sets the driving force at the aluminum-bath interface to transport dissolved metal into the bath where it becomes reoxidized. A stable aluminum-bath interface promotes a thick boundary layer that slows this transport. An unstable metal pad results from the motor action produced by horizontal currents in the aluminum interacting with the vertical magnetic field of the cell. Horizontal currents are caused primarily by undissolved alumina (muck) under the aluminum and to a degree by poor anode current balance. Muck forms in the bath filled capillary between the aluminum and the cathode (bottom of the cell). When the cathode is wetted by aluminum, there is no capillary and hence no place for muck to form and destabilize the cell. If cell design proceeds a step further to a wetted and drained cathode, the ultimate aluminum-bath interface stability is achieved and the maximum current efficiency for a given bath chemistry results.

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Al-Si CARBOTHERMAL REDUCTION USING HIGH-TEMPERATURE SOLAR PROCESS HEAT: *Jean P. Murray*[1]; [1]Colorado School of Mines, Engineering Division, Golden, CO 80401 USA

Aluminum production by carbothermal reduction is a very high-temperature, energy-intensive process. The temperature required, in the range 2300-2500K, is too high for practical process heat addition from combustion sources alone. Fuels must be burned in pure oxygen to reach 2000K, with very little process heat available, and combustion products present in the metal-forming zone interfere with the recovery of metal. Combustion-generated electricity contains only about a third of the energy in the fuel, and losses in the arc furnace cause further losses. Highly concentrated sunlight is capable of supplying adequate process heat at high temperatures. Alcoa researched and patented a carbothermal reduction process to make an Al-Si alloy suitable for further refining to pure aluminum or for use directly as an alloy. This process has been adapted to accept solar process heat and has undergone preliminary testing in a solar furnace.

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INVESTIGATION OF THE CONCENTRATION OF IMPURITIES FROM THE INERT ANODES IN THE BATH AND METAL DURING ALUMINIUM ELECTROLYSIS: *S. Pietrzyk*[1]; R. Oblakowski[1]; [1]University of Mining and Metallurgy, Dept. of Metallurgy Faculty of Non-Ferrous Metals, Al. Mickiewicza 30, Krakow 30-059 Poland

Three types of anodes: NiO-Fe_2O_3-CuO, NiO-Fe_2O_3-Cu, Cr_2O_3-NiO-CuO, were presented as candidates for inert anodes in Hall-Heroult process. The anodic behavior of anodes was studied during the electrolysis of Al_2O_3 in the 2,7 NaF,AlF_3 melt at 980ŶC. Inert anodes are subjects of simple mass transport controlled chemical dissolution into the electrolyte with subsequent transfer to cathodic metal surface where they are being reduced and dissolved into aluminium. The purpose of the present paper is to determine the factor governing the rates of anode dissolution and contamination of aluminium product.

CAST SHOP TECHNOLOGY: Cast House Safety

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

Wednesday PM Room: 6C
March 3, 1999 Location: Convention Center

Session Chair: Seymour G. Epstein, The Aluminum Association, Washington, DC 20006 USA

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THE ALUMINUM ASSOCIATION'S MOLTEN METAL SAFETY PROGRAM: *Seymour G. Epstein*[1]; [1]The Aluminum Association, Inc., 900 19th Street, NW, Washington, DC 20006 USA

The aluminum industry has made an extensive effort to gain an understanding of molten metal explosions, the conditions under which they occur, and how they may be prevented. The Aluminum Association, the organization that represents the aluminum industry in the United States, has long considered the handling of molten aluminum its single greatest safety priority and has established an ongoing molten metal safety program to address the issues. The program includes research into the causes and prevention of molten aluminum water explosions; development and dissemination of guidelines for handling molten aluminum, for scrap receiving and inspection, and for sow casting and charging; a molten metal incident reporting program; a scrap rejection notification program; testing of fabrics to protect employees exposed to molten metal and pot bath; and a series of workshops, presentations and training aids to increase awareness. These will be discussed in the presentation.

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REDUCTION OF SOW DRYING CYCLE TIME USING MATHEMATICAL MODELING AND EXPERIMENTAL CONFIRMATION TESTS: *A. Giron*[1]; R. T. Richter[1]; [1]Aluminum Company of America, Alcoa Technical Center, Alcoa Center, PA 15069 USA

One of the most potentially dangerous cast house practices can be the charging of sows into molten metal that have not been dried or have been insufficiently dried. Aluminum sows can contain large shrinkage cavities which will very often be partially or completely filled with water. Several explosions causing severe burns, massive equipment damage and even fatalities have resulted when sows containing water were charged into molten metal. Alcoa, like other companies, have used very conservative sow drying practices to insure the moisture has completely evaporated from these shrinkage cavities. This, however, has caused production constraints due to limited drying furnace capacity. A program was undertaken using mathematical modeling supported by experimental testing to establish reduced, but still conservative, drying times for 1500 lbs. sows.

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INVESTIGATION OF COATINGS WHICH PREVENT MOLTEN ALUMINUM/WATER EXPLOSIONS: *D. D. Leon*[1]; R. T. Richter[1]; T. L. Levendusky[1]; [1]Aluminum Company of America, Alcoa Technical Center, Alcoa Center, PA 15069 USA

The Aluminum Association contracted Alcoa in 1995 to identify and test new protective coatings for casting pits as a replacement for Porter International's 7001 (Tarset Standard). Three new coatings have been identified through a series of selection criteria including: 1) An industry standard molten metal explosion test, 2) A multiple exposure test to measure durability, and 3) An external shock impact test. The results of this program will be reviewed. This study only tested protective coatings at the "in service cure time", as defined by the manufacturer. These curing times can be excessive for a production casting facility. The Aluminum Association has contracted Alcoa in a second program to investigate the effect of reduced cure times on coating adhesion and their effectiveness in preventing molten metal/water explosions. A status update of this new two year program is provided.

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FUNDAMENTAL STUDIES ON MOLTEN ALUMINUM-WATER EXPLOSION TRIGGERING IN CASTING PITS: *Rusi P. Taleyarkhan*[1]; S. H. Kim[1]; [1]Oak Ridge National Laboratory, Engineering Technology Division, Bldg. 9204-1, MS 8045, Oak Ridge, TN 37831-8045 USA

Oak Ridge National Laboratory (ORNL) is conducting research on understanding fundamentals of molten metal-water explosion prevention with the Aluminum Association (AA). Phenomenological issues related to surface wettability, gas generation from coatings, charring of coatings, external shocks, melt/water temperature, inertial constraint, etc. are being investigated systematically to gage their relative impact on triggerability of surface assisted explosions. The Steam Explosion Triggering Studies (SETS) facility was designed and constructed to cost-effectively and safely address these issues. Hundreds of tests have been conducted to assess the viability of this approach to predict onset of explosion triggering. Uncoated surfaces included stainless and carbon steels, rusted steel, aluminum and concrete. Coatings tested included lime, silicone paint, graphite pain, greases, various coal tar epoxies, solid epoxies and epoxy mastics. Tests were also conducted to assess the impact of durability and bare spots. Data from studies with SETS facility were compared against field data taken over the past 50 years with excellent (100% correlation) results. The full paper will describe details of results of testing over coated and uncoated surfaces. Also described in the full paper will be a description of on-going work related to theoretical modeling, and future work dealing with addressing key issues related to impact of curing time, and field validation experiments of a novel prevention technique based on intentional gas injection at vulnerable locations.

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WILL AUTOMATION MAKE YOUR ALUMINUM CASTHOUSE A SAFER PLACE TO WORK?: *John E. Jacoby*[1]; [1]Consultant, 3398 North Hills Rd., Murrysville, PA 15668 USA

Techniques have been developed to start, run and complete drops of rectangular aluminum ingot cast by the DC, EMC and LHC processes without having personnel on the casthouse floor exposed to the hazards of molten aluminum explosions. During the entire cast all personnel are in a protected control room observing the cast on monitors. No operator intervention is required to start or end a drop. Do these automation systems improve casthouse safety and productivity? Answers to these important questions will be provided.

4:25 PM PANEL DISCUSSION

CAST SHOP TECHNOLOGY: Continuous Casting / Commercial Casting

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

Wednesday PM Room: 6D
March 3, 1999 Location: Convention Center

Session Chair: Dr. Jerry Dassel, Commonwealth Aluminum Corp., Uhrichville, OH 42351 USA

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HEAT TRANSFER IN THE SINGLE-ROLL STRIP CASTING PROCESS: *E. N. Straatsma*[1]; L. Katgerman[1]; W. H. Kool[1]; [1]Delft University of Technology, Laboratory of Materials Science, Rotterdamseweg 137, Delft, Zuid Holland 26 28 AL The Netherlands

In this research the influences of process parameters on the quality of 1 mm strip has been determined. Casting with a vertical feeding system is not recommended because of introducing turbulences resulting in bad strip quality. The quality can be classified with micrographs, roughness measurements and with thickness determinations. The stripcast process can also be simulated to determine the heat transfer coefficient between strip and wheel. The heat transfer coefficient between substrate and strip is one of the most important facts which influences the solidification rate. The determination of the heat transfer coefficient is very difficult and most of the simulation models assume values in a range of 8-20 kW/m2K. We are able to determine the heat transfer coefficient by online temperature measurements in the wheel of a single roll strip caster at laboratory scale. Together with the continuous measurement of the final strip temperature it is possible to compute the heat transfer coefficient.

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STRIP CASTING OF ALUMINIUM USING TWIN ROLL CASTERS: *Manish Gupta*[1]; Dan P. Cook[2]; Yogeshwar Sahai[1]; [1]The Ohio State University, Mats. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Reynolds Metal Company, Corporate Research and Development, Richmond, VA 23219 USA

Twin roll casters are the most commonly used machines for strip casting of aluminium. These casters offer low investments and very high operational flexibility. In the present work, turbulent fluid flow, heat transfer and solidification during strip casting of aluminium by twin roll caster was simulated by a two-dimensional finite element model. From this model, roller speed, contact angle, gap heat transfer coefficient and melt super heat were identified as important process variables which affect the thickness of cast strip. The model also predicts velocity and temperature profile in melt pool and the roller. Effect of angle of injection of molten metal into melt pool on the process was also studied. From the present study, roller speed, contact angle and gap heat transfer coefficient were found to be the main parameters affecting the strip thickness. Cooling fluid temperature, melt superheat, roller thickness and roller material were found to have little effect. Applicability of this mathematical model in industrial production of aluminium strips was also verified.

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TWIN ROLL CASTING OF ALUMINUM AT 2.5 MM GAUGE - PRODUCTION EXPERIENCE AND PROCESS IMPROVEMENTS: *S. Hamer*[1]; D. Smith[1]; B. Taraglio[1]; C. Romanowski[1]; [1]Fata Hunter, 6147 River Crest Dr., P.O. Box 5677, Riverside, CA 92507 USA

Assan Demir Sac Sanayi A.S. in Turkey has now purchased four FATA Hunter SpeedCaster machines. The first of these machines was commissioned two years ago and has been casting 2.5 mm thick aluminum strip on a production basis for almost one European and Near East markets. To achieve stable and high quality production, some of the equipment and casting parameters required optimization. This paper describes these process improvements together with the resulting production rates and cast strip properties. This long-term operation has also revealed some aspects of the process which needed further refinement. Development programs which addressed these issues and plans for further down gauging are briefly reviewed. The operational experience gained has been incorporated into the next generation of casting line automation systems. Key features of these new automation systems are highlighted.

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COMMONWEALTH ALUMINUM -STATE OF THE ART CONTINUOUS CASTING: *Stan Platek*[1]; [1]Commonwealth Aluminum, 7319 Newport R., SE, Uhrichsville, OH 44683 USA

The operating experience of Commonwealth Aluminum in continuous strip casting spans over twenty years and four Hazelett twin belt casters. Billions of pounds of strip have been cast in widths ranging from 12-52 inches. Alloy and production capabilities have been dramatically expanded in recent years by virtue of improved casting belt surfaces, magnetic caster mold stabilization techniques and new and improved metal feeding systems. Improved belt surfaces provide a more consistent and higher level of cast slab surface quality. Powerful magnetic means constrain the casting belts from thermal distortion providing an opportunity to employ a greater range of cooling rates for specific alloys. The use of unique closed pool feeding nozzles employing a combination of refractories and metals reveals the subtle effects of metal flow on surface metallurgy. The chronology of the emergence of Commonwealth continuous cast product into the aluminum sheet mainstream is presented along with a detailed review of the most current technologies employed and the resulting metallurgy and product.

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CASTEX CONTINUOUS BAR CASTING FOR THE NEXT MILLENNIUM:B. Maddock[1]; P.M. Thomas[1]; R. Wilkinson[1]; [1]Holton Conform Ltd., United Kingdom

Over the past decade a number of changes have occurred in the production of aluminum strip products. There has been a general trend towards downgauging and a move toward true mini-mills. If the production of feedstock for products other than strip is examined, it is clear that the same logic can be applied to these processes. The number of process operations can be reduced with the cast gauge/cross section much closer to that of the finished product. Most of the feedstock for extrusions and wire and rod products is produced either by traditional billet casting or by wheel and belt casters and in both cases, considerable downstream equipment is required, and the productivity requirements for economic production high. Conform is an established technology for the production of a variety of extrusions and rod and wire products and is capable of processing a number of different feedstocks. The feedstock can be bar that is continuously cast immediately prior to the Conform machine and there is the potential for using molten metal as the feedstock. The paper will describe a Castex installation where continuously cast feedstock is produced and the results and process issues will be discussed. In addition, an experimental facility to evaluate direct liquid metal feed will be described.

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A GENERAL APPROACH FOR DETERMINATION OF RELEASE DIRECTIONS FOR DIECASTINGS: *Lu Hongyuan Lu*[1]; W. B. Lee[1]; [1]Hong Kong Polytechnic University, Manufacturing Engineering, Hung Hom, Kowloon, Hong Kong China

In design of diecasting dies, the determination of parting surface is particularly important and heavily depends on the individual experience of designer. In order to assist design automation of diecast die, a computer-aided approach is presented for choice of parting direction and parting surface based on the geometry features. According to the translation principle of a rigid body, all candidate parting directions (CPD are solved out. A CPD distribution order is used for evaluating the priority of each CPD acting as parting direction in removability. Then a preferred parting direction and parting surface can be determined. The geometry features are extracted by sweeping the shape of cast component to be cast. The CPD distribution order is obtained by finding the intersection of removable direction polygons.

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AN EXPERIMENTAL AND COMPUTATIONAL EVALUATION OF THE INFLUENCE OF PERMANENT MOULD DESIGN ON THE SOLIDIFICATION OF Al7SiMg CASTINGS: *John Anthony Spittle*[1]; Stephen G. R. Brown[1]; Hannelore Wishart[1]; [1]University of Wales Swansea, IRC in Materials for High Performance Applications, Singleton Park, Swansea SA2 8PP UK

An experimental permanent mould, for Al7SiMg alloy castings having a tensile test specimen geometry, has been used to assess the influence of mould geometry and water cooling on the steady-state temperature distribution in the mould and the macro-freezing pattern of the casting. From observation, a steady-state was assumed to be achieved in any batch run after 20 castings. The temperature distribution was derived from thermocouples located in the mould and was compared with the distribution predicted by the MAVIS heat transfer/solidification simulation package. The freezing pattern was experimentally evaluated, from the variation in secondary dendrite arm spacing in the casting, and was compared with the macrofreezing pattern predicted by MAVIS. It was found that mould sculpting, casting fins and mould water cooling had little influence on the freezing pattern which was dominated by the casting geometry.

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INFLUENCE OF MOLD COATING PROPERTIES ON ALUMINUM ALLOY CENTRIFUGAL CASTINGS: *Kevin Gilbert Cook*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

Tubular Al-Si parts were produced using a permanent mold horizontal centrifugal caster. The effects of mold coating, rotation speed, and mold temperature on the casting micro and macrostructures were evaluated. Permeabilities of boron nitride, graphite, bentonite, and alumina coatings were measured using a modified green sand perimeter. The coatings were applied to the cylindrical low carbon steel mold and preheated before pouring the molten metal. Mold preheating temperatures were varied from 50-250ŸC (122-482ŸF). The cast products were examined for surface and structural defects, and the samples were sectioned

and analyzed using optical microscopy, scanning electron microscopy, and x-ray diffraction. Results showed that element distribution in the cast structure is strongly dependent on rotation speed. Typically, higher mold temperatures and high permeability coatings produced better casting structures. Heat transfer coefficients were also calculated for the corresponding coating systems.

CREEP BEHAVIOR OF ADVANCED MATERIALS FOR THE 21ST CENTURY: Low Stress Creep Mechanisms: A Discussion 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

Wednesday PM Room: 15A
March 3, 1999 Location: Convention Center

Session Chair: A. K. Mukherjee, University of Canifornia, Davis, CA 95616 USA; T.R. Bieler, Michigan State University, East Lansing, MI 48824-1226 USA

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2:10 PM INVITED PAPER
QUANTITATIVE PREDICTIONS OF THE DISLOCATION NETWORK THEORY OF HARPER-DORN CREEP: *Alan J. Ardell*[1]; Marek Przystupa[1]; [1]University of California, UCLA, Dept. of Materials Science & Engineering, 6531-G BH, Los Angeles, CA 90095 USA

It has been demonstrated that the dislocation network theory of high-temperature deformation is capable of explaining many features of Harper-Dorn (H-D) creep. The observation that the dislocation density in the H-D creep regime is independent of the applied stress is explained by the frustration of dislocation network coarsening, which arises because of the exhaustion of Burgers vectors that can satisfy Frank's rule at the nodes in the network. The theory also accounts for the dramatic reduction in the steady-state creep rate that obtains when the initial dislocation density is very large, for example in specimens deformed by cold-working prior to creep testing. Other characteristics of H-D creep, such as the transition stress from H-D to power-law creep and the reduction in the dislocation density during primary creep in the H-D regime, are also satisfactorily explained. No other theory of H-D creep can account for the totality of these experimental observations. In this work we attempt to solve the equations of the network theory in order to provide a quantitative description of the experimentally measured distributions of dislocation link lengths in Al, and a self-consistent description of creep curves in Al deformed in the H-D regime.

2:35 PM INVITED PAPER
DIFFUSION CREEP IN CERAMICS: *Atul H. Chokshi*[1]; [1]Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India

At sufficiently low stresses, where intragranular dislocation mobility is rather limited, plastic deformation can occur solely by the diffusion of vacancies either through the matrix (Nabarro-Herring) or along grain boundaries (Coble). The process of diffusion creep has been modeled theoretically for over 50 years now, although there still remain some doubts over the experimental validation of the models. It has been suggested that low intragranular dislocation mobility leads to more frequent observations of diffusion creep in ceramics compared to metals. In ceramics, the diffusion creep process is more complex than in metals due to the need to account for charge balance and the transport of two or more ionic species along two different paths (lattice or grain boundary). The present report will evaluate critically the experimental observations of diffusion creep in some oxide-based ceramic systems. In addition, the process of ambipolar diffusion in ceramics will be examined with two different considerations: (a) the total flux to grain boundaries is in the appropriate stoichiometric ratio, so that the cations and anions may be transported along different paths, and (b) the flux along each transport path is in the appropriate stoichiometric ratio. It will be demonstrated that the above two considerations lead to substantially different predictions on rate controlling processes.

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DIFFUSIONAL CREEP AND HARPER-DORN CREEP AT INTERMEDIATE TEMPERATURES: *Lubos Kloc*[1]; Jaroslav Fiala[1]; Josef Cadek[1]; [1]Academy of Sciences of the Czech Republic, Institute of Physics of Materials, Zizkova 22, Brno CZ-61662 Czech Republic

Viscous creep was observed at temperatures close to one half of absolute melting point at very low creep rates in many materials. The results were interpreted as Coble diffusional creep and/or Harper-Dorn dislocation creep. Some of the results are in very good agreement with the Coble theory of diffusional creep and can support the diffusional creep as an important deformation mechanism under certain conditions. Nevertheless, the theory of diffusional creep seems to be too simplified to describe accurately the diffusional creep processes. In several materials, dependence of the creep rate on grain size corresponds to Coble diffusional creep for grain sizes below approximately 100 μm, while this dependence is replaced by large data scatter for coarse grain sizes. This behaviour used to be interprete as a transition from diffusional to Harper-Dorn creep regime. The structural parameter responsible for the large scatter of creep rates at large grain sizes has not been identified yet.

3:20 PM
EXPERIMENTAL STUDIES OF CREEP PHENOMENA AT LOW STRESSES: *K. R. McNee*[1]; Howard Jones[1]; [1]University of Sheffield, Dept. Engineering Materials, Mappin Street, Sheffield S1 3JD UK

Some creep formulations indicate a continuity of relationships throughout large ranges of stress and temperature. Other evidence has also been presented indicating distinct regimes each representing the predominance of a specific deformation mechanism. In low stress creep the theory is well established to indicate that directional diffusion has an important role and provides a quantitative dependence of creep rate on measurable parameters. Experiments are described which provide evidence of the stress, temperature and grain size dependence of creep which, in combination with microstructural observations, provides clear support for the occurrence of diffusional creep processes.

3:40 PM
PRIMARY CREEP OF METALLIC MATERIALS AT LOW STRESSES AND INTERMEDIATE TEMPERATURES: *Lubos Kloc*[1]; Vaclav Sklenicka[1]; [1]Academy of Sciences of the Czech Republic, Institute of Physics of Materials, Zizkova 22, Brno CZ-61662 Czech Republic

Some creep mechanisms acting at low stresses have not their own primary stage. Nevertheless, the primary creep has been observed in almost all low stress creep experiments. Hence, special primary creep mechanisms must be responsible for primary creep under such loading conditions. Better understanding of these mechanisms seems to be important for engineering practice, because the overall strain allowed for some components in high temperature technology may be essentially exhausted by primary creep. The primary creep parameters like primary strain, the duration of primary creep stage, initial creep rate and initial to secondary creep rate ratio are analyzed for several metallic materials from pure metals to heat-resistant steel. It can be concluded, that no mechanism proposed till now for primary creep at low stresses is capable to explain all observed features of primary creep under given conditions.

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PRIMARY CREEP AND ANELASTICITY AT LOW STRESSES: *R. S. Mishra*[1]; A. K. Mukherjee[1]; [1]University of California, Department of Chemical Engineering and Materials Science, One Shields Avenue, Davis, CA 95616 USA

At low stresses, three deformation mechanisms are dominant; diffusional flow, grain boundary sliding and viscous dislocation creep. The relative contribution is governed by the grain size and temperature. The magnitude of primary creep is often significantly higher than that predicted by theoretical or phenomenological models for these mechanisms. Some anelasticity results show that the primary creep at low stresses is fully recoverable. This raises doubts about the applicability of previous models for primary creep at low stresses. The advantages of anelasticity studies for understanding of low stress creep mechanism are discussed.

4:20 PM DISCUSSION ON LOW STRESS CREEP MECHANISMS

ELECTRICAL AND THERMAL PROPERTIES OF MATERIALS: Session II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sungho Jin, Lucent Technologies, Bell Labs, Murray Hill, NJ 07974 USA; Anthony Mulligan, Advanced Ceramics Research, 851 East 47th St., Tuscon, AZ USA; King Ning Tu, University of California, Dept. of Mats., Sci. & Eng., Los Angeles, CA 90095 USA

Wednesday PM Room: 16B
March 3, 1999 Location: Convention Center

Session Chair: S. Jin, Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974 USA

2:00 PM INVITED PAPER
HEAT CAPACITY, PHONONS, AND VIBRATIONAL ENTROPY OF NANOCRYSTALS: *B. Fultz*[1]; H. Frase[1]; J. L. Robertson[2]; [1]California Institute of Technology, Mail 138-78, Pasadena, CA 91125 USA; [2]Oak Ridge National Laboratory, Mail 6393, P.O. Box 2008, Oak Ridge, TN 37831 USA

The vibrational entropy and the heat capacity of a solid are determined primarily by its "phonon density of states DOS." This spectrum of interatomic vibrations is altered considerably when crystallites are 10 nm or smaller. From measurements by inelastic neutron scattering on Ni_3Fe [Phys. Rev. B57, 898 (1998)], we have identified two significant changes in the phonon DOS of nanocrystals: 1) an enhancement in the number of vibrational modes at low energies, and 2) a broadening of features in the phonon DOS, especially at high energies. The extra low energy modes can be attributed to the density of grain boundaries in this material. The broadening of the DOS at high energies can be changed independently of the grain size. We present evidence that this broadening is sensitive to the internal structure of grain boundaries. These two alterations of the phonon DOS change considerably the vibrational entropy of nanocrystalline materials, although they do so in opposing ways.

2:30 PM INVITED PAPER
FIBER-TYPE MMCS FOR ELECTRONICS THERMAL MANAGEMENT: *S. Shiga*[1]; J. Ninomiya[1]; A. Hideno[1]; K. Mihara[1]; Y. Oyama[1]; [1]Furukawa Electric Co., Ltd., Metal Research Center, R&D Div., 500 Kiyotaki, Nikko 321-0942 Japan

High thermal conductivity materials along with tailored coeffecients of thermal expansion (CTE) are still in the developing area of materials and industrial technology. High thermal conductivity ceramics and powder-metallurgy-processed metal matrix composites (MMCs) are both commercially used. They are mostly non-directional in thermal performance. Two types of fiber-containing MMCs (FRM) were developed for industrial applications. Their usefulness was demonstrated in terms of the nature of the FRMs; 1) High thermal conductivity carbon fiber/Al or Cu composites exhibit the maximized thermal performance in one-/or two-dimensional orientation along the fiber length. In the case of the two-dimensional orientation, the conductivity in the x-y plane doubles that in the y-axis while keeping the CTE tailored. 2) Cr-fiber/Cu MMCs in which fine Cr-fibers are in-situ incorporated in high-purity Cu matrix, show thermal conductivity values comparable to that of Cu, and CTE values comparable to those of glass ceramics and plastic packaging materials. The thermal properties of these composites appear to be independent of fiber directionality. The material property characterization and some examples of applications will be presented.

3:00 PM INVITED PAPER
CONTROL OF THERMAL EXPANSION BEHAVIOR IN ELECTRONIC MATERIALS: *S. Jin*[1]; H. Mavoori[1]; [1]Bell Laboratories, Lucent Technologies, 600 Mountain Ave., Murray Hill, NJ 07974 USA

Thermal expansion behavior of materials, especially the mismatch in the coeffecient of thermal expansion (CTE) among various component materials in electronic devices and packages, is an important issue in terms of device performance and reliability. The CTE is generally considered to be an intrinsic property of materials. However, there are some novel ways of modifying the thermal expansion behavior, e.g., so as to obtain very small, very large, near-zero, or negative CTE values by utilizing magnetic transition or phase transition near room temperature. Some examples of CTE control in electronic materials and composite structures, as well as their potential device applications will be discussed.

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3:50 PM INVITED PAPER
INCREASING THE THERMAL CONDUCTIVITY OF BORON NITRIDE AND ALUMINUM NITRIDE PARTICLE EPOXY-MATRIX COMPOSITES BY PARTICLE SURFACE TREATMENTS: Yunsheng Xu[1]; *Deborah D. L. Chung*[1]; [1]State University of New York at Buffalo, Dept. of Mech. & Aero. Engr., 608 Furnas Hall, Amherst, NY 14260 USA

The thermal conductivity of boron nitride and aluminum nitride particle epoxy-matrix composites was increased by up to 97% by surface treatment of the particles prior to composite fabrication. The increase in thermal conductivity is due to decrease in the filler-matrix thermal contact resistance through the improvement of the interface between matrix and particles. Effective treatments for BN involved acetone, acids (nitric and sulfuric) and silane. The most effective treatment involved silane such that the coating resulted from the treatment amounted to 2.4% of the weight of the treated BN; less coating was less effective. The effectiveness of a treatment was higher for a larger BN volume fraction. At 57 vol.%, the thermal conductivity reached 10.3 W/m.C. The treatment had little effect on the specific surface area of the BN particles. Silane treatments were also effective for AlN. At 60 vol.% AlN, the thermal conductivity reached 11 W/m.c.

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THERMAL CONTACT CONDUCTANCE STUDIED BY TRANSIENT LASER FLASH METHOD: Xiangcheng Luo[2]; *Deborah D. L. Chung*[1]; [1]State University of New York at Buffalo, Dept. of Mech. & Aero. Engr., 608 Furnas Hall, Amherst, NY 14260 USA

The thermal contact conductance between two copper disks as well as between copper and silver epoxy was studied by transient laser flash method. The parameters, which have influence on the thermal contact conductance between copper disks, include compressive pressure applied between copper disks, surface roughness and surface treatment of copper disk surfaces. The higher the contact pressure, the higher thermal contact conductance and furthermore, the contact conductance between two copper disks with rough surface increases more rapidly with the increase of contact pressure than the one with smooth surfaces. A heat sink compound greatly increases the thermal contact conductance between copper disks. The thermal contact conductance between copper and silver epoxy can also be increased by acid washing of copper sur-

faces. Finite element program ABAQUS was used to calculate the thermal contact conductances through time vs. temperature curves which were obtained by experiment.

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THERMAL AND OPTICAL PROPERTIES OF POROUS ALUMINUM OXIDE: *Dennis W. Readey*[1]; Jesus Vazquez[1]; [1]Colorado School of Mines, Dept. of Metall., 1500 Illinois Ave., Golden, CO 80401 USA

The optical reflectivity of ceramic powders depends on the number of scattering interfaces per unit thickness and the optical absorption of the material itself. As sintering occurs, necks between particles reduce the reflective area between particles and, hence, the reflectivity. In contrast, the thermal conductivity of porous ceramics should increase as the interparticle contact area increases during sintering. Models and experiments of both optical reflectivity and thermal conductivity of Al2O3 are compared. Vapor phase sintering in HCl is used to produce increased grain size and interparticle contract without increasing density.

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ALUMINUM NITRIDE WITH HIGH THERMAL CONDUCTIVITY FROM ALUMINUM METAL COMPACTS: *T. Okada*[1]; M. Toriyama[2]; S. Kanzaki[2]; [1]Fine Ceramics Research Association, Nagoya 462-8510 Japan; [2]National Industrial Research Institute of Nagoya, Nagoya 462-8510 Japan

Aluminum nitride (AlN) with high thermal conductivity was synthesized by direct nitridation of a mixture of aluminum (Al) powder with 5 wt% of yttria, and followed by sintering at 2173K. To prevent changes in the compacts shape, before and after processing, nitridation was performed at nominal temperatures below the melting point of Al and nitrogen pressures 0.5-7 MPa. After nitridation, the generated AlN compacts reached 75-80% of the theoretical density. Densified over 95% of the theoretical density was achieved by sintering at 2173K for 4h in nitrogen atmosphere. The thermal conductivity of 170 W/mK was achieved by controlling the nitridation condition. The role of final oxygen concentration and nitridation conditions on the thermal performance is discussed.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Melting and Refining Processes

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

Wednesday PM Room: 2
March 3, 1999 Location: Convention Center

Session Chairs: David G.C. Robertson, University of Missouri-Rolla, Rolla, MO 65409-1460 USA; R. L. Guthrie, McGill University, Montreal, Quebec H3A 2A7 Canada

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SIMULATION OF IMPINGING JET APPARATUS FOR GAS-LIQUID REACTION RATE MEASUREMENT: *M. Philip Schwarz*[1]; [1]CSIRO Minerals, P.O. Box 312, Clayton South, Vic 3169 Australia

The rate of reaction of oxidizing gases with iron/carbon melts is of importance in direct ferrous smelting because the extent of back-reaction must be minimized in these processes. Several researchers, including those at Carnegie Mellon University (CMU), have measured rates for CO2 and H2O with an iron/carbon/sulphur melt using a small experimental reactor in which the gas jet impinges on the melt. Some attempts have been made to perform such an experiment with oxygen, but in this case it may not be possible to operate in the chemical kinetics limited regime because of the high rate of reaction of O2 at the surface. The interpretation of rate measurements in such cases could be assisted by Computational Fluid Dynamics (CFD) simulation in which both gas side mass transfer and chemical kinetics are taken into account. A CFD model for the gas-side of an impinging jet iron bath reactor has been developed to assist in the interpretation of such experiments and in their design. Heat transfer and reaction at the surface have been included. A free surface model, which solves for the liquid motion within the bath as well as the gas motion above the bath, has also been developed. This model also predicts extent and shape of the dimple formed on the liquid surface by the impinging jet. Both models have been run for conditions typical of jet impingement experiments using CO2, H2O and O2. The main conclusions are results from the model for gas-side mass transport are in good agreement with the empirical correlation of Belton and Belton (1980)· Experimentally determined rates of decarburization by CO2 should be corrected for mass transfer because the experiments are often carried out under mixed rate control, eg for gas flow rate 10 L/min when the sulphur concentration is lower than 0.045%. If the rate constant for oxygen reaction is greater than about 5 mol/m2.s.atm, it cannot be determined using a gas flow rate of 10 L/min or lower because of mass transfer control. A cavity of depth 4.5 mm is predicted for a gas flow rate of 10 L/min and this substantially changes the flow field compared with a simulation in which the surface is assumed to be flat. The predicted effective reaction rate can be much lower than expected when the cavity is so deep that the flow detaches from the bath surface at the lip of the cavity. This effect, if it actually occurs in the experiments, could bias measured rates.

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COMPARATIVE STUDY OF THREE CFD CODES IN MODELLING TWO-PHASE FLOW IN LADLE SYSTEMS: *J. Pitkala*[1]; J. L. Xia[1]; J. Vaarno[1]; [1]Helsinki University of Technology, Laboratory of Materials Processing and Powder Metallurgy, PB 6200, FIN-02015, HUT Finland

The present paper is to make a comparative study of three CFD codes in modelling the two phase flow in a water model ladle with central gas injection. The codes examined are CFX, Phoenics and Fluent. A Eulerian two phase model is used. Predictions are compared with experimental data available. Results show that the three codes can reasonably predict the gas-liquid plume in the ladle, and predictions are generally in a good agreement with experimental data. But differences do exist in predicting the gas volume fraction and velocity profiles. It appears that CFX gives the best prediction for the case considered.

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MODELLING THE MIXING OF LEAD BULLION DURING THE REFINING PROCESS.: *C. Bailey*[1]; K. Suman[1]; M. Patel[1]; T. Piper[2]; R. Forsdick[2]; [1]University of Greenwich, School of Computing and Mathematical Sciences, Wellington St., Woodwich, London SE18 6PF UK; [2]Britannia Refined Metals Ltd., Gravesend England

Lead bullion is mixed in kettles to remove a number of impurities including copper, silver, bismuth, etc. During the copper drossing process, lead ingots are melted and then mixed and cooled to a temperature where the copper plus a quantity of lead appears in a solid form (dross) at the liquid surface. Mixing is carried out using specific impeller designs, which form a vortex that ensures greater mixing of the dross. This results in finer dross, with a higher copper content, at the surface. To optimise this process it is important that vortex break-up does not occur at the impeller blades as this will oxidise the lead. This paper will present both water modelling (experimental) and computational fluid dynamic (mathematical) techniques currently being used to help understand this process. Validation of the mathematical models with water models and real plant data will also be presented.

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MELT REFINING: CFD MODELLING OF PARTICLE DEPOSITION TO GAS BUBBLES: *Knut Halvard Bech*[1]; *Stein Tore Johansen*[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2b, Trondheim N-7034 Norway

Removal of inclusion particles from melted aluminium can be facititated by means of a rotor system that supplies kinetic energy and gas to the floating metal. The surface of the gas bubbles collect particles as the bubbles rise from the point of gas injection to the surface, where the slagg can be removed. When applying computational fluid dymanics (CFD) to model such a system, one experiences difficulties with the large range of turbulent length-scales encoutered, from the large geometrical scales of the rotor and container, through the bubble size and down to the particle diameter. A mathematical model for the complete system is complex and require an extreme spatial resolution. Another way of dealing with the problem is to model the microscale phenomena separately and apply the results to create a model for the macroscopic system. The present work deals with turbulent particle flotation to idealized bubbles. Of the total turbulent kinetic energy generated by the rotor's work on the floating metal, only a fraction may contribute to enhanced flotation. By using a model spectrum for the turbulence, the fraction of turbulent energy in eddies smaller than the bubble diameter is calculated. A two-dimensional CFD model was then constructed to calculate the collision efficiency between particles and bubbles of various sizes. The results from the CFD parametric study were processed using multivariate analysis. The first principal component of the data set turned out to be the turbulent intensity, which is the ratio between the root-mean-square turbulent velocity and the rise velocity of the bubble. Another important finding was the if the bubble Reynolds number was too low, the collision efficiency decreased drastically. The bubble Reynolds number is proportional to the bubble size, which again is governed by the stirring power supplied to the rotor.

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THERMOPHYSICAL PROPERTIES FOR THE MODELLING FLUID AND HEAT FLOW IN HIGH TEMPERATURE PROCESSES: P. A. Day[1]; R. F. Brooks[1]; K. C. Mills[1]; *P. N. Quested*[1]; [1]National Physical Laboratory, Centre for Materials Measurement and Technology, Teddington, Middlesex TW11 0LW UK

Methods have been developed to provide reliable property data such as viscosity, density and surface tension for liquid commercial alloys required to describe fluid flow in high temperature processes. For viscosity, an oscillating viscometer was developed with a maximum temperature capability of 1650 degrees centigrade. Electromagnetic levitation techniques with a potential maximum temperature of 2000 degrees centigrade were developed for the measurement of density and surface tension. Changes in the frequency spectra during surface tension experiments will be discussed and are thought to be caused by the accumulation of oxide on the surface of the drop. Models have been applied to estimate these properties (and heat capacity and thermal conductivity) from the chemical composition of the alloy. The predicted results are compared with the experimental data.

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SURFACE PHENOMENA DURING SULPHUR INTERPHASE TRANSFER SLAG TO METAL: *Francisco Anastácio de Oliveira Neto*[1]; [1]Escola Politecnica da Universidade Federal da Bahia, Dept. Engenharia Hidraulica e Saneamento, Rua Aristides Novís, 2, Federação, Salvador 40210-630 Brazil

Interphase dispersion have been observed in presence of sulphur interphase transfer slag to metal at 1600ϒC. Possible mechanism given rise to these phenomena are analysed and discussed based upon recent published results of calcium aluminosilicate slag surface tension. Further insight into surface phenomena involving such melts during interphase mass transfer are highlighted. The results suggest a sulphur exchange mechanism which generates gas at the slag-metal interface and may also involve reduction of alumina. The reaction results in bubble nucleation at the slag-metal interface which leads to simultaneously dispersion of fine metal fragments into the slag by a fluid drag mechanism. It is possible that this effect will increase the overall reaction rates by increasing the effective interfacial area.

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ENTRAINMENT OF INCLUSIONS FROM THE DROSS IN STIRRED REACTORS FOR MELT TREATMENT: *Stein Tore Johansen*[1]; Svend Graadahl[1]; Thomas F. Hagelien[1]; [1]SINTEF, Materials Technology, Alfred Getz vei 2B, Trondheim, Trøndelag 7034 Norway

The dross appearing at the surface of an aluminium melt consists, in addition to aluminium, of mainly oxide films but also other non metallic inclusions. The inclusion particles are often poorly wetted by the metal and accumulate at the metal surface. However, in impeller stirred reactors for melt refining stirring may be exceedingly strong and inclusions accumulated at the metal surface may be entrained into the bulk metal. Concerning inclusion removal, such effects may deteriorate the effectiveness of melt refining. In this paper we investigate the surface entrainment of buoyant particles in a water model of an impeller stirred refining reactor. Under varying operating conditions the particle concentration in the bulk liquid is measured by laser light attenuation. The observed entrainment of particles is explained theoretically and metallurgical consequences of the findings are discussed.

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EFFECT OF FLUID FLOW ON INCLUSION COARSENING IN LOW ALLOY STEEL WELDS: *S. S. Babu*[1]; S. A. David[1]; T. Hong[2]; T. DebRoy[2]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; [2]Pennsylvania State University, University Park, PA 16802 USA

Oxide inclusion forms in welds due to deoxidation reaction in the liquid steel. The inclusions control the weld microstructure development. Thermodynamic and kinetic calculation of oxidation reaction can describe inclusion characteristics such as number density, size, and composition. Experimental work has shown that fluid flow gradients in liquid weld pool can accelerate inclusion growth by collision and coalescence. Moreover, fluid flow in welds can transport inclusions to different temperature regions that may lead to repeated dissolution and growth of inclusions. The above phenomena are being studied with the help of computational coupled heat transfer - fluid flow - thermodynamic - kinetic models. The results show that the inclusion formation in steel welds can be described as a function of welding process, process parameters, and steel composition.

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A MATHEMATICAL MODEL FOR ESTIMATING THE EFFICIENCY OF CERAMIC FOAM FILTERS: F. A. Acosta-Gonzalez[1]; *A. H. Castillejos E.*[1]; [1]Centro De Investigacion y Estudios Avanzados del IPN, Unidad Saltillo, Apdo. Postal 663, Saltillo, Coahuila 25000 Mexico

Filtration as a terminal refining step for metals is widely used, particularly for the casting of aluminum alloys, but still more knowledge is required to understand how several factors affect the efficiency of removal of inclusions. In this study, both, initial and long term filtration efficiencies of ceramic foam filters have been comuted from the numerical solution of the two-dimensional Navier-Stokes equation and the motion equation for the solid particles, entering the pores of the filter. The complex structure of the foam filters is represented by a unit cell which is formed by a pair of pores with average structural parameters. The inlet boundary condition was defined from a physical model of the filter, which has similarity in Reynolds and Gravitational numbers. The fluid velocity measurements were done using particle image velocimetry (PIV); the measured and computed fluid flow fields showed good agreement. Also it was found, that the filtration efficiency depends strongly on the inclusions trajectory which is affected by the Reynolds, Gravitational and the Aspect Ratio numbers. High Gravitational numbers favors the collision of the particles with the wall increasing filtration efficiency. On the other hand, for low Gravitational numbers the particles follow the streamlines, avoiding contact with the wall and decreasing the filtration efficiency. Computed filtration efficiency values agree well with experimental measurements reported in the literature.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Solidification and Casting: Flow Processes

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

Program Organizers: Nagy El-Kaddah, University of Alabama, Dept. of Met. & Mats. Eng., Tuscaloosa, AL 35487-0202 USA; Stein Tore Johansen, SINTEF Materials Technology, Dept. of Proc. Metall.& 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

Wednesday PM Room: 5B
March 3, 1999 Location: Convention Center

Session Chairs: Chris Bailey, University of Greenwich, Center of Numerical Modeling and Process Analysis, Woolwich, London SE18 6pf UK; Haavard Thevik, SINTEF, Mats. Tech., Trondheim N-7034 Norway

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DYNAMIC BEHAVIOUR AND CONTROL OF MOLTEN METAL FLOW IN A COOLED PIPE: A. R. Firth[1]; *N. B. Gray*[1]; A. K. Kyllo[1]; B. N. McCurry[1]; [1]The University of Melbourne, Dept. of Chem. Eng., Parkville, VIC 3052 Australia

Dynamic control of the formation of a solidified layer between a pipe wall and a flowing melt enables the flow rate to be adjusted in real time. This solidified layer can be generated by using a coolant on the outside of a pipe wall to remove the required heat. The coolant and hence the solidified layer can be controlled remotely. The dynamic behaviour of the solidified layer is being investigated by both physical experiments and mathematical modelling. The experiments are being carried out using molten tin in a recirculating circuit and a cooling system that is instrumented for accurate computer control. The circuit is designed for flowrates up to 24kg/s (86 tph) of molten tin. A mathematical model has been developed using a finite difference technique to describe conduction through the pipe wall and solidified layer. A pseudo-first order time constant of 120s for a step change in an input variable is predicted by the mathematical model.

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MODELING HEAT TRANSFER AND FLUID FLOW IN GTA WELDING OF GAMMA TITANIUM ALUMINIDES: *Mario Arenas*[1]; Viola L. Acoff[1]; Nagy El-Kaddah[1]; [1]The University of Alabama, Metall. and Mats. Dept., P.O. Box 870202, Tuscaloosa, AL 35487 USA

A computational procedure to calculate heat and fluid flow in a stationary GTA weld pool is presented. Heat transfer and fluid flow in the molten pool can significantly influence such factors as the weld pool geometry, temperature gradients, local cooling rates and the solidification structure. The importance of liquid motion in a weld pool has been widely recognized recently. The present mathematical formulation considers buoyancy, electromagnetic, and surface tension as driving forces for the fluid motion. The problem is defined to be axially symmetric and transient to a steady state limit. The molten surface is flat. All thermophysical properties are constant. The numerical model was applied to a gamma titanium aluminide intermetallic alloy.

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FINITE ELEMENT MODELING OF MAGNETICALLY-DAMPED 3-D CONVECTION DURING SOLIDIFICATION: *Ben Q. Li*[1]; H. C. De Groh[2]; [1]Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99163 USA; [2]NASA Lewis Research Center, Cleveland, OH 44135 USA

A fully 3-D numerical model is developed to represent fluid instability and magnetic damping of complex fluid flow, heat transfer and electromagnetic field distributions in a melt cavity. The model is developed based on our in-house finite element code for the fluid flow, heat transfer and electromagnetic field calculations. The numerical model is tested against numerical and experimental results for water reported in literature. Various numerical simulations are carried out for the Sn-35.5% Pb melt convection and temperature distribution in a cylindrical cavity with and without the presence of a transverse magnetic field. Numerical results show that magnetic damping can be effectively applied to reduce turbulence and flow levels in the melt undergoing solidification and over a certain threshold value a higher magnetic field resulted in a higher velocity reduction. Also, a fully 3-D representation of the magnetic damping effects, the electric field induced in the melt by the applied DC magnetic field does not vanish, as some researchers suggested, and must be included even for molten metal and semiconductors. Furthermore, for the study of flow instability, a long enough time has to be applied to ensure the final fluid flow recirculation pattern.

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MODELING OF THE OXIDE FILM MOVEMENT, BREAKUP, AND ENTRAPMENT IN ALUMINUM CASTINGS: J. Lin[1]; *M. A. Sharif*[1]; J. L. Hill[1]; [1]The University of Alabama, Aerospace Engineering, P.O. Box 870280, Tuscaloosa, AL 35487-0280 USA

A numerical algorithm has been developed to simulate the movement, breakup, and entrapment of the oxide film inclusions encountered during the mold-filling processes of aluminum castings. The flowfield is solved using the well-known Marker and Cell (MAC) method by a time marching process. The Volume of Fraction (VOF) method is used to track the free surface boundaries. A kinematic approach is employed to track the movement and breakup of the oxide films on the free surface or in the bulk liquid metal. A series of computer simulations of two-dimensional mold-filling have been carried out. The computer program based on the proposed algorithm is able to model the flow behavior and oxide film movement, breakup, and entrapment in mold cavities for aluminum castings. The predictions are in good agreement with the experiments of other researchers.

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EXPERIMENTS AND COMPUTATIONS ON MOULD FILLING OF HORIZONTAL THIN-WALL CASTINGS.: *Robert van Tol*[1]; Laurens Katgerman[1]; Harrie Vanden Akker[2]; [1]Delft University of Technology, Laboratory of Metallurgy, Rotterdamseweg 137, Delft, Zuid-Holland 2628 AL The Netherlands; [2]Delft University of Technology, Kramers Laboratorium, Prins Bernhardlaan 6, Delft, Zuid Holland 2628 BW The Netherlands

Mould fillings of horizontal thin-wall castings have been captured using video-taping, contact measurements and thermo-couples. Additional to these measurements in actual castings, the integral velocity field has been measured in a water model, using particle image velocimetry (PIV). Together, these techniques reveal the displacement of the free surface, local temperatures and the velocity field during mould filling. Computer simulations have been performed, incorporating free surface flow with surface tension, heat transfer and solidification. A penetration theory based heat transfer coefficient at the metal mould interface was used, to prevent cell size dependence of the heat transferred on a mould filling time scale. Computational results show free surface shapes and velocity fields similar to experiments. Cold runs during mould filling can be predicted, using a criterion for the pressure at the ingot.

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AN INVESTIGATION OF HEAT TRANSFER RATES FOR MOLTEN DROPLETS FALLING IN A STAGNANT GAS: *J. Barry Wiskel*[1]; *Hani Henein*[1]; [1]University of Alberta, AM&PL, 536 Chemical-Mineral Bldg., Edmonton, Alberta T6G 2G6 Canada

A study was conducted to establish the effective heat transfer coefficient for a single molten droplet moving in a gaseous medium. Towards

this objective, a series of quench experiments were performed on molten droplets of AA6061 aluminum generated with the Impulse Atomization technique (IAP). IAP is a single fluid atomization process capable of producing powders with a predictable mean particle size and a relatively tight standard deviation under controlled atmospheric conditions. Microstructural analysis of the atomized powder was used to establish the extent of pre-quench solidification. A mathematical model was then employed to correlate the Ranz-Marshall and Whitaker equations with the observed particle cooling behaviour. These equations were found to provide a reasonable estimation of the heat transfer conditions only when the variation in gas thermophysical properties across the boundary layer were accounted for in the model. This model formulation of droplet heat transfer has important implications with regards to active spray and atomization processing operations.

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LIQUID PERMEABILITY MEASUREMENTS IN SOLIDIFYING ALUMINUM ALLOYS: A. J. Duncan[1]; Q. Han[1]; *S. Viswanathan*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Bldg. 4508, MS 6083, Oak Ridge, TN 37831-6083 USA

Measurements of liquid permeability in the mushy zones of Al-Cu and Al-Si alloy samples have been performed isothermally just above the eutectic temperature, using eutectic liquid as the fluid. A modified method has been developed to determine the specific permeability, K_S, as a function of time during the test from the data collected on these alloys. Factors affecting permeability measurements are discussed. Permeabilties are observed to vary throughout the experiment. This is attributed to microstructural coarsening and channeling that occurs in the sample during the experiment. The permeability is related to the microstructure of the sample using the Kozeny-Carman equation. The correlation between the measured K_S, liquid fraction, g_L and the specific solid surface area, S_V, improves markedly when compared to results from previous studies in which microstructural coarsening was ignored.

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THE ESTIMATION OF THERMAL RESISTANCE AT VARIOUS INTERFACES: *Stavros A. Argyropoulos*[1]; Norman J. Goudie[2]; Michael Trovant[3]; [1]University of Toronto, Dept. of Metall. and Mats. Sci., Walberg Bldg., Room 142, 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Bethlehem Steel Corporation, Home Research Laboratories, Bethlehem, PA 18016 USA; [3]Hatch Associates Ltd., Mississauga, Ontario L5K 2R7 Canada

Experimental and computational techniques were devised for the estimation of thermal resistance at various interfaces. Interfaces such as metal-metal, metal-oxide, and metal-mold were studied. For the first two interfaces, the experimental approach involved dipping a cold cylindrical metal into a liquid metal or liquid oxide, respectively. In this case, a shell freezes around the cylindrical addition and a thermal resistance develops during the existence of the shell. Data collected from a series of experiments were used as input into a model, which solves the inverse heat conduction problem in terms of a resistance estimate. Results for a variety of metal-metal and metal-oxide combinations indicated a relationship between the estimated and the mismatch of thermal expansion coefficients and thermal conductivities of interface materials. For the last interface metal-mold, the air gap size was measured and subsequently correlated with the interface thermal resistance. Various correlations will be presented, which predict the interfacial thermal resistance from the knowledge of the air gap at the metal-mold interface. Finally, some general guidelines will be presented on the values of thermal resistance at various interfaces.

5:00 PM

VISCOSITY MEASUREMENT OF ALUMINUM ALLOY CONTAINING SiC PARTICULATES: *Zhijing Zhang*[1]; Ramana G. Reddy[1]; Srinath Viswanathan[2]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., A129 Bevill Bldg., 126 Seventh Ave., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6083 USA

The viscosity measurements were carried out on melts of 380 aluminum alloy containing 20 volume % SiC particulates under various shear rates at different temperatures using Brookfield viscometer. Results showed that viscosities of the melts increased with decreasing temperature and decreased with increasing shear rate. These results were compared with those of 356 alloy and it was found that the temperature had a more significant effect on viscosity of 356 alloy than that of 380 alloy containing SiC particulates. Based on the present experimental data an empirical viscosity model, which considers the effects of both solid fractions in the melt and shear rate, was proposed.

FUNDAMENTALS OF LEAD AND ZINC EXTRACTION AND RECYCLING: Pyroprocessing of Lead and Zinc—Primary Operations

Sponsored by: Extraction & Processing Division, Lead, Zinc, and Tin Committee

Program Organizers: A. Morris, San Diego, CA 92128 USA; Markus Reuter, Delft University of Technology, Netherlands

Wednesday PM Room: 7A
March 3, 1999 Location: Convention Center

Session Chairs: Arthur E. Morris, Thermart Software, San Diego, CA 92128-2720 USA; David G. Robertson, University of Missouri-Rolla, Dept. of Metallurgical Engineering, Rolla, MO 65409-1460 USA

2:00 PM

DEBISMUTHISING OF LEAD BY CENTRIFUGING: A. Arnold[1]; H. Sasker[2]; *Markus A. Reuter*[2]; J. Kruger[1]; [1]Aachen University of Technology, Institute for Nonferrous Process Metallurgy, Intzestrasse 3, Aachen Germany; [2]Technical University Delft, Raw Materials Processing, 120 Mijnbouwstraat, Delft 2628 RX The Netherlands

One method for removing bismuth during the refining of lead is the Kroll-Betterton process. In this final step of lead refining a calcium magnesium alloy is stirred into the lead creating compounds such as Bi2CaMg2, that can be skimmed off from the surface of the lead. This paper discusses methods to improve the recovery of bismuth and decrease the usage of magnesium and calcium as a "reagent" by presenting results on the following aspects: a fundamental thermodynamic study of the system to determine the conditions at which all possible compounds of Bi, Ca and Mg are created in the Pb - Bi - Mg - Ca system; for each of these compounds equilibrium constants and the theoretical stoichiometric quantity of Ca and Mg are determined; discuss the application of an experimental centrifuge to facilitate the bismuth refining; and apply the obtained fundamental information mentioned above to run tests in the above mentioned centrifuge to optimally recover bismuth from lead.

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FLASH ROASTING OF ZINC CONCENTRATES AND LEACH RESIDUES USING A TORBED REACTOR: *Chris E. Dodson*[1]; [1]Torftech (Canada) Inc., 2395 Speakman Drive, Mississauga, Ontario L5K 1B3 Canada

Conventionally hydrometallurgical zinc recovery processes from sulphide concentrates include roasting, leaching, precipitation, impurity removal and electrowinning. The use of the novel TORBED process reactor for the flash roasting of zinc sulphides will be proposed. The advantages in its application will be discussed including fine particle processing capability, lower cost smaller roasters, waste minimization, improved leaching efficiency and overall process simplification. Results from the ongoing pilot trials will be given.

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THE BEHAVIOUR OF GALLIUM DUIRNG JAROSITE PRECIPITATION: *J.E. Dutrizac*[1]; [1]CANMET, 555 Booth Street, Ottawa, K1A 0G1Canada

The behaviour and deportment of gallium during the precipitation of jarosite-type compounds from $Fe(SO_4)_{1.5}$ - M_2SO_4 (where M is Na, K or NH_4) solutions were investigated. Gallium is readily precipitated n all the jarosite-type compounds, and the extent of gallium precipitation increases as the Ga concentration of the solution increases. The Ga^{3+} ion replaces Fe^{3+} in the jarosite structure to form a nearly ideal solid solution series. The molar partitioning coefficient, $(Ga/Ga+Fe)_{solid}$ $(Ga/Ga+Fe)_{solutions}$ 1,0 for the three jarosite species studied. Increasing retention times significantly increase the amount of precipitate formed, but have only a minor effect on the composition of the jarosite products. Increasing concentrations of $Fe(SO_4)_{1.5}$, in the presence of a constant $Ga(SO_4)_{1.5}$ concentration, proportionally increase the amount of product, and result in a significant reduction of its Ga content. In contrast, increasing $ZnSO_4$ concentrations have a negligible effect on the amount of product formed or its Ga content. A comparison with previously published results for indium, shows that both Ga ad In behave very similarly during the precipitation of jarosite-type compounds.

3:00 PM BREAK

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OPTIMISATION OF A TIN-LEAD-SMELTING FURNACE WITH THE AID OF DATA RECONCILIATION MODELLING TECHNIQUES: *S. C. Grund*[1]; *Markus A. Reuter*[2]; R. Janssen[2]; A. Nolte[3]; [1]Metallurgical Consultant, Alter Postweg 12, Dorsten 46282 Germany; [2]Technical University Delft, Raw Materials Processing, 120 Mijnbouwstraat, Delft 2628 RX The Netherlands; [3]Hüttenwerke Kayser AG, Kupferstrasse 23, Lunen D-44532 Germany

In secondary copper smelting as it is performed at Hüttenwerke Kayser in Lünen (Germany), zinc-, tin- and lead-containing flue dusts are produced as intermediate products. These flue dusts are further processed in a hearth furnace. Followed by certain refining operations a saleable tin-lead-alloy as well as a saleable zinc flue dust are produced. Even though the hearth furnace technology has been in use for this purpose for a long time, its performance is constantly challenged by the increasing complexity of the raw materials (Zn/Sn/Pb-flue dusts), and tightening economical and ecological boundaries. Therefore, continually optimum processing conditions have to be searched for in a system that is often fundamentally poorly definable. The use of data reconciliation for the calculation of accurate statistical sound mass balances is demonstrated for the tin-lead hearth furnace. These reliable mass balances are the basis for any further modelling of the process with the aim of robust furnace optimisation. Incorporating metallurgical knowledge and experience as well as different mathematical metallurgical modelling techniques, further studies are presented in which the recoveries of valuable and minor elements and their dependency on various process parameters are fundamentally examined. The aim of this modelling is: to optimise the recoveries of the valuable elements as a function of various process parameters (also poorly defined parameters such as feed characteristics, etc.); to minimise the energy consumption and the processing costs; and thus to keep the process competitive in a harsher economical and ecological surrounding. The modelling principles discussed here are transferable to other metallurgical operations and are especially suited for ill-defined pyrometallurgical processes.

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A DIRECT REDUCTION ZINC PROCESS: *Lamar S. Todd*[1]; David G. C. Robertson[2]; Hongjie Li[2]; [1]Process Development Associates, 107 N. Brier Rd., Amherst, NY 14228-3450 USA; [2]University of Missouri-Rolla, Metallurgical Engineering, 215 Fulton Hall, Rolla, MO 65409-1460 USA

The direct production of metal from a sphalerite concentrate is an attractive route for primary zinc. A series of operations and conditions have been selected to efficiently cause the following overall exothermic reaction: $ZnS + O_2 = > Zn + SO_2$. The process uses a continuous circulation of molten copper for material and energy transfer between the unit operations. Sulfide concentrate is reduced with an excess of superheated copper to make a copper alloy and white metal. The alloy is stripped of volatile metals under vacuum and some metals can be separately condensed. The residual copper and the white metal are fed into a copper converter to be blown with oxygen for desulfurizing and the superheated copper is recycled. The paper will include a detailed material flow sheet for the process, discussion of the operating conditions of the individual units, behavior of minor elements, and zinc quality.

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SOLAR THERMOCHEMICAL PRODUCTION OF ZINC: *A. Steinfeld*[1]; P. Haueter[1]; S. Miller[1]; R. Palumbo[1]; A. Weidenkaff[1]; [1]Paul Scherrer Institute, High-Temperature Solar Technology, Villigen PSI CH-5232 Switzerland

The solar thermal production of zinc is considered for the conversion of solar energy into storable and transportable chemical fuels. The ultimate objective is to develop a technically and economically viable technology that can produce solar zinc. The program strategy for achieving such goal involves research in two paths: a direct path via the solar thermal dissociation of ZnO, and an indirect path via the solar carbothermal and CH4-thermal reduction of ZnO. Both paths make use of concentrated solar radiation as the source for high-temperature process heat. The thermal dissociation requires elevated temperatures (above about 2000K) and the development of a novel solar process technology, while the carbothermal reduction requires more moderate temperatures (above about 1300K) and uses a combination of solar and conventional energy technologies. The use of natural gas as the reducing agent combines in a single process the reduction of ZnO and the reforming of CH4 for the co-production of zinc and synthesis gas. The chemical thermodynamics and kinetics for the reactions involved are briefly reviewed. A solar chemical reactor for reducing ZnO with natural gas is also described and recent experimental results of solar tests conducted at PSI solar furnace are presented.

GENERAL ABSTRACTS: Session 8 - Melting, Solidification & Microstructure Characterization

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

Wednesday PM Room: 12
March 3, 1999 Location: Convention Center

Session Chairs: John Silvestri, Allvac, Monroe, NC 28111-0539 USA; Anthony Mulligan,Advanced Ceramics Research, Tuscon, AZ USA

2:00 PM

MELTING OF PB NANOCRYSTALS: Kevin F. Peters[2]; *Jerome B. Cohen*[1]; Yip-Wah Chung[3]; [1]Northwestern University, Robert R. McCormick School of Engineering and Applied Science, 2225 North Campus Dr., MLSB, Room 2036, Evanston, IL 60208-3108 USA; [2]European Synchrotron Radiation Facility, Surface Science Group - ID3, B.P. 220, Grenoble, Cedex F-38043 France; [3]Northwestern University, Department of Materials Science and Engineering, 2225 North Campus Dr., MLSB, Room 2036, Evanston, IL 60208-3108 USA

The size-dependent melting and surface melting in ultra high vacuum has been demonstrated by x ray powder diffraction. Whereas some prior studies have measured the size-dependent melting temperature via the diffraction intensity, it is shown here that crystallite reorientation makes the diffraction intensity an unreliable indicator of melting. Instead the diffraction peak shape reveals the size-dependent melting via changes in the crystallite size distribution. Measurements showed that the melting temperature varies inversely with the crystallite size and quantitatively favors the liquid-skin melting model over the homogeneous melting model. Surface melting is demonstrated via the reversible growth of a

liquid skin just below the size-dependent melting temperature. This research was supported in part by the U. S. Department of Energy, Grant No. DE FG02 84ER45097.

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THE COOLING AND SOLIDIFICATION BEHAVIOR OF ALUMINUM AND ALUMINUM ALLOYS WELD: *Kimioku Asai*[1]; Eisaku Tokuchi[1]; [1]Musashi Institute of Technology, Dept of Mechanical Engineering, 1-28-1 Tamazutumi, Setagaya-ku, Tokyo 158 Japan

Weld solidification has always posed experimental difficulties. Experiments which had not ever been approached in this respect were successfully carried out in the TIG arc spot welding on a thin plate. In this first research, the actual temperature in welding thermal cycle was carefully measured with extremely small CA-thermocouples; a high speed camera was also set for direct observation of the crystal growth, by which operation the initial solidification point on the cooling curves were surely decided. The results were compared to Aluminum, 99.99% purity, and two significantly typical commercial Al-alloys, A2024 and A5083. Our results should be useful in consideration of any aspect of cooling behavior that effectively relate to an occurrence of the welding defects such as porosity or hot cracking. Mainly, the dynamic behaviour of solidification rate, cooling rate, and temperature gradient in weld metal are discussed.

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DEVELOPMENT OF A REFINED MICROSTRUCTURE BY DECOMPOSITION OF A METASTABLE PRECURSOR: *Katherine C. Chen*[1]; Fuming Chu[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory, Materials Science and Technology, Mail Stop G770, Los Alamos, NM 87545 USA

Laves phase (AB_2) intermetallics hold great potential as high-temperature structural materials if their characteristic low-temperature brittleness can be circumvented. A two phase alloy (C15+bcc) in the Nb-Cr-Ti system has been shown to have promising mechanical properties. Development of this alloy has required specific alloy design methodologies, such as alloying and novel processing techniques. While conventional casting often produces large grains with the Laves phase along the grain boundaries, melt-spinning has been utilized to rapidly solidify the alloy into a metastable bcc phase. Upon consolidation (HIP) of these melt-spun ribbons, the metastable alloy decomposes into a desired microstructure with extremely fine phase distributions of the Laves phase. Mechanical properties are expected to improve significantly over the cast material. The microstructural control from this metastable processing route offers an approach to tailor the phase distributions necessary for optimized properties in Laves phase alloys. Characterization by optical microscopy, SEM, TEM, and XRD, as well as results from mechanical testing, will be presented.

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EFFECTS OF ELECTROMAGNETIC VIBRATIONS ON THE STRUCTURE OF Al-Si ALLOYS: *Alireza Radjai*[1]; Kenji Miwa[2]; [1]Japan Science and Technology Corporation, Materials Processing Dept., National Industrial Research Institute of Nagoya, 1-1 Hirate-cho, Kita-ku, Nagoya, Aichi 462 Japan; [2]National Industrial Research Institute of Nagoya, Materials Processing Dept., 1-1 Hirate-cho, Kita-ku, Nagoya, Aichi 462 Japan

Electromagnetic Vibrations induced in a conducting liquid by simultaneous application of alternating electric and stationary magnetic fields may lead into the formation and collapse of cavities, which can affect the solidification structure. In order to investigate this matter, hypo-eutectic and hyper-eutectic Al-Si alloys were subjected to strong electromagnetic vibrations under different cooling conditions in an experimental apparatus designed for this purpose. Profound effects were observed for both alloys. The mechanism of the effects was studied by inducing vibrations in a hyper-eutectic Al-Si alloy containing suspended silicon particles and interrupting the process at different temperatures before and after the start of solidification by water quenching. Photomicrographs clearly revealed that suspended silicon particles were crushed into small pieces by cavitation phenomenon both above and below the liquidus temperature. The crushed particles started to agglomerate just after the start of solidification and expelled to the outer boundaries of the sample as solidification proceeded.

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MICROSTRUCTURAL CHARACTERIZATION OF WASTE FORM INGOTS CAST FROM IRRADIATED MATERIALS: *Dennis D. Keiser*[1]; [1]Argonne National Laboratory, Nuclear Technology, P.O. Box 2528, Idaho Falls, ID 83403-2528 USA

A metallic waste form alloy that consists primarily of stainless steel (SS) and zirconium is being developed by Argonne National Laboratory to contain metallic waste constituents that are residual from an electrometallurgical treatment process for spent nuclear fuel. Actual ingots have been cast in an induction furnace in a hot cell using leftover cladding hulls from an electrorefiner. These ingots have been sampled using a core-drilling and an injection-casting technique. In turn, generated samples have been characterized using chemical analysis techniques and a scanning electron microscope equipped with energy-dispersive and wavelength-dispersive spectrometers. As-cast ingots contain the expected concentration levels of the various constituents, and the phases that develop are very similar to those for alloys generated using non-radioactive surrogates for the various fission products. One exception is the appearance of a small precipitate phase that contains the fission product tellurium.

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TEM CHARACTERIZATION OF THE EFFECT OF MIXED PROTON/NEUTRON FLUX ON MICROSTRUCTURAL EVOLUTION OF STRUCTURAL MATERIALS FOR ACCELERATOR PRODUCTION OF TRITIUM.: *Bulent Hakan Sencer*[1]; Gillian Mary Bond[1]; Frank A. Garner[2]; Stuart A. Maloy[3]; Walter F. Sommer[3]; M. James[3]; [1]New Mexico Tech, Materials and Metallurgical Engineering, Campus Station, Socorro, NM 87801 USA; [2]PNNL, Materials Resources Department, 902 Battelle Blvd., P.O. Box 999, Richland, WA 99352 USA; [3]Los Alamos National Laboratory, APT/TPO, MS H809, Los Alamos, NM USA

Materials in the target and blanket regions of spallation neutron sources (SNS) will be subjected to high fluxes of high-energy protons and neutrons. There are no available data on materials performance under the conditions that are be experienced by Production of Tritium (APT) target and blanket materials. As particle energy increases to above 200 MeV or so, a major new variable becomes important: copious transmutation products are generated and enter the material as impurities. High-energy proton/neutron irradiation is also expected to generate very high levels of helium and hydrogen; for example, high-energy protons produce about 100 times more helium than fourteen MeV neutrons. To investigate the effects of these conditions on the microstructure, TEM specimens of several alloys have been irradiated in the Los Alamos Spallation Radiation Effects Facility (LASREF) at the 800 MeV accelerator. The effects of this simulated APT irradiation on Al6061-T6, SS316L and Inconel 718 are being characterized to give a better understanding of the microstructural evolution and consequent mechanical properties.

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HIGH-RESOLUTION AEM STUDY OF PARAEQUILIBRIUM CEMENTITE PRECIPITATION IN ADVANCED ULTRA-HIGH STRENGTH STEELS: *Gautam Ghosh*[1]; Gregory Bruce Olson[1]; [1]Northwestern University, Materials Science and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

To support quantitative design and to understand secondary hardening behavior of advanced ultra-high strength steels, the precipitation of cementite prior to the precipitation of coherent M_2C phase is investigated using model alloys. The microstructure of cementite is investigated by transmission electron microscopy techniques. The concentration of substitutional alloying elements in cementite are quantified in high-resolution analytical electron microscopy using extraction replica specimens. Quantification of substitutional elements in cementite confirms its paraequilibrium state with ferrite at the very early stage of tempering. The thermodynamics and energetics of paraequilibrium cementite nucleation are analyzed using Thermo-Calc software, and the growth of paraequilibrium cementite is simulated using DICTRA software. These simulations provide further insight of the experimentally

observed microstructures. The implications of the results will be discussed in the context of alloy design.

GENERAL RECYCLING OF MATERIALS: Physical and Hydrometallurgical Processing

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizers: Ilaria Accorsi, Chrysler Corporation, Product Quality, Toledo, OH 43606 USA; Isrun Bohlinger, Technical University of Berlin, Institute of Metallic Materials, Berlin D-10623 Germany

Wednesday PM Room: 1A
March 3, 1999 Location: Convention Center

Session Chairs: Isrun Bohlinger, Technical University of Berlin, Institute of Metallic Materials, Berlin, D-10623 Germany; Andréa Moura Bernardes, Universidade Federal do Rio Grande do Sul, LACOR-DEMAT-UFRGS, Porto Alegre, Rio Grande do Sul (RS) 900035-190 RS Brazil; Courtney A. Young, Montana Tech, Dept. of Metallurgical Engineering, Butte, MT 59701 USA

2:00 PM OPENING REMARKS

2:05 PM
PROCESSING OF DRY CELL BY ORE PROCESSING METHODS: *Jorge Alberto Soares Tenório*[1]; Denise Corrêa de Oliveira[1]; Arthur Pinto Chaves[2]; [1]Escola Politécnica. University of São Paulo, Dept. of Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, Cidade Universitária, São Paulo 05508-900 Brazil; [2]Escola Politécnica da USP, Mining Engineering, Av. Prof. Mello Moraes, 2373, Cidade Universitária, São Paulo 05508-900 Brazil

Household zinc based batteries contain some heavy metals such as zinc, manganese and mercury. There is a large effort from manufacturers in order to eliminate the last one. As a municipal waste, the disposal of batteries has become an increasing worry. The aim of the present work was to characterize dry batteries scraps, defining parameters and yields of each stage. This was done using unit processes of ore treatment. Such procedures have been chosen due to its low cost. The employed methods were hammer mill grinding, size classification, magnetic separation and flotation. The characterization step was done by chemical analysis and x-ray diffraction. After size classification, it was verified that 76.6% of the total amount of zinc was above 1.70mm. This fraction was constituted basically of zinc and some coarse pieces from the steel body, paper and plastics. This fraction was submitted to magnetic separation followed by aqueous flotation. The manganese concentration in the fractions above 6.35 mm was less tha n 2%. This means that there was low contamination by the paste in this fraction. This was due to the entrapment of the paste by scraps from the steel body and zinc cup during grinding.

2:30 PM INVITED PAPER
BEHAVIOR OF FERROMAGNETIC PARTICLES ON THE EDDY CURRENT ROTOR SEPARATING NON-FERROUS SCRAP METAL CONCENTRATE FROM AUTOSHRED: *Adam J. Gesing*[1]; Russel Jahnke[1]; Kevin Dehetre[1]; Richard Griser[1]; Dennis Reno[1]; Richard Wolanski[1]; [1]Huron Valley Steel Corporation, 41000 Huron River Dr., Belleville, MI 48111 USA

An eddy current separator, (ECR) is designed to separate non-magnetic (non-ferrous) electrically conducting (metallic) particles from non-conductive residue. An ECR consists of a rotor - a fast spinning roll surfaced with rows of permanent magnets of alternating polarity. The rotor is housed in a fiber-reinforced composite shell which acts as a conveyor headpulley. The non-ferrous metallic particles are repelled by the rotor out of the material stream passing over a conveyor headpulley effecting the desired metal/non-metal separation. The behavior of ferromagnetic particles - mainly iron and iron oxide - is quite different. These are strongly attracted to the magnets and small particles tend to align in the direction of the magnetic flux. Since the direction of the flux changes with the passage of each magnet pole the small particles spin at very high rates causing wear of the belt and the composite outer shell enclosing the rotor. Large particles can not spin but can be heated by the eddy current effects which can burn through the composite headpulley shell. We discuss practical implications of this behavior of iron and rust on the design, performance and maintenance of eddy current separators.

2:55 PM
WASTE PROCESSING OF MgO BAGHOUSE DUST USING PLASMA ARC TECHNOLOGY: *Steven Wayne White*[1]; Ramana G. Reddy[2]; [1]University of Alabama, Dept. of Chem.l Eng., P.O. Box 870203, Tuscaloosa, AL 35487 USA; [2]University of Alabama, Metallurgical & Materials Engineering, P.O. Box 870202, Tuscaloosa, AL 35487 USA

Plasma arc technology is increasingly being used to process a number of waste materials. In present research, plasma technology is being investigated to treat waste magnesium baghouse dust, which is a waste material from the Magnetherm process. This dust contains mostly MgO, but also contains Na_2O, CaO, and K_2O. Plasma energy is to be used along with a reducing agent to produce pure gaseous magnesium while keeping the other materials in a slag. The magnesium will then be collected by rapid quenching. A non-transferred arc plasma torch is ideal for this reaction because of its high enthalpy and improved energy efficiency over conventional methods. Thermodynamic calculations using the Gibb's Free Energy Minimization method are being employed to determine the most stable compositions of the materials at various temperatures. The reactor setup will eventually allow for treatment of other waste materials.

3:20 PM INVITED PAPER
CONTROL OF THE PROPERTIES OF ANODIC SLIMES IN COPPER ELECTROREFINING: *Gerardo Cifuentes*[1]; S. Hernández[1]; P. Navarro[1]; J. Simpson[1]; C. Reyes[1]; N. Cornejo[2]; [1]Universidad de Santiago de Chile, Departamento de Ingenieria Metalúrgica, Av. Lib. B.O'Higgins 3363, Santiago Chile; [2]Ventana-ENAMI, Superintendencia Refinería, Carretera F30 E Nordm; 58270, Puchuncaví, V Región Chile

This paper presents the relationship between the composition of the cathode and anodic slimes at copper electrorefining's Ventana-ENAMI Company. It is known that the purity of the cathode and the electrolytic treatment affect solids present in the bath. Anodic slimes properties (for example chemical and crystalline composition, sedimentation) were studied. The properties were modified by composition of the anode and current density. For the experiments anode material was doped with different amounts of lead. Appearance of Bindheimite ($Sb_2Pb_2O_7$ was confirmed. The specific gravity of this species is approximately 7.3 - 7.5 (25ºC/25ºC). At these values the most probability of sedimentation is given, at smaller values slimes decrease the sedimentability.

3:45 PM BREAK

4:00 PM INVITED PAPER
THE APPLICATION OF ELECTRODIALYSIS ON THE TREATMENT OF EFFLUENTS WITH HEXAVALENT CHROMIUM: Marco Antonio Siqueira Rodrigues[1]; *Andrea Moura Bernardes*[1]; Jane Zoppas Ferreira[1]; [1]Universidade Federal do Rio Grande do Sul, LACOR-DEMAT-UFRGS, Av. Osvaldo Aranha 99/706, Porto Alegre, Rio Grande do Sul (RS) 90035-190 Brazil

The possibilities for treating rinse waters from chromating bath by electrodialysis are discussed together with the possibilities of optimizing the treatment process for waste reduction and reuse of water and chemical products. An industrial effluent with 4600 ppm of Cr(VI), in addition to other metallic contamination, was used as work solution. This solution has been recirculated on an electrodialysis cell of three compartments, using exchange membranes Selenion AMT and CMT. A migration of dichromate ions into the anodic compartment was obtained, so that an extraction of circa 99.9% of Cr(VI) present on the solution was observed, resulting in water with a Cr(VI) concentration of 4 mg/l.

These results show that electrodialysis can be used to treat these effluents, and this water could be reused as rinse water. The solution of the anodic compartment, consisting at the beginning of the experiment of H_2SO_4, 0.1 N, reached 7200 mg/l of Cr(VI). This concentrate could be reused on the chromating bath, itself.

4:25 PM INVITED PAPER

ELECTROLYTIC OXIDATION OF CYANIDE ON PbO_2-COATED STAINLESS STEEL: *Gerardo Cifuentes*[1]; L. Cifuentes[2]; Roland Kammel[3]; J. Torrealba[1]; [1]Universidad de Santiago de Chile, Departamento de Ingenieria Metalúrgica, Av. Lib. B.O. Higgins 3363, Santiago Chile; [2]Universidad de Santiago de Chile, Departamento de Ingenieria de Minas, Tupper 2069, Santiago Chile; [3]Technische Universität Berlin, Institut für Metallische Werkstoffe, Metallhüttenkunde, Sekr. BH 15, Str. des 17. Juni 135, Berlin D-10623 Germany

Electrolytic oxidation offers an environmental safe way in order to remove cyanides from electroplating waste waters. In this paper process parameters are discussed. Lead(IV)-dioxide coatings have been produced on anodes made of stainless steel 316 and 316I. Precoating of the substrate with Ni allows the deposition of α-Lead(IV) dioxide (orthorhombic) and β-Lead(IV) dioxide (tetragonal), the β-modification being the most abundant. Catalytic surfaces were produced in order to accelerate the oxidation of cyanide. The electrochemical parameters were determined by experiment and simulation. The values obtained for the exchange current density (i_0), the charge transfer coefficient (α), and the limiting current density (i_L) are similar for both methods: i_0 -2 Am^2, α -0.87, and i_L -120 Am^{-2} for a 0.5 g/l solution of sodium cyanide.

4:50 PM INVITED PAPER

CONVERSION AND PURIFICATION OF COPPER ARSENATE PRODUCED FROM LIBERATION CELLS AT COPPER REFINERIES LTD, TOWNSVILLE, AUSTRALIA.: *Stuart Peter Johnston*[1]; [1]Mount Isa Mines, Ltd., Copper Refineries, Ltd., Technical Superintendent, Hunter St., Stuart, Townsville, Queensland 4811 Australia

Copper Refineries Ltd is the largest producer of refined copper cathode in Australia. Feed stocks include 100mtpa arsenic contained in anode material received from Mt Isa. The main recovery of arsenic material is achieved via the copper liberation section prior to nickel sulphate purification. Rather than stockpiling or dumping the impure copper arsenate material, the product is reprocessed, the copper recovered and the arsenic purified into a valuable arsenate byproduct. This product is sold as direct feed stock for the treatment of plantation timbers. The end result, arsenic entering the Copper Refineries flow sheet is managed to produce an environmentally safe, value added and marketable product.

5:15 PM CLOSING REMARKS

INTERCONNECTPACK; INTERCONNECTIONS FOR ELECTRONICS PACKAGING: Conductive Polymer

Sponsored by: Electronic, Magnetic & Photonic Materials Division,
Program Organizers: Gautam Ghosh, Northwestern University, Dept. of Mat. 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

Wednesday PM Room: 17A
March 3, 1999 Location: Convention Center

Session Chairs: E. Suhir, Lucent Technologies; R. A. Fournelle, Marquette University

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2:00 PM INVITED PAPER

Z-AXIS ANISOTROPIC ELECTRICALLY CONDUCTIVE POLYMER-MATRIX COMPOSITE MATERIALS WITH ONE NICKEL PARTICLE PER CONDUCTION PATH: Yunsheng Xu[1]; *Deborah D. L. Chung*[1]; [1]State University of New York at Buffalo, Dept. of Mech. & Aero. Engr., 608 Furnas Hall, Amherst, NY 14260 USA

Z-axis anisotropic electrically conductive polymer-matrix composite materials in adhesive or resilient stand-alone film (for separable interconnections) forms are useful for electronic packaging, as one film can replace an array of soldered joints. We have developed both forms by using nickle particles and a polymer matrix (epoxy for the adhesive form and polyvinylidene fluoride for the film form), such that there is one nickel particle per conduction path. The adhesive form was screen printable. The electrical, mechanical and microstructural characteristics of both forms are addressed.

2:30 PM INVITED PAPER

DEVELOPMENT OF CONDUCTING ADHESIVE MATERIALS FOR MICROELECTRONIC APPLICATIONS: *Sung K. Kang*[1]; S Purushothaman[1]; [1]IBM, T. J. Watson Research Center, P.O. Box 218, Rm. 37-250, Yorktown Heights, NY 10598 USA

Electrically and/or thermally conducting adhesive materials are classified into two categories depending on their conduction modes: isotropic and anisotropic materials. Silver-particle filled epoxy is the most common example of the class of isotropic materials which are conductive in all directions. This material has been long used in the electronic applications as a die-bonding material, where its good thermal conduction rather than its electrical conduction property is utilized. The silver-filled epoxy material has several limitations for high performance electrical interconnections, such as low electrical conductivity, increase in contact resistance during thermal exposure, low joint strength, corrosion issue due to silver migration, difficulty in rework, and so forth. The anisotropic conducting material provides electrical and/or thermal conduction only in one direction. An anisotropic conducting film (ACF) is used for interconnecting TAB mounted chips to a liquid crystal display panel, where fine pitch interconnection and low temperature assembly are required. In this paper, a brief review of the state-of-art conducting adhesive technology is provided. Subsequently, development of new conducting adhesive materials is presented for several different applications, which include high temperature materials for ceramic substrates, and lower temperature materials for organic substrates.

3:00 PM INVITED PAPER

UNDERSTANDING OF ANISOTROPIC CONDUCTIVE FILMS(ACFS)/ADHESIVES(ACAS) FOR PACKAGING APPLICATIONS: *Kyung W. Paik*[1]; Myung J Yim[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. and Eng., 373-1 Yusung-Gu, Gusung-Dong, Taejon, Chung-Nam Korea

Anisotropic conductive films (ACFs) or adhesives (ACAs) composed of an adhesives polymer resin and fine conductive fillers such as metallic particles or metal-coated polymer balls are key materials for fine pitch chip-on-film (COF) and chip-on-glass (COG) LCD packaging and also flip chip on organic board technologies. To understand the efficiency of electrical conduction in ACFs/ACAs, the theoretical electrical conduction model with physical contact mechanism has been simulated and experimentally proved. Three pressure dependent models - 1) elastic deformation Hertz contact model, 2) plastic deformation model and 3) FEM model, were developed. It was shown that the electrical contact resistance of ACF depends on numerous variables such as applied bonding pressure, bonding temperature/time (resin curing characteristics), number, size, mechanical and electrical properties of conducting particles. Electrical conduction through the pressure engaged contact area between conductive particles and conducting substrate pads is the major conduction mechanism in ACFs/ACAs interconnection. The effects of these variables on the conduction will be presented. Environmental effects on contact resistance and adhesion strength of ACFs/ACAs such as thermal aging, high temperature/humidity aging, and temperature cycling were investigated. And for the reparability of ACFs/ACAs interconnect, the adhesion property at various temperatures will be also presented. Finally, as one of the flip chip interconnect alternatives,

issues of using ACFs/ACAs materials for flip chip on organic substrates will be discussed.

3:30 PM BREAK

3:40 PM INVITED PAPER

BARE CHIP INTERCONNECTION USING AN ANISOTROPIC CONDUCTIVE ADHESIVE: *T. Sato*[1]; H. Date[1]; H. Tokuhira[1]; M. Sasaki[1]; E. Horikoshi[1]; M. Kawarada[1]; [1]Fujitsu Laboratories, Ltd., 10-1 Morinosato-Wakamiya, Atsugi 243-0197 Japan

A new anisotropic conductive adhesive, which might be a promising candidate for less 100 μm fine pitch flip-chip face down interconnect, has been developed. This material contains a filler which consists of a silver particle coated with a thin dielectric resin and an epoxy base adhesive. The conductive adhesive shows a very low interconnecting resistance of several milliohms, and a maximum permissible current of 4000 mA. Moreover, when LSIs are mounted on a circuit board, the adhesive shows a high electrical resistance even after a 500 h high-temperature high-humidity bias test (85°C, 85% R.H., and DC 5V). The change in electrical resistance was found to be less than 10% after thermal cycles test of 1000 cycles in the temperature range of -55°C to +125°C. These reliability characteristics would be sufficient for use in most of the microelectronic devices. An example of practical application in a hard disk drive circuit board will be given in this paper. Also, the effects of parameters such as adhesive strength, adhesive layer thickness, deformation behavior of the electrode on the substrate, and quality and quantity of the metal filler on the reliability charateristics will be discussed.

4:10 PM INVITED PAPER

TRANSIENT LIQUID PHASE SINTERING CONDUCTIVE ADHESIVES: POLYMER ADHESIVES WITH METALLURGICAL BONDS: *Catherine Shearer*[1]; Bryan Shearer[1]; Goran Matijasevic[1]; [1]Ormet Corporation, 2236 Rutherford Rd., #109, Carlsbad, CA 92008 USA

Conductive adhesives which have a metallurgically alloyed web with an interpenetrating polymer have been developed to mitigate some of the deficiencies of standard particle-filled conductive adhesives. The metal network is formed in situ by a process known as transient liquid phase sintering (TLPS) and is mutually reinforcing with the polymer network. Bulk as well as interface electrical connections are metallurgically alloyed providing stable electrical and thermal conduction. These new conductive adhesive compositions are compatible with bare copper as well as alloy surface finishes. The TLPS conductive adhesives utilize conventional solder paste dispensing and processing equipment. Electrical conductivity results indicate that the values are close to those of solder alloys. This type of adhesive has been tested for surface mount component attach, in which boards were subjected to drop tests and environmental exposure. All components, including J-lead type, survived this test, as would be expected of typical solder joints. Reliability testing including humidity exposure and thermal cycling has demonstrated that this type of adhesive performs substantially better than standard, passive filler loaded conductive adhesives.

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FRACTURE BEHAVIORS OF LEADFRAME/EPOXY INTERFACE UNDER VARIOUS LOADING CONDITIONS: *H. Y. Lee*[1]; Jin Yu; [1]Korea Advanced Institute of Science and Technology, Dept. of Mats. Sci. and Eng., Taejon 305-701 Korea

Popcorn cracking of thin plastic packages is a serious problem in the microelectronics packaging industry, and ways to improve inherently poor adhesion between leadframe and epoxy is an important issue. Here, copper based leadframes were oxidized in two kinds of hot alkaline solutions, brown and black oxide forming, and molded with epoxy molding compound. Then, the adhesion strengths of the leadframe/epoxy interfaces were measured by using sandwiched double cantilever beam (SDCB), pull-out, and sandwiched Brazil-nut (SBN) specimens. Loading conditions of the SDCB and pull-out specimens were close to the mode I and mode II, respectively, and in-between mode I and mode II for the SBN specimens. After the adhesion measurements, fracture surfaces were analyzed by SEM, XRD and EDX. Results indicates that adhesion strength, in terms of critical energy release rate(Gc)or the pull strength, is directly relate to the formation of acicular CuO precipitates at the interface. However, once a continuous layer of CuO precipitates formed either on the leadframe or underlying Cu2O layer, interface toughness saturated regardless of further oxide thickening. For SBN specimens, Gc increased with phase angle, but kinking of interface cracks into epoxy were observed in a systematic manner. For all cases, size of the acicular precipitates was found to have secondary effects on the toughness.

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INTERFACIAL FRACTURE ENERGY MEASUREMENTS IN THE Cu/Cr/POLYMIDE SYSTEM: *Y. B. Park*[1]; I. S. Park[1]; Jin Yu[1]; [1]Korea Advanced Institute of Sciece, Dept. of Mats. Sci. and Eng., Taejon 305-701 Korea

The interfacial fracture energies(%C) of the Cu/Cr/polyimide system were deduced under varying Cu film thickness and pretreatment conditions based on two methods X-ray measurement by Park and Yu[1] and theoretical methods by Moidu et al.[2]. The two methods showed reasonable agreement for most cases, imparting validity for both approaches. Estimated %C were quite independent of the metal film thickness and increased with the rf plasma power density of polyimide pretreatment as expected. Estimated %C values were 46.8 - 17.8, 170.3 - 42.9 and 253.9 - 44.4 J/m^2 for the rf plasma power density of 0.03, 0.036 and 0.05W/cm2 respectively. To the author's knowledge, this work is the first in the actual estimation of the interfacial energy in the Cu/Cr/polyimide system. Then, well known fracture mechanics solutions for two layer (substrate/thin film) structure were extended to cover the three layer(substrate/interlayer /thin film) structure using the path independence of the J integral. Application of the result to the peel test of Cu/polyimide/alumina system using FEM showed that the phase angle was nearly constant with the film thickness , but using the two layer solution led to a substantially large underestimation of the phase angle. [1] I.S.Park and Jin Yu: Acta Mater. 46, 2947 (1998) [2] A.K.Moidu, A.N.Sinclaire and J.K Spelt: J. Testing Eval. 26,247(1998)

INTERNATIONAL SYMPOSIUM ON ADVANCES IN TWINNING: Transformation Twinning and Mechanisms

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 Laboratory, Mats. Sci. & Tech. Div., Washington, D.C. 20375-5000 USA

Wednesday PM Room: 17B
March 3, 1999 Location: Convention Center

Session Chairs: Manfred Wuttig, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20742-2115 USA; K. T. Ramesh, The Johns Hopkins University, Dept. of Mech. Eng., Baltimore, MD 21218-4316 USA

2:00 PM INVITED PAPER

NANO-AND MESO-TWINNING IN TRANSFORMING ALLOYS: Harsh Deep Chopra[1]; *Manfred Wuttig*[1]; [1]University of Maryland, Materials and Nuclear Engineering, Bldg. 090, Rm. 2135, Stadium Drive, College Park, MD 20742-2115 USA; [1]SUNY at Buffalo, Mech. & Aerospace Eng. Dept. & Center for Advanced Photonic & Electronic Materials, Materials Progam, Room 613, Furnas Hall, Buffalo, NY 14260-4400 USA

This paper covers nano-twinning in In-Tl, In-Cd and Fe-Pd alloys of average fcc high temperature (austenitic) and meso-twinning in In-Tl of fct low temperature (martensitic) symmetry. In the former nano- twinning manifests itself through the low frequency "elastic" shear anisot-

ropy, 2C44/(C11-C12), which approaches a value of one as the temperature is lowered towards the martensitic transformation temperature. Bending experiments of martensitic In-Tl polydomain single crystals in the ferroelastic and rubberlike temperature regimes, accompanied by in situ video recordings of the evolving mesostructure, revealed that the deformation is controlled by the rotation of twin boundaries. The experimental ratios of the twin and elastic modules demonstrate that twins of the second hierarchy control the deform.

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THE SHAPE MEMORY EFFECT IN RUTHENIUM ALLOYS: *Richard W. Fonda*[1]; [1]The Naval Research Laboratory, Physical Metallurgy Branch, Code 6324, Washington, D.C. 20375 USA

Near-equiatomic alloys of niobium-ruthenium and tantalum-ruthenium have recently been shown to exhibit a shape memory behavior at elevated temperatures. These alloys have a B2-ordered cubic structure at high temperatures which transforms during cooling to a highly twinned tetragonal microstructure. Some compositions undergo an additional transformation to a monoclinic structure. The cubic-to-tetragonal transformation, which is responsible for the shape memory effect in these alloys, varies with composition from near room temperature to near 1000ϒC for Nb-Ru and to well over 1000ϒC for Ta-Ru. These are among the highest temperature shape memory transitions yet discovered. This talk will describe the shape memory effect in these alloys and how it relates to the observed transformations and microstructures.

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TWIN INTERSECTIONS WITH GRAIN BOUNDARIES: *Alexander H. King*[1]; [1]State University of New York, Dept. of Mats. Sci. and Eng., Nicolls Rd., Stony Brook, NY 11794-2275 USA

We consider the thermodynamic stability of the junctions between twins and grain boundaries in a formalism that includes anisotropic interfacial energies. General relationships between the interfacial energies and the dihedral angles at twin-grain boundary intersections are derived and we show that there exists a number of special cases. Although the dihedral angles opposite twins are frequently observed to be close to 180 degrees, this angle can only be achieved under very special circumstances: we show that these are not, however, restricted to the case of negligible twin boundary energy. We derive general rules and relationships that allow for grain boundary energy to be measured by using the twin boundary as a "probe" of known energy. Acknowledgment: this work is supported by the NSF, grant number DMR9530314.

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3:45 PM INVITED PAPER

POLYTWIN HETEROSTRUCTURES: *Alexander L. Roytburd*[1]; [1]University of Maryland, Mats. and Nuclear Eng., Bldg. 090, Rm. 1104, College Park, MD 20742 USA

The polytwin heterostructures can be formed as a result of phase transformation or deformation in constrained layers, composed of epitaxial couples or multilayers. Due to the elastic interaction between the layers of a heterostructure, these layers transform into sets of periodically alternating lamellae, or elastic domains. A polydomain layer can consist of either differently oriented domains of the same phase (twins) or domains of different phases. The former case is considered here. The goal of this paper is to determine the parameters of the polytwin heterostructures and the conditions for their formation, i.e., their dependence on the characteristics of phase transformations, lattice misfits and the film thickness, as well as external stress. If the interdomain interfaces are mobile, their movement under external stresses results in superelastic deformation. The giant increase of compliance and susceptibility should be expected at the critical thicknesses of the polytwin layer. These critical thicknesses correspond to loss of stability of the polytwin structure or change of polytwin symmetry. This work was supported by Office of Naval Research under Grant No.: N00014-93-10506 and National Science Foundation under the Grant No.: DMR-9633638.

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COMPETING EFFECTS OF SLIP AND TWINNING ON THE FLOW STRESS OF HADFIELD MANGANESE STEEL SINGLE CRYSTALS: Ibrahim Karaman[1]; Ken Gall[1]; *Huseyin Sehitoglu*[1]; Yuriy I. Chumlyakov[2]; [1]University of Illinois, Mech. & Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Siberian Physical-Technical Institute, Revolution Sq. 1, Tomsk 61801 Russia

A systematic study on Hadfield Manganese steel single crystals (Fe-13wt.%Mn-1.0wt.%C) has been performed to better understand the work hardening mechanisms in polycrystalline Hadfield Steel. The artificially grown single crystals are free of grain boundary interactions, and hence facilitate useful discussions on the deformation mechanisms of fcc alloys with a low stacking fault energy (SFE) and high interstitial content. The main objective of this single crystal study is to examine the dependence of the monotonic tensile and compressive stress-strain behavior on crystal orientation, loading direction, and test temperature. A strong orientation dependence of the governing deformation mechanism, strain hardening coefficient, and extent of different strain stages were detected along the [111], [001], [321] directions. To determine the governing deformation mechanism, the deformed samples were studied with both metallurgical and diffraction techniques. It is shown that twinning is primary deformation mechanism in [111] crystals under tension and in [001] crystals under compression at all test temperatures ranging from 113K to 293K. In the aforementioned crystals, twinning is observed as a softening mechanism and an extended Stage I deformation regime is realized, which is not common for these orientations. During stage II deformation, primary twin-secondary twin and slip-twin interactions play an important role in the strain hardening of these single crystals. In [321] crystals deformed at 293K, after an extended stage I hardening, primary slip-primary twin interactions cause an anomalous stage II hardening. Conversely, the lower stage II hardening coefficient of these crystals at 113K is attributed to the difficulty of cross-slip and easiness of twinning at low temperatures. From these single crystal results, it can be summarized that the competing effects of slip and twinning and the interaction of the two deformation mechanisms make substantial contributions to the work hardening. The increased friction force due to high content of interstitial carbon is proposed to have a contribution to the observed dependence of deformation mechanism along different orientations. The passage of the leading partial dislocation in a low stacking fault energy fcc material transforms the position of the interstitial atom from an octahedral to a tetrahedral site. This transition causes orthorombic distortion providing an extra friction force on the trailing partials, and further increasing the work hardening of this material.

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TWIN COMPONENTS IN RECRYSTALLIZATION TEXTURES OF ROLLED Fe-Ni ALLOYS AND SILVER ELECTRODEPOSITS: Hyo-Tae Jeong[1]; Hyo-Seung NAM[1]; *Dong Nyung Lee*[2]; [1]Seoul National University, Research Center for Thin Film Fabrication and Crystal Growing of Advanced Materials, San 56-1, Shinrimdong, Seoul, Kwanakgu 151-742 Korea; [2]Seoul National University, Materials Science and Engineering, San 56-1, Shinrimdong, Seoul, Kwanakgu 151-742 Korea

The evolution of recrystallization textures from plastically deformed and electrodeposited metals has been relatively well explained based on the energy release maximization theory(ERMT) advanced by one of the present authors(LEE). In the model, the absolute maximum normal stress direction of a deformed or fabricated material becomes parallel to the direction of the minimum elastic modulus of recrystallized grains, whereby the energy release during recrystallization can be maximized. However, the recrystallization textures of cold rolled Fe - 36 to 51% Ni alloys and silver electrodeposits, which have low stacking fault energies, were found to have major orientation components predicted by ERMT and their twin components. The relative intensity of twin component in the recrystallization of the Fe-Ni alloys decreased with increasing Ni content due to the increase in stacking fault energy. Therefore, the twin component cannot be avoided in the recrystallization textures of low stacking fault energy materials.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: New Alloys and Oxidation

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

Wednesday PM Room: 8
March 3, 1999 Location: Convention Center

Session Chairs: Hubert I. Aaronson, Department of Materials Science & Engineering, Carnegie Mellon University, Pittsburgh, PA 15213 USA; Shigeji Taniguchi, Osaka University, Dept. of Mats. Sci. and Proc., Suita, Osaka 565 Japan

2:00 PM INVITED PAPER
DEFORMATION AND FRACTURE OF BI-PST AND TRI-PST CRYSTALS AND DS INGOTS OF TiAl-BASE ALLOYS: Dai Imamura[1]; Hiroaki Hoshikawa[1]; Kyousuke Kishida[1]; Haruyuki Inui[1]; *Masahara Yamaguchi*[1] [1]Kyoto University, Dept. of Mat. Sci. & Eng., Yoshida Honmachi, Sakyo-ku, Kyoto 606-8501 Japan

We have currently been working to develop directional solidification (DS) techniques to grow ingots of TiAl-base alloys composed of many columnar grains with the lamellar structure aligned along the growth direction. One of the major factors controlling the mechanical properties of such DS ingots is expected to be compatibility requirements imposed at grain boundaries of columnar grains since each columnar grain, which is a PST crystal, deforms in quite an anisotropic manner. Thus, to understand the mechanical properties of DS ingots, the conditions governing the compatibility of deformation for two adjacent columnar grains and their interaction in terms of both microscopic and macroscopic effects are required. For this purpose, we have been carrying out a systematic experimental study on the deformation and fracture of bi-PST and tri-PST crystals. Recent results of the study are summarized and their implications for the deformation of DS ingots are then discussed.

2:30 PM INVITED PAPER
MICROSTRUCTURE AND PROPERTIES OF HIGH-Nb CONTAINED TiAl-BASED ALLOYS: *Guoliang Chen*[1]; W J. Zhang[1]; Z. C. Liu[1]; S. J. Li[1]; *Young-Won Kim*[2]; [1]Univeristy of Science & Technology, State Key Laboratory for Advanced Metals & Materials, Beijing 100083 PR China; [2]UES, 4401 Dayton-Xenia Road, Dayton, OH 45432 USA

This paper summarizes our recent studies in the phase relationships, microstructure evolution/control, and tensile and creep properties in the Ti-(44~47)Al-(8~10Nb) alloys. The following aspects have been experimentally observed: 1) The Nb additions decrease the Ta and stabilize the B2 phase; 2) The a field is narrowed down by these additions; 3) A ternary g+a+B2 phase field was confirmed at 1050~1250℃; 4). The stacking fault energy in the g-TiAl phase is reduced, thereby promoting twinning and improving high temperature creep resistance; 5) Activation of both ordinary and super-dislocations was observed in Ti-45Al-10Nb deformed at RT as well as 900℃. The CRSS for ordinary dislocation slip in the g phase is increased; 6) The presence of B2 phase appears to be detrimental to the RT ductility and high temperature strength; 7) Various types of substructures formed by hot deformation may influence the microstructural stability at high temperature. From these results, key points for the future development of high-Nb containing gamma TiAl alloys are discussed.

3:00 PM
TiAl-BASED INTERMETALLICS: THE PHASE EQUILIBRIA GAMMA TiAl-ALPHA2 Ti3Al-PSI IN THE SYSTEMS WITH V,Cr,Mn,Fe,Co,Ni,Nb,Mo,Ru,Rh,Pd,Ta,W,Re,Os,Ir AND Pt: *Peter Franz Rogl*[1]; Jin Jun Ding[1]; Helmut Schweiger[1]; *Raimund Podloucky*[1]; [1]Universität Wien, Institut für Physikalische Chemie, Währingerstrasse 42, Wien A-1090 Austria

Strengthening of gamma-TiAl based intermetallics via solution and/or precipitation hardening involving third metal components, essentially depends on a detailed knowledge of the temperature and solute dependent solubility limits as well as of the chemical and crystallographic nature of the precipitate. We present the phase relations in a series of partial ternary systems Ti-Al-M particularly in the region bound by the phases gammaTiAl-alpha2Ti3Al-psi where M=V,Cr,Mn,Fe,Co,Ni,Nb,Mo,Ru,Rh,Pd,Ta,W,Re,Os,Ir,Pt and psi is the ternary compound in equilibrium with gamma and alpha2. The investigations are based on quantitative X-ray powder diffraction, light optical microscopy and EPMA. Solubility limits at 950℃ are derived for M in both gamma-TiAl and alpha2-Ti3Al. The phases psi and their crystal structures have been identified. Results of first principles calculations studying the site occupation of ternary additions of 3d, 4d, 5d elements are presented.

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THE OXIDATION BEHAVIOUR OF HIGH STRENGTH TiAl ALLOYS UNDER ISOTHERMAL AND CYCLIC EXPOSURE: *Alan Partridge*[1]; [1]DERA, Structural Materials Centre, Ively Rd., Farnborough, Hants GU14 0LX UK

Currently the maximum operating temperature of g-TiAl based alloys for aero-engine applications is limited to around 750℃; as a consequence of the rapid deterioration in oxidation resistance at higher temperatures. Recently a series of alloys have been developed, which contain up to 8 at.% (Nb, Hf, Zr, Ta) alloying additions. The oxidation behaviour of these alloys has been evaluated under both isothermal and cyclic oxidation conditions, and have been observed to display much improved oxidation resistance compared to traditional g-TiAl alloys with lower alloying additions. Detailed analyses of the oxidised samples have been performed; including high resolution EBSD analysis of the phases at the scale-metal interfaces. The results of these studies will be presented and mechanisms to explain the improved oxidation resistance will be proposed.

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A COMPARATIVE STUDY OF OXIDATION RESISTANCE OF ENGINEERING GAMMA TiAl ALLOYS: *Michiko Yoshihara*[1]; Young-Won Kim[2]; [1]Yokohama National University, Mech. Eng. and Mats. Sci., 79-5, Tokiwadai, Hodogaya-ku, Yokohama, Kanagawa 240-8501 Japan; [2]UES, Inc., Materials and Processes Division, 4401 Dayton-Xenia Road, Dayton, OH 45432-1894 USA

Gamma TiAl alloys have attractive properties for high temperature structural applications, with their use temperature limited by the oxidation and/or creep resistance. For the last ten years, various engineering gamma alloys have been developed in an effort to enhance the performance levels, often without the oxidation-resistance given a serious consideration. In this study, systematic experiments have been conducted to quantify and compare the oxidation behavior of typical engineering gamma alloys. The selected alloys include: Cast 4822 (Ti-47Al-2Cr-2Nb), cast 45XD (Ti-45Al-2Mn-2Nb-0.8TiB_2), cast 47XD (Ti-47Al-2Mn-2Nb-0.8TiB_2), wrought 395 (Ti-46.5Al-2Cr-2Nb-1Mo-0.2B), wrought K5S (Ti-46.2Al-2Cr-3Nb-0.2W-0.1Si), wrought KDCBS (Ti-46.4Al-2Cr-3Nb-0.2W-0.1B-0.2C-0.1Si), wrought 96F (Ti-46.5Al-2Cr-2.8Nb-0.2W-1Hf), wrought 95G (Ti-45Al-10Nb) and wrought 97G (Ti-45Al-8Nb). The oxidation tests were performed in air, with the isothermal exposures up to 200h and 2h-cyclic exposures up to 1000h at 760℃ and 870℃. Two Ti-Al binaries (Ti-46Al and Ti-50Al) and a conventional superalloy (Inconel 713C) were also tested as references. Detailed results will be discussed.

4:00 PM INVITED PAPER
ISOTHERMAL AND CYCLIC OXIDATION BEHAVIOR OF TITANIUM ALUMINIDES: *Lorenz Singheiser*[1]; Willem J. Quadakkers[1]; [1]Forschungszentrum Juelich GmbH, Institut für Werkstoffe und Verfahren

der Energietechnik 2, Leo-Brandt-Str., Juelich, NRW D-52425 Germany

The paper reviews the oxidation behavior of different titanium aluminides under isothermal and cyclic oxidation conditions in the temperature range from 700ϒC to 900ϒC. Special attention will be given on the role of different alloying elements as well as on the influence of gas phase composition on the oxidation kinetics and oxide composition. The effect of niobium in terms of its influence on the aluminum and titanium activity as well as on the microstructure of the subscale composition will be discussed. Niobium has a significant influence on the solubility of oxygen as well as nitrogen and seems to stabilize a so-called x-phase, which corresponds to an Al-rich oxygen containing Ti3Al. As long as this subscale phase is uniform alumina will be formed. Results will be presented how the stability of this phase can be increased by further alloying elements resulting in significant improved oxidation resistance of modified titanium aluminides compared with niobium containing alloys. The current status of coating development for titanium aluminides will be reviewed as well and the different approaches to improve oxidation and hot corrosion resistance will be discussed.

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INFLUENCE OF WATER VAPOR ON THE MECHANICAL PROPERTIES OF TiAl AT HIGH TEMPERATURES: *Andreas Zeller*[1]; Franz Dettenwanger[1]; *Michael Schütze*[1]; [1]DECHEMA e.V., Karl-Winnacker-Institut, Theodor-Heuss-Allee 25, Frankfurt am Main, Hessen D-60486 Germany

Recently, more attention is given to the influence of water vapor on the oxidation behaviour and the mechanical properties of TiAl, since it is known that water vapor embrittles intermetallic compounds, leading to a deterioration of the mechanical properties. At high temperatures a competition between surface oxidation and hydrogen attack seems to occur for TiAl. In order to evaluate the influence of water vapor on the oxidation and mechanical behaviour of TiAl based alloys, thermogravimetric analysis, creep and LCF-tests were conducted at 700ϒC in dry air and air containing a defined content of water vapor. In creep tests, the presence of water vapor leads to an increase in the primary creep strain, but the following minimum strain rate in the secondary creep regime does not show any significant difference. Therefore the cracking frequency of the protective oxide scale during deformation seems to play the key role for the extent of hydrogen attack on TiAl at 700ϒC. In LCF-tests the negative influence of water vapor is more pronounced, since the oxide scale is continuously damaged and the attack can proceed throughout the whole test. The paper will report and discuss the results of the mechanical tests, with focus on the influence of water vapor on oxide scale formation, on the Al-depleted subsurface layer as well as on crack initiation and propagation processes.

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THE INFLUENCE OF HIGH TEMPERATURE OXIDATION ON THE PERFORMANCE OF TWO GAMMA TiAl-BASED ALLOYS: *Marinus Frederik Stroosnijder*[1]; Jan Sunderkoetter[1]; Helmut Clemens[2]; Hans Peter Martinz[3]; Wolf Knabl[3]; [1]European Commission, Institute for Advanced Materials, Via Fermi 1, Ispra 21020 Italy; [2]Universitaet Stuttgart, Institut fuer Metallkunde, Seestrasse 71, Stuttgart 70174 Germany; [3]Plansee AG, Technology Center, Reutte, Tirol 6600 Austria

The isothermal and cyclic oxidation behaviour of two gamma TiAl-based alloys, i.e. Ti-47Al-2Cr-0.2Si and Ti-48Al-2Cr-2Nb (at%), was studied at 800ϒC in air. Ti-48Al-2Cr-2Nb showed the lowest oxidation rate and the highest spallation resistance. For Ti-47Al-2Cr-0.2Si a low isothermal oxidation rate but pronounced susceptibility for oxide spallation was found. During oxidation a complex multi-phased and -layered scale was formed on the surface of the alloys. In order to study the influence of oxidation on mechanical properties, 4-point bending tests were conducted at room temperature and 800ϒC on specimens which were pre-oxidized in air for different exposure times at 800ϒC. Although rather massive oxide scales had formed, no negative effect on the bending properties (strength, bending angle) at high temperature was observed. At room temperature, however, a significant degradation occurred. This behaviour can be attributed to brittle phases at the interface, which act as crack initiation sites below the brittle-to-ductile transition temperature.

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AN INVESTIGATION OF FRACTURE AND FATIGUE CRACK GROWTH IN CAST LAMELLAR Ti-48Al- 2Cr-2Nb: C. Mercer[1]; L. Jun[1]; F. Ye[1]; W. O. Soboyejo[1]; [1]Department of Materials Science and Engineering, The Ohio State University, 2041 College Road, Columbus, OH 43210-1179

The results of a recent study of fracture toughness and fatigue crack growth in cast lamellar Ti-48Al-2Cr-2Nb will be presented in this paper. These include: the effects of loading rate on fracture initiation toughness and resistance- curve behavior, and the effects of temperature on fatigue crack growth. Loading rates corresponding to stress intensity factor increase rates between 1 and 100 MPa ”m per second will be shown to have only a limited effect on the fracture initiation toughness (approx. 20 MPa ”m) and resistance-curve behavior at room-temperature. However, temperature is shown to have a significant effect on the fatigue thresholds and growth rates between 25 and 700 C. The trends in the fatigue crack growth rates and thresholds are rationalized by considering the combined effects of crack-tip deformation and oxide-induced crack closure. The observed crack-tip shielding mechanisms are also modeled using micromechanics concepts.

LONG TERM STABILITY OF HIGH TEMPERATURE MATERIALS: Stability of Ni-base Alloys - II

Sponsored by: Structural Materials Division, High Temperature Alloys Committee, Physical Metallurgy Committee
Program Organizers: Gerhard E. Fuchs, Lockheed Martin Corporation, Schenectady, NY 12301-1072 USA; Kathryn A. Dannemann, Southwest Research Institute, San Antonio, TX 7828-0510 USA; Todd C. Deragon, Special Metals Corporation, New Hartford, NY 13413-5392 USA

Wednesday PM Room: 9
March 3, 1999 Location: Convention Center

Session Chair: Kathryn A. Dannemann, Southwest Research Institute, San Antonio, TX 7828-0510 USA

2:00 PM INVITED PAPER

INVESTIGATION OF THE FORMATION OF TOPOLOGICALLY CLOSE PACKED PHASE INSTABILITIES IN NICKEL-BASE SUPERALLOY RENE N6: *Frank J. Ritzert*[1]; David R. Arenas[2]; Vijay K. Vasudevan[3]; [1]NASA Lewis Research Center, Materials Division, 21000 Brookpark Rd., MS 49-1, Cleveland, OH 44135 USA; [2]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., 209 Steidle Bldg., University Park, PA 16802 USA; [3]University of Cincinnati, Dept. of Mats. Sci. and Eng., 497 Rhodes Hall, ML #12, Cincinnati, OH 45221 USA

Topologically close packed (TCP) phase instability in third generation Ni-base superalloys is understood to hinder component performance when applied in high-temperature jet engine applications. The detrimental effects on high temperature performance from these brittle phases includes weakening of the Ni-rich matrix through the depletion of potent solid solution strengthening elements. Thirty-four compositional variations of polycrystalline Rene N6 were defined from a design-of-experiments approach and then cast, homogenized, and finally aged to promote TCP formation. Our prior work reported on the results of the multiple regression modeling of these alloys in order to predict the volume fraction of TCP. This paper will present further results on these alloys including elemental partitioning, kinetics, and comparison to other models.

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FORMATION OF ALPHA-Cr IN ALLOY 718 WITH VARYING Cr AND Nb LEVELS DURING LONG TERM AGING: *B. A. Lindsley*[1]; G. E. Maurer[1]; J. F. Radavich[2]; [1]Special Metals Corporation, 4317 Middle Settlement Rd., New Hartford, NY 13413 USA; [2]Micromet, West Lafayette, IN USA

Alloy 718 is commonly used in gas turbine disks at operating temperatures up to 649°C. Above this critical temperature, the γ" strengthening phase rapidly transforms in to delta phase and γ', resulting in a loss of tensile and impact properties. Below this temperature, the γ" is relatively stable, however, impact properties were found to drop off significantly after exposures below 649°C. Early work by Radavich found that α-Cr precipitated in retired Alloy 718 turbine disks after 28,000 hours of service. The α-Cr formed primarily along grain boundaries in association with delta phase at operating temperature of 593°C up to 718°C and was responsible for the drop in impact properties. A study was undertaken to assess the effect of composition on α-Cr formation by varying the levels of Cr and Nb in the alloy. Each alloy was given the following four thermo-mechanical treatments: direct age, solution and age, solution and age, and age plus cold worked reductions of 10% and 20%. Samples were heat treated for 500, 1000 and 2000 hours at 649°C. Microstructural evaluations by light optical microscopy, SEM and XRD of extracted particles were performed and α-Cr formation was found to be dependent on both chemistry and processing conditions. In addition, impact properties were found to correlate with precipitation and growth of α-Cr.

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RELATIONSHIP OF MICROSTRUCTURE WITH MECHANICAL PROPERTIES OF ALLOY 625, 718 AFTER LONG TERM EXPOSURE: *Xishan Xie*[1]; [1]University of Science and Technology Beijing, High Temperature Alloy Division, Dept. of Mats. Sci. and Eng., Beijing 100083 P.R. China

Microstructures and phase transformations of the world-wide most used superalloy Alloy 718 at high temperatures(593 -704°) very long time exposure (til to 50,000 hours) was investigated by means of transmission electron microscope analyses and the relevant mechanical properties were also determined for the evaluation of strengthening and brittleness. The degradation of strengthening are concluded on the following three main reasons: 1. The coarsening of separately precipitated strengthening phases of gamma-double prime and gamma-prime; 2. The phase transformation of gamma-double-prime to stable delta phase with large plate-like morphology; 3. The formation of brittle sigma phase. Detail microstructure analyses of Alloy 625 at 1100F-1400F up to 10,000 hours were also studied using SEM, TEM. The experimental results show that the reduction of impact energy is attibuted to the precipitation of M6C type carbides and alpha-Cr particles along and within grain boundaries during long term exposure in alloy 625.

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LONG-TERM THERMAL STABILITY OF THERMO-SPAN ALLOY: *R. B. Frank*[1]; [1]Carpenter Specialty Alloys, Cartech R&D Center, P.O. Box 14662, Reading, PA 19612-4662 USA

Thermo-Span is an age-hardenable, Ni-Co-based superalloy that combines low thermal expansion characteristics with improved oxidation resistance compared to previous controlled-expansion superalloys. Heat-treated samples were exposed for 1500 hours at temperatures ranging from 900°F to 1250°F. Evaluations included tensile tests at room temperature, 1100°F and 1200°F, stress-rupture tests at 1250°F and post 1150°F creep tensile tests at 1150°F. The results showed that Thermo-Span alloy exhibits stable tensile properties and microstructures after 1500-hour exposures at temperatures within the range of 900°F and 1200°F. No embrittlement occurred at these temperatures. Exposures at 1250°F reduced strength but did not degrade stress-rupture properties. Neither stressed or un-stressed exposures at 1150°F had a significant effect on tensile properties at 1150°F.

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CREEP-RUPTURE OF ALLOY 718 AFTER THERMAL AGING UP TO 50,000 H: *Gary E. Korth*[1]; [1]Lockheed Martin Idaho Technologies Company, Metals and Ceramics, P.O. Box 1625, Idaho Falls, ID 83415-2218 USA

Alloy 718 given the conventional 718/621ϒC age-hardening treatment was thermally aged at 593, 621, and 649ϒC for times up to 50,000 hours. These temperatures bracket the lowest of the duplex age-hardening temperature. Subsequent creep-rupture tests conducted at the same temperature of thermal aging show 593ϒC aging to be beneficial up to 10,000 h and only slightly detrimental after 50,000 h. Material aged for 50,000h and tested at 621ϒC shows the rupture life to be approximately 60-65% of the unaged material, but at 649ϒC the aged material rupture life is of the order of 15% of the unaged material. Room temperature tensile properties of the aged material will also be presented with some microstructure evolution with the long-term aging.

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EFFECT OF THERMAL AGING ON MICROSTRUCTURE/COMPOSITION AND MECHANICAL PROPERTIES IN ALUMINIZED CoCrAlY COATINGS: *Jun Kameda*[1]; Tamra E. Bloomer[1]; Yuji Sugita[2]; Akihiro Ito[2]; Shigeo Sakurai[3]; [1]Iowa State University, Metallurgy and Ceramics, Ames Laboratory, 206 Metals Development Bldg., Ames, IA 50011 USA; [2]Electric Power R&D Center, Chubu Electric Power Co., 20-1 Kitasekiyama, Aza Otaka, Midori-ku, Nagoya 458 Japan; [3]Hitachi, Ltd., Mech. Eng. Research Laboratory, 3rd Dept., 1-1 Saiwaicho 3-Chome, Hitachi 317 Japan

The effect of thermal aging at 870ϒC for 8000 h in air on the microstructure/composition and mechanical properties (RT and 870ϒC) has been studied in aluminized CoCrAlY coatings consisting of four layered structure (region I-IV) of advanced gas turbine blades. Thermal aging led to a little oxidation/nitridation and a decrease in the Al content in a near surface region I. In a coating region II, coarse Cr rich σ precipitates formed during the thermal aging. Thermally aged internal (III) and near interface (IV) coating regions showed extensive dispersion of σ and/or Al/Ni rich β/α eutectic precipitates. Small punch tests at RT and 870ϒC in air have shown that the coating regions I and II of unaged and aged blades indicated easier formation of brittle cracks regardless of the composition change. The ductility of the regions III and IV at RT and 870ϒC, and the low cycle fatigue life of the region III were reduced by the thermal aging. The mechanical degradation at elevated temperatures in the aged coating regions III and IV is elucidated by taking into account the microstructure/composition evolution and environmental oxidizing effects. 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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DEGRADATION OF MICROSTRUCTURE OF NICKEL SUPERALLOY <001> SINGLE CRYSTALS UNDER THE INFLUENCE OF TEMPERATURE AND/OR STRESS.: *Bakhteeva Natalia Dmitrievna*[1]; *Petrova Sofya Nikolaevna*[1]; Vinogradova Nina Ivanovna[1]; [1]Instittute of Metal Physics, Ural Branch of Russian Academy of Sciences, 18 S.Kovalevskaya Str., Ekaterinburg GSP-170, Sverdlovsk Region 620219 Russia

The microstructure of <001> single crystals of richly alloyed nickel superalloy containing 65% volume fracture of gamma-prime phase subjected to crystallization, heat treatment, tests for high-temperature creep and subsequent heatings has been investigated by various methods: metallography, electron microscopy and X-raying. The degradation of microstructure (intermetallide and carbide phases) has been revealed under the action of temperature and/or stress. The stability of lamellar gamma/gamma-prime structure at high-temperature heating was studied. The tight correlation is shown to exist between the morphology of gamma-prime particles and the high-temperature properties of single-crystal samples.

MANUFACTURING ISSUES IN RAPID THERMAL PROCESSING: Session II

Sponsored by: Electronic, Magnetic and Photonic Materials Division, Thin Films and Interfocus Committee

Program Organizers: N.M. Ravindra, New Jersey Institution of Technology, Dept. of Phys., Newark, NJ 07102 USA; Daniel F. Downey, Varian Ion-Implant System, Gloucester, MA 01930 USA; Anthony T. Fiory, Lucent Technologies, Bell Labs., Room 1D468, Murray Hill, NJ 07974-0636 USA; Steven D. Marcus, AST Elektronik USA Inc., Tempe, AZ 85284 USA; B. Sopori, National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA

Wednesday PM Room: 14A
March 3, 1999 Location: Convention Center

Session Chair: Steven D. Marcus, STEAG-AST Elektronik, Tempe, AZ 85284 USA

2:00 PM KEYNOTE
THE APPLICATION OF RAPID THERMAL PROCESSING TECHNOLOGY TO THE MANUFACTURE OF INTEGRATED CORCIOTS=AN OVERVIEW: Alan Bratschun; SEMATECH

Like many of the technologies used to process integrated circuits, the road to manufacturing for RTP has been twisted. What began as a speculative laboratory apparatus, has evolved into a cornerstone of IC technology. Qualities that make RTP desirable for IC manufacture include the ability to process wafers individually, the ability to minimize the time wafers spend at elevated temperature, the convenience of clustering RTP to other systems, and the possibility of maintaining cold reactor walls. This paper will review how these properties make RTP desirable. The paper will also present an overview of the difficulties surrounding the use of RTP and describe how many of the serious hurdles have been overcome. It will summarize the evolution of RTP from a curiosity to a mainstay technology in building integrated circuits. It will then describe SEMATECH's role in working with RTP, ending with a direction for future applications of RTP based on the SIA Roadmap for semiconductors.

2:30 PM INVITED PAPER
CONTROL OF INTERFACE PROPERTIES OF Si-Al CONTACTS USING OPTICAL PROCESSING: *Bhushan Sopori*[1]; Wei Chen[1]; [1]National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA

Control of the interface properties is important in making semiconductor-metal contacts with desirable properties. Fabricating contacts on solar cells poses unique challenges because they necessitate control of both their electronic and optical properties. This paper describes how Optical Processing can be applied to fabricate Si-metal contacts that satisfy both optical and electronic properties for solar cells. This process allows one to control the thickness of the semiconductor-metal melt during the contact formation, producing an abrupt Si-Al contact of very low contact resistivity and high optical reflectance. In addition, the contacts with a gradient in the Al concentration can be fabricated. We will also discuss the properties of Si-Al interfaces formed under various process conditions. Of particular interest are the reflection behavior and the formation of the P/P+ interface for high-efficiency silicon solar cells. We will show the measured optical and spreading resistance data and compare it with theoretical results.

3:00 PM INVITED PAPER
WAFER TEMPERATURE MEASUREMENT IN A RAPID THERMAL PROCESSOR WITH MODULATED LAMP POWER: *A. T. Fiory*[1]; B. Nguyenphu[2]; [1]Bell Laboratories, Lucent Technologies, Inc., Murray Hill, NJ 07974 USA; [2]Lucent Technologies, Inc., Microelectronics Group, Orlando, FL 32819 USA

Wafer temperature measurements similar to Accufiber's ripple technique were employed in a pyrometer to compensate for variable wafer back side emissivities. Power to the heating lamps was modulated with oscillatory functions of time. Fluctuating and quasi-steady components in detected radiation were analyzed to suppress background reflections from the lamps and to correct for effective wafer emissivity. Wafer process results for unpatterned wafers, as film thickness for thermal oxidation and as sheet resistance for annealing of high-dose shallow implants, were used to inter wafer4o-wafer process temperature variability over a range in back side emissivity. Emissivities were varied by depositing or growing one or more layers of silicon dioxide, silicon nitride, or polycrystalline silicon on the backsides.

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CALCULATION OF THE EMISSIVITY OF Si WAFERS: *Bhushan Sopori*[1]; Wei Chen[1]; Jamal Madjdpour[1]; N. M. Ravindra[2]; Sufian Abedrabbo[2]; [1]National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden, CO 80401 USA; [2]New Jersy Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

This paper will discuss a computer-software for calculating the emissivity of silicon wafers of any surface morphology, for a given temperature and dopant concentration. The calculations use an optical model that includes surfaces of any shape - planar, rough, or textured. The optical constants, i.e. refractive index and absorption coefficient, are calculated as a function of temperature using an empirical model for an indirect bandgap semiconductor. We will compare the results of this model with other methods of emissivity calculations and with the experimental data. This software can be very useful for monitoring wafer temperature in RTP systems.

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TEMPERATURE UNIFORMITY OPTIMIZATION FOR A SILICON IMPLANT ANNEAL INTO GAAS USING OPUS♦ SIMULATION SOFTWARE: *Helmut Francz*[1]; Zaid Farukhi[1]; Steven D. Marcus[1]; Andreas Tillrnann[2]; Juergen Niess[3]; [1]Motorola, CS-1, Tempe, AZ 85284 USA; [2]STEAG AST Elektronik, Gambh, Dornstadt Germany; [3]STEAG AST Elektronik, Tempe, AZ 85284 USA

OPUS♦, which stands for "Optimized Uniformity Simulator", has been described in the literature as a powerful tool to assist in the optimization of temperature uniformity on the STEAG AST Elektronik series of rapid thermal processors. Historically, this tool, OPUS™, has been used extensively for a variety of silicon processing.

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THE APPLICATION OF RAPID THERMAL PROCESSING TECHNOLOGY TO THE MANUFACTURE OF INTEGRATED CIRCUITS-AN OVERVIEW: *Alan Bratschun*[1]; [1]SEMATECH

Like many of the technologies used to process integrated circuits, the road to manufacturing for RTP has been twisted. What began as a speculative laboratory apparatus, has evolved into a cornerstone of IC technology. Qualities that make RTP desirable for IC manufacture include the ability to process wafers individually, the ability to minimize the time wafers spend at elevated temperature, the convenience of clustering RTP to other systems, and the possibility of maintaining cold reactor walls. This paper will review how these properties make RTP desirable. The paper will also present an overview of the difficulties surrounding the use of RTP and describe how many of the serious hurdles have been overcome. It will summarize the evolution of RTP from a curiosity to a mainstay technology in building integrated circuits. It will then describe SEMATECH's role in working with RTP, ending with a direction for future applications of RTP based on the SIA Roadmap for semiconductors.

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RAPID PHOTOTHERMAL PROCESSING IN SEMICONDUCTOR MANUFACTURING IN THE 21ST CENTURY: *R. Singh*[1]; V. Parihar[1]; K. F. Poole[1]; [1]Clemson University, Department of Electrical and Computer Engineering, Center for Silicon Nanoelectronics, Clemson, SC 29634-0915 USA

Abstract Not Available.

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EMISSIVITY MEASUREMENTS AND MODELING OF INFRARED TRANSPARENT MATERIALS: *Manish Babladi*[1]; Oktay Gokce[1]; F. M. Tong[1]; Daniel E. Pierce[2]; Gyuido Guazzoni[3]; N. M. Ravindra[1]; [1]New Jersey Institute of Technology, Newark, NJ USA; [2]William Peterson University, Wayne, NJ USA; [3]US Army, CECOM, Fort Monmouth, NJ USA

Experimental results of the temperature dependent radiative properties of infrared transparent materials (quartz, lucalox and sapphire) have been reported in this study. These measurements have been performed using a spectral emissometer operating in the wavelength range of 0.8 to 20 microns and the temperature range of 17 to 900 ^{0}C. For double side polished materials, the measured optical properties have been deconvoluted to yield fundamental optical constants. Comparisons of the obtained results are made with those available in the literature. Applications of this study to rapid thermal process systems have been identified and explained.

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EFFECT OF ULTRAVIOLET (UV) AND VACUUM ULTRAVIOLET (VUV) PHOTONS IN RAPID PHOTOTHERMAL PROCESSING: *V. Parihar*[1]; R. Singh[1]; K. F. Poole[1]; A. Rohatgi[2]; [1]Clemson University, Department of Electrical and Computer Engineering, Center for Silicon Nanoelectronics, Clemson, SC 29634-0915 USA; [2]Georgia Institue of Technology, School of Electrical Engineering, Atlanta, GA 30322 USA

Abstract Not Available.

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APPLICATION OF SPECTRAL EMISSOMETRY FOR THE ESTIMATION OF CONDUCTION INTRABAND TRANSITIONS IN n AND p SILICON: *S. Abedrabbo*[1]; J. C. Hensel[1]; A. T. Fiory[3]; N. M. Ravindra[2]; [1]New Jersey Institute of Technology, Newark, NJ USA; [1]Kearfott Guidance and Navigation Corporation, Wayne, NJ USA; [3]Bell Laboratories, Lucent Technologies, Murray Hill, NJ USA

n-Si exhibits an absorption band in the wavelength range of 1-5 mm that is attributed to transitions of electrons from conduction band minima to the next higher band. This has been reported first by Spitzer and Fan for various doping concentrations ranging from 10^{14} – 10^{19} cm^{-3} at room temperature. It has been reported also that p-Si does not exhibit aa similar band structure in the extrinsic regime. In this work the first detailed temperature dependent conduction intraband transitions are reported as function of wavelength for p-Si in its intrinsic regime. The band peak has shown negligible dependence on temperature as expected and remains at l~2.3mm, while the peak intensity has risen as the temperature is increased indicating an increase in the thermally generated electrons ready to make the transition.

MATERIALS PROCESSING FUNDAMENTALS: Ferrous

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

Program Organizers: W.D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Huimin Liu, UES, Inc., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Lab, P.O. Box 2008, Bldg. 4508, Oak Ridge, TN 37831-6083 USA

Wednesday PM Room: 5A
March 3, 1999 Location: Convention Center

Session Chairs: Janusz Majta, University of Mining and Metallurgy, Dept. of Metall. and Mats. Sci., Krako 30-059 Poland; Derek O. Northwood, Ryerson Polytechnic University, Dept. of Eng. & Applied Sc., Toronto, Ontario M5B2K3 Canada

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ISOTHERMAL NORMALIZING SYSTEM BY UTILIZING THE RESIDUAL FORGED HEAT IN ALLOY CARBURIZING PRODUCTS: *Zhenbo Zhao*[1]; Cheng Liu[2]; Yunxu Liu[2]; *Derek O. Northwood*[3]; [1]University of Windsor, Mechanical & Materials Engineering, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Jilin Institute of Technology, Materials Engineering, 76 Yanan Dalu, Changchun, Jilin 130012 P.R.China; [3]Ryerson Polytechnic University, Engineering & Applied Science, 350 Victoria Street, Toronto, Ontario M5B 2K3 Canada

The processes and equipment for INURFH (Isothermal Normalizing by Utilizing the Residual Forged Heat) of alloy carburizing steel products are presented. It is shown that the four key parameters: namely (I) cooling rate before isothermal normalizing; (II) the lowest cooling temperature before isothermal normalizing; (III) isothermal temperature ; and (IV) isothermal time, should be controlled in order to meet the requirement of the isothermal normalizing treatment. The prediction and calculation of these parameters can be achieved on the basis of components treated and the technical requirements of steel parts by an on-line expert system for this isothermal normalizing system. It is believed that cost-reduction due to the energy savings resulting from this system for automobile parts heat-treatment is most promising.

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MICROSTRUCTURE AND CRYSTALLORGRAPHIC TEXTURE OF STRIP-CAST 4.3WT% SILICON STEEL SHEET: *Jae Young Park*[1]; Kyu Hwan Oh[1]; Hyung Yong Ra[1]; [1]Seoul National University, Division of Materials Science and Engineering, Shirim-dong 56-1, Kwanak-ku, Seoul 151-742 Korea

Grain oriented silicon steels have been mainly manufactured by continuous casting, hot rolling, cold rolling and recrystallization. In comparison to these conventional processes, the manufacture of grain oriented silicon steels by the strip casting process has several kinds of merits such as the omission of making progress and the increasement of silicon content etc., but because the hot rolling process is bypassed, through-the-thickness variation of texture is different with that of the hot-rolled sheet. In this report, Fe-4.3wt%Si steel sheet was prepared by the vertical type twin roll strip casting process and then the microstructure and texture at each thickness level of the strip were studied by optical metallography and quantitative X-ray texture analysis. The inhomogeneity of the texture and the microstructure through the sample thickness was investigated in the twin roll strip cast steel sheet.

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EVOLUTION OF NON-METALLIC INCLUSIONS IN ALUMINIUM KILLED LOW ALLOYED STEEL: *Rob Dekkers*[1]; *Bart Blanpain*[1]; *Patrick Wollants*[1]; *Frank Haers*[2]; *Carina Vercruyssen*[2]; *Leo Peeters*[2]; [1]Katholieke Universiteit Leuven, Metalllurgy and Materials Engineering, W. de Croylaan 2, Leuven, Vlaams Brabant B-3001 Belgium; [2]Sidmar N.V., Steel plant, J.F. Kennedylaan 51, Gent, Oost Vlaanderen B-9012 Belgium

Non-metallic inclusions in liquid steel are mainly formed during killing and cooling. As far as these particles are not captured by the slag, they are found in the cast steel where they can deteriorate the mechanical properties. In almost all cases inclusions disturb the continuous casting process, affecting thus directly the quality of the steel and the production capacity. Steel pin samples from the Sidmar plant (Belgium) were taken before, during and after killing/alloying of the steel in the ladle, after correction/alloying stage in the ladle and in the tundish. Also slab samples were studied. All samples concern the same type of aluminium killed low alloyed steel. The non-metallic inclusions were isolated from the steel matrix and investigated with scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS). The evolution of the non-metallic inclusions is described by their number, size, shape and elemental composition. The main non-metallic inclusions present are aluminium oxides and spinel type compounds. Decrease of the amount of small (less than 1 μm) spherical inclusions during the process is related to coalescence of inclusions, which is illustrated by a counting methodology and by SEM micrographs.

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COMPUTER MODELING FOR TEXTURE DEVELOPMENT IN COLD ROLLED FERRITE: Guanglei Liu[1]; *Ruoju Zhang*[2]; [1]The University of Hong Kong, Dept. of Mechanical Engineering, Pokfulam Rd. Hong Kong; [2]The University of Hong Kong, Dept. of Electrical & Electronic Engineering, Pokfulam Road, Hong Kong

Microstructural investigation has shown that grain splitting or deformation banding develops progressively with increasing strains during cold rolling. Of particular significance to simulating texture development in rolled ferrite is the occurrence of deformation banding in the rolling plane since this allows another degree of freedom. Deformation Banding (DB) model includes DB in the rolling plane and applies the Relaxed Constraints (RC) mechanism for other shears, this gives an improved fit between experiment and theory within Taylor-Chin framework. The predicted texture with DB model and { 110 } + { 112 } slip systems is in good agreement with experimental observation. DB modeling is able to predict the evolution of specific textural features in cold rolled pure iron, especially, from different initial textures.

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RELATIONSHIP OF HOT DEFORMATION CONDITIONS TO MICROSTRUCTURE DEVELOPMENT IN TWO-PHASE REGION OF MICROALLOYED STEEL: *Anna K. Zurek*[1]; Janusz Majta[2]; [1]Los Alamos National Laboratory, MST-8, MS:G755, Los Alamos, NM 87454 USA; [2]University of Mining and Metallurgy, Metallurgy and Materials Science, al. Mickiewicza 30, Krakow 30-059 Poland

Most of the metal forming processes is performed in conditions where finish deformation temperature occurs in austenite region. However, it is well established that new very attractive possibilities of properties can be obtained when final product has a structure that came from deformation performed in two-phase region. In such produced steels, the main problem that should be solved is good inhomogeneity control of microstructure and resulted mechanical behaviors for metal forming operations. It is well known, that most effective and powerful tool to control such complicated deformation conditions is the computer modeling. In this paper a model of thermomechanical behavior and microstructural development during hot compression of microalloyed steel will be presented. We will test several conditions, for hot compression tests, at a number of strain rates of deformation of steel. Finally, a new concept of phase transformation model verified by the above experimental data will be introduced.

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IN-SITU OBSERVATION OF THE RATE OF DENITOROZATION OF MOLTEN STEEL: Muneyuki Iwasaki[1]; Takashi Ikeda[1]; *Masafumi Maeda*[1]; [1]University of Tokyo, IIS, 7-22-1 Roppongi, Minato, Tokyo Japan

Mass spectroscopy was used for the direct observation of the rate of denitrization. Nitrogen molecules or atoms were detected by a mass spectrometer set above the melt. Continuous monitoring of the intensity of the fixed mass number reflected the transfer of nitrogen from the melt to the vacuum. Direct observation enabled to study the rate of denitrization. The results of that with those by conventional the quench, sampling and chemical analysis technique agreed.

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KINETIC STUDY OF THE REDUCTION OF MnO SLAGS AND THE CARBON DISSOLUTION IN Fe-Mn BATHS.: José Roberto de Oliveira[1]; *Jorge Alberto Soares Tenório*[1]; [1]University of São Paulo, Metallurgical and Materials Dept., Av. Prof. Mello Moraes 2463, Butantã, São Paulo 05508-900 Brazil

In a smelting reduction process the reduction reaction occurs predominantly in the liquid state. The main reduction agent is the carbon from the carbon saturated iron bath. The aim of the present work was to determine the velocity equation and the mass transfer coefficient of the reduction rate of MnO content in a MnO-SiO2-FeO slag by iron saturated carbon and the carbon dissolution rate in a Fe-Mn bath. The manganese content in the iron bath was in the range of 5 to 40 wt.%. It was observed that the rates of carbon dissolution in all Fe-Mn baths are faster than the rates of MnO reduction by the carbon saturated liquid iron.

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OPTICAL PYROMETRY IN METALLURGY AND IN PHYSICAL METALLURGY - SOME NEW THEORETICAL AND EXPERIMENTAL RESULTS: Evgeny D. Glazman[1]; [1]3T - True Temperature Technology, Theradion Industrial Park, Misgav 20179 Israel

In this work we describe a new approach to the problem of non - contact measurement the true thermodynamic temperature of fluid and solid materials in industrial processes. Based on this technique, an optical system for the measurement of the actual temperature was designed. The main advantage of the specified multispectral method is the ability to give an information about the true radiating efficiency (emissivity) of the measured object surface, together with the information about the brightness and color temperature. The indicated system is widely applicable to variety of industrial processes for measurement the temperature of fluid steel, aluminum and aluminum alloys, copper, nickel, cobalt and other materials being in liquid or solid state. The high accuracy of the system was demonstrated to give a reading error not greater than ± (1º1.5%). Amongst the industrial process applications of the system are: Blast furnace of fluid steel, continuous casting and rolling of steel and aluminum, nickel and cobalt melts, as well as extrusion processes of aluminum and its alloys [1]. The Introduction of the new pyrometer to industrial use allows not only to solve the problem of non-contact true temperature measurement of high temperature processes, but also to efficiently combine it with complex and automatic control systems. References: 1. E.D. Glazman, Measurement of Thermodynamic Temperature in the Extrusion Process of Aluminum and Aluminum Alloys, Light Metal AGE, 46-8, April 1998.

MICROMECHANICS AND MICROMECHANISMS OF DEFORMATION AND FRACTURE: A SYMPOSIUM IN HONOR OF PROFESSOR ALI S. ARGON: Ali S. Argon Symposium VI

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

Wednesday PM Room: 14B
March 3, 1999 Location: Convention Center

Session Chairs: Robert O. Ritchie, UC-Berkeley, Dept. Mat. Sci. & Mineral Eng., Berkeley, CA 94720-1760 USA; J. G. Williams, Imperial College of Science Technology & Medicine, Dept. of Mech. Eng., London SW7 2BX UK

2:00 PM INVITED PAPER

ANALYZING CUTTING VIA FRACTURE MECHANICS: *J. G. Williams*[1]; [1]Imperial College of Science, Mechanical Engineering Department, Technology and Medicine, Exhibition Road, London, SW7 2BX UK

Cutting a strip with a blade or wedge is used to assess the fracture toughness of many systems. It can be used for cleaving layers as in mica or in breaking adhesive joints. A very similar mechanism is involved in cutting layers as in microtomes or planes. In all these cases energy is dissipated in friction and plastic work as well as fracture. The plastic work gives curling of the strip and this provides a method of determining the plastic work. Friction can be measured from blade forces. This paper will describe an analysis scheme to correct the data for both friction and plastic dissipation, so that the true fracture energy can be found. Examples will be given for adhesives and the cutting of polymers and biological samples.

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PREDICTING CLEAVAGE CRACK GROWTH IN BCC POLYCRYSTALS: *Frank A. McClintock*[1]; [1]Massachusetts Institute of Technology, Department of Mechanical Engineering, Room 1-304, Cambridge, MA 02139-4307 USA

A preliminary model of polycrystalline cleavage was based on square cylinders normal to the macrocrack. Each cylinder was randomly assigned one of five cleavage orientations. The linkage of cracks was assumed to occur by shear sliding off and cracking along the grain boundaries. Approximate hand calculations gave an extremely tortuous crack path, as observed fractographically. The height differences that developed gave a work per unit area comparable to the lower shelf value for initial macro-crack growth. Here, after a review of the fractographic literature, possible extensions of the model are explored. Hexagonal cylinders, even with only one tilt angle, involve some dozen different tilt-twist misorientations across grain boundaries. For any one grain-to-grain penetration of cleavage into a neighboring grain, calculation of the driving paramaters and resistance functions would involve a multi-scale calculation. The scales involved range from the atomic to the grain-sized boundary conditions on the inhomogeneous polyslip in the grain being penetrated. For small twist angles, multiple initiation sites result in river patterns. These difficulties led to quantifying the square-cylinder model to study the effects of the length of, and the initial roughness along, the polycrystalline crack.

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MICROMECHANICAL MODELLING OF THE PLASTIC BEHAVIOR AND DAMAGE NUCLEATION OF A DUPLEX STAINLESS STEEL: *Jacques Besson*[1]; Stephane Bugat[1]; Anne-Francoise Gourgues[1]; Andre Pineau[1]; [1]Ecole des Mines de Paris, Centre des materiaux CNRS UMR 7633, BP 87, Cedex, Evry 91003 France

Ductile rupture is frequently initiated in second phase particles. A micromechanical description of damage nucleation therefore requires an estimation of strains and stresses in the different phases. In the present study, this approach is applied to a duplex stainless steel in which the ferritic phase is embrittled by a phenomenon known as "475°C embrittlement". Consequently, the hardness of the ferritic phase increases and induces a significant decrease of ductility and fracture toughness. The investigated duplex steel consists of large ferrite grains of about 10~mm formed during the early stages of solidification. As cooling proceeds, austenite grains (size 1~mm) are formed in the ferrite according to the Kurdjumov - Sachs relationships. These grains are defined as areas where the austenitic phase keeps the same crystallographic orientation. Both phases are interconnected and percolated and form laths whose spacing equals 10~microns. As cleavage is primarily controlled by tensile stresses, any attempt to model damage nucleation should be based on a realistic estimation of the local stress and strain fields. This study is motivated by the fact that damage is heterogeneous; highly damaged zones correspond to some austenitic grains evidencing the role of crystallography in the damage process. Damage rate is affected by the orientation of the ferrite with respect to both the macroscopic load and the surrounding austenite. A micromechanical model, accounting for crystal plasticity, has been developed to represent average stresses and strains in both phases over a representative volume element corresponding to the interconnected network. This model is used to simulate, using the Finite Element Method, small tensile samples tested in a Scanning Electron Microscope. Local grain orientations are measured before testing using Electron Back Scattering Patterns. They are used as input for the simulation. Comparison of computed stresses in the ferrite with observed damage nucleation, allows to derive a cleavage criterion relative to the ferrite only and independent of the local crystallographic orientations.

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THE THEORETICAL STRENGTH IN SHEAR: C. R. Krenn[1]; D. Roundy[2]; M. L. Cohen[2]; *J. W. Morris, Jr.*[1]; [1]University of California, Department of Materials Science, Berkeley, CA 94720 USA; [2]University of California, Department of Physics, Berkeley, CA 94720 USA

The theoretical shear strength of a material is defined as the shear stress a defect-free specimen can withstand before unstable deformation. It is, therefore, the ultimate measure of hardness and the most fundamental indication of mechanical properties. While theorists have attempted estimates of the theoretical shear strength for many years, it has recently become possible to compute it through ab initio electronic structure calculations, using the local density approximation, with full atom relaxation. Results will be presented for several prototypic elements and compounds. The results include the energy-strain and shear stress-strain curves for shear displacement to instability, the atom displacement paths, and the evolution of the electronic structure, which helps to explain whythe theoretical strengths have the values they do.

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FRACTURE AND FATIGUE OF BULK AMORPHOUS METALS: Christopher J. Gilbert[1]; Valeska Schroeder[1]; *Robert O. Ritchie*[1]; [1]University of California, Berkeley, Materials Science and Mineral Engineering, 463 Evans Hall #1760, Berkeley, CA 94720-1760 USA

The fracture and fatigue properties of the bulk metallic glass alloy, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (at.%), are characterized with specific emphasis on controlling microstructural mechanisms. It is found that the fully amorphous alloy exhibits a high fracture toughness of ~55 MPa m, although rounded-notch Charpy impact energies are less than 10 J. Moreover, the K_{Ic} toughness is markedly sensitive to strain rate and the level of precrack damage, and shows no resistance-curve behavior. Under both quasi-static and dynanmic loading, fracture surfaces exhibit a characteristic vein morphology with apparent evidence for local melt-

ing during fracture. The latter is consistent with spectroscopy measurements of extremely high temperatures (>2000K) during fracture, associated with marked fracto-luminescence. This fracture mode is consistent with Argon and Salama's model of a variant of the Taylor instability, i.e., associated with the tendency of a "fluid" meniscus propagating under a positive pressure gradient to break up into a series of "fingers". In addition, upon crystallization the alloy becomes severely embrittled with K_{Ic} dropping to ~1 MPa m. Under cyclic loading, crack-propagation behavior in the amorphous structure is similar to that observed in traditional polycrystalline alloys, with alternating blunting and resharpening of the crack tip, as evidenced by striations on fatigue fracture surfaces. However, in sharp contrast, the (unnotched) stress-life properties are markedly different. Specifically, crack initiation occurs quite readily due to the lack of microstructural barriers which normally provide crack-arrest points; this results in a very low fatigue limit of ~1/25 of the tensile strength.

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COMPATIBILITY STRESSES AT GRAIN BOUNDARIES AND THEIR EFFECT ON CRACK INITIATION IN HIGH CYCLE FATIGUE: *Peter Neumann*[1]; [1]Max-Planck Institut fur Eisenforschung GmbH, Max-Planck-Str. 1, 10237 Dusseldorf Germany

The compatibility stresses at elastically incompatible grain boundaries are discussed and presented in analytical form. These incompatibility stresses give rise to a logarythmic singularity of stresses at the intersecting line between a grain boundary and the specimen surface. Approximate analytical solutions for these stresses are given based on the work of Mushkelishvili. Furthermore, some examples of three-dimensional calculations of stress-concentrations at grain boundaries are given. These theoretical results are compared with the observation of fatigue crack initiation experiments with copper and austenitic steel polycrystals. In the high cycle i.e. low plastic strain regime (cycles to failure>300,000) the initiation sites were found to be twin boundaries almost exclusively. By local orientation measurements at hundreds of grain boundaries quantitative estimates of local stress concentration were obtained and compared with the theoretical results. An almost one to one correlation was found between the occurrence of high local stress concentrations at twin boundaries and crack nucleation sites. Thus the compatibility stresses seem to be the main cause for fatigue crack initiation in high cycle fatigue, when other faults like inclusions are missing.

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WORKPIECE WEAR MECHANISMS IN DEFORMATION PROCESSING: Gilbert J. Baker[2]; *Roger N. Wright*[1]; [1]Rensselaer Polytechnic Institute, Materials Science and Engineering Department, 206 MRC Building, Troy, NY 12180 USA; [2]Essex Group, Inc., Fort Wayne, IN 46801 USA

Workpiece wear, as opposed to tool wear, has received little attention in deformation processing analysis, except perhaps for the relatively gross losses associated with sticking friction. This paper focuses on the subtle, but important, liberation of particles from the workpiece surface (sometimes called "fines"). Emphasis is placed on copper wire drawing, where "fines" are a major issue vis-a-vis lubrication, surface quality, and subsequent insulation application. Detailed laboratory study reveals that "fine" generation involves the classical mechanisms of delamination wear and rapid wear. In the case of copper wire drawing, delamination wear involves damage to the near-surface from prior processing. The rapid wear observed is consistent with a modified Archard equation. Delamination wear is retarded by the application of back tension, and rapid wear can be minimized by die angle optimization.

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MICROSTRUCTURE AND MECHANISMS OF CYCLIC DEFORMATION OF ALUMINUM SINGLE CRYSTALS AT 77K: *M. E. Kassner*[1]; [1]Oregon State University, Mechanical Engineering, Rogers Hall, Corvallis, OR 97331 USA

Aluminum single crystals were cyclically deformed in single slip at small strain amplitudes at 77K to presaturation. The dislocation substructure was analyzed in detail. The maximum dipole height suggests a strength approximately equal to the flow stress. The stress to move a dislocation through a dipole bundle is also calculated to be approximately equal to the flow stress. In situ cyclic reverse (shear) deformation experiments in the high-voltage transmission electron microscope (HVEM) were successfully performed using the X-Y technique where thin foils are stressed in alternating perpendicular directions. The experiments indicate that loops frequently expand from the dipole bundles into the channel and the edge component is absorbed by nearby bundles, leaving screw segments behind. The screw dislocations that span the channel move easily and reverse direction with shear reversal. There is no obvious evidence for internal backstresses that assist plastic deformation on reversal of the applied shear.

MINIATURE STRUCTURES & COMPONENTS UNDER CYCLIC LOADING; FATIGUE & INTERNAL FRICTION: Session IV

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee; Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee; ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: H. D. Merchant, Gould Electronics, Inc., Eastlake, OH 44095-4001 USA; Thomas R. Bieler, Michigan State University, Dept. of Mats. Sci. & Mech., East Lansing, MI 48824-1226 USA; James C. Earthman, University of California, Dept. of Chem. Eng. & Mats. Sci., Irvine, CA 92717-2535 USA; M. Wuttig, University of Maryland, Dept. of Mats. & Nuclear Eng., College Park, MD 20743-2115 USA

Wednesday PM Room: 11B
March 3, 1999 Location: Convention Center

Session Chairs: Hans Conrad, North Carolina State University, Dept. of Mats.Sci. and Eng., Raleigh, NC 27695-7907 USA; James C. M. Li, Rochester University, Dept. of Mech. Eng., Rochester, NY 14627 USA

2:00 PM INVITED PAPER

ADHESION AND PROGRESSIVE DEBONDING OF INTERFACES IN THIN FILM STRUCTURES: *Reinhold H. Dauskardt*[1]; [1]Stanford University, Dept. of Mats. Sci. and Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305-2205 USA

The reliability of microelectronic devices and their packages containing thin film structures is strongly influenced by the interfacial adhesion of the many resulting bimaterial interfaces. Further, delayed failure and lifetimes of devices may be dictated by progressive debonding along one (or more) interface. These fractures are driven by residual stresses, thermo-mechanical cycling and mechanical loading. Time dependencies reflect subcritical cracking enhanced by temperature (creep), moisture or corrosive species (stress corrosion), or even cyclic loading (fatigue). Long term reliability and life prediction must be addressed in terms of the above time dependent failure mechanisms. In this presentation, techniques that have been developed to accurately measure the interfacial fracture energy are described for interfaces found in multilayer thin film structures as well as in larger scale packaging structures. Unique time-dependent delamination data for debonding of Si02 interlayer dielectric films from Al lines in interconnect structures will be presented together with progressive debonding behavior of various polymer/metal interfaces measured under static and cyclic fatigue loading conditions. Time dependent delamination data is compared to critical adhesion values. Behavior is rationalized in terms of the salient subcritical debonding mechanism involving environmental interactions and even processes of classic fatigue. The effect of interface morphology, adjacent layer thickness, loading parameters and environment on delamination will be considered. Finally, implications for life prediction are discussed.

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IMPRESSION AND INDENTATION FATIGUE TESTING: *James C. M. Li*[1]; [1]University of Rochester, Mats. Sci., College of Arts, Sciences and Engineering, Rochester, NY 14627 USA

Fatigue experiments were done locally by an indenter to obtain information about the local material properties such as its anelasticity, plastic zone propagation and crack initiation. While under a static load a plastic zone develops but then stops, a cyclic load of the same magnitude can continuously propagate the plastic zone so that the indenter sinks to a steady rate. This steady rate can be affected by a few cycles of overloading or underloading. Generally a cycle of overloading to a higher load and then return to the original load, the initial rate is very low and it takes many cycles at the original load to return to the original state rate. A few cycles of underloading produces the opposite effect. This and the number of cylcles needed to initiate a crack and its relation to anelastic behavior will be reported. Work supported by NSF through DMR 9623808 monitored by Dr. Bruce MacDonald.

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INTERNAL FRICTION ASSOCIATED WITH SURFACE OXIDIZED LAYERS OF METALS: *Masahiro Koiwa*[1]; Osamu Yoshinari[2]; [1]Kyoto University, Dept. of Mats. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan; [2]Nagoya Institute of Technology, Department of Materials Science and Engineering, Showa-ku, Nagoya, Aichi 466 Japan

Internal frictions of metals are often measured by use of a torsion pendulum, the vibrational frequency being typically 1Hz. Since the strain is the largest at the surface of a specimen in a torsional mode, thin surface layers can contribute significantly to the observed internal friction. We have observed the oxygen Snoek peak associated with the surface oxidation of initially oxygen-free specimens of V, Nb and Ta. Some effects associated with the formation of oxide films have been reported for Fe and Co. Such effects of surface layers on the internal friction of metals will be reviewed.

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3:50 PM INVITED PAPER

DAMAGE CHARACTERIZATION OF COPPER/POLYMER CONSTRUCTIONS IN TENSILE, BENDING AND CYCLIC LOADINGS: *Douglas M. Shinozaki*[1]; Harish D. Merchant[2]; [1]University of Western Ontario, Department of Mech. Eng., London, Ontario N6A5B9 Canada; [2]Gould Electronics, 34929 Curtis Boulevard, Eastlake, OH 44095 USA

Thin copper foil, copper film or deposit on polyimide film, polyimide/copper/adhesive/polyimide multilayer and etched copper lines sandwiched symmetrically between the adhesive and polyimide layers are characterized. Following tensile, bending, or cyclic loading, several types of damage are observed: (i) strain localization in polyimide or copper, (ii) delamination between copper and adhesive layers, (iii) surface roughening and notching of copper, (iv) geometrical changes, specifically thinning or necking of copper and (v) edge cracking of copper lines when the line width to thickness ratio is about four or lower. The fatigue cracks originate on the free delaminated copper surface in the tensile strain field and propagate across the line width and through the line thickness. In roll fatigue, the fatigue cracks propagating though copper thickness are arrested at mid-thickness, change direction by 90 degrees and propagate across the line width, significantly enhancing the fatigue life. The cracks generally propagate along grain boundaries, finer grain structure providing a more torturous path for crack propagation.

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INTERNAL FRICTION OF SUPERCONDUCTING YBCO WIRES AT 200KHZ WITH IN SITU HEAT TREATMENT: *Masakuni Ozawa*[1]; M. Inagaki[1]; S. Suzuki[1]; [1]Nagoya Institute of Technology, Ceramics Research Lab, Asahigaoka, Tajimi 507 Japan

We developed a new instrument for the measurement of short wires with a dimension of ca.1mm diameter and 5-10mm length using piezoelectric effect of a LiNbO3 and SiO2 crystal at 100-300kHz. The system consists of impedance analyzer, controlling PC and a furnace. Piezoelectric d-matrix was determined by previous data of LiNbO3 and quartz. This method was applied to measure Young's modulus and internal friction (IF) of a superconducting polycrystalline YBa2Cu3O6+x (YBCO) wire which was subjected to in situ cyclic heat treatment at temperatures of 30-to-500C in air. Two IF peaks were observed, depending the oxygen content of samples. We determined an activation enthalpy and relaxation time of oxygen-site relaxation in YBCO crystal from IF data. This instrument will be applied to the elastic measurement of small ceramic, polymer and metal samples atca.100KHz in various temperature and atmosphere conditions.

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ANALYSIS OF THERMO-MECHANICAL INTERACTION IN MINIATURE SOLDER SYSTEM UNDER CYCLIC FATIGUE LOADING: *Bor Zen Hong*[1]; [1]IBM, Microelectronics Division, 1580 Route 52, Hopewell Junction, NY 12533 USA

Over the past decade, an effort aimed at improving the capability of complex lifetime analysis for miniature solder systems was motivated by a continuous surge of interest in developing reliable solder interconnection structures with applications to the higher I/0 and power density microelectronic packages. Such structures, in particular interest of solder bump type are the flip chip C4 (controlled chip connection collapse), BGA (ball grid array) and CGA (column grid array), could be exposed to various cyclic thermomechanical loads with resulting fatigue failure during manufacturing, test and use in service. Some of the critical lifetime analysis issues, on the macroscopic, microstructural and mesomechanical (interrelation between microstructure response and external loads exerting on the continuum scale of structure) scales, related to the thermo-mechanical interactions of solder systems include the difficulties associated with: (1) characterizing time-dependent, nonlinear deformation response and predicting fatigue life, (2) modeling and analyzing of various coupled and uncoupled thermo-mechanical interactions in the isothermal, adiabatic and anisothermal fatigue environments, and (3) simulating the transient, nonlinear thermal (heat transfer)-structural (thermal stress) response in a convectively-cooled packaging structure under power cycling. In this paper, the goal is set toward threefold and can be summarized as follows: (1) identification of the possible phenomena or mechanisms involved in thermo-mechanical interactions in solder system under cyclic fatigue loads, such as time-dependent deformation induced mechanical energy being transferred into heat and further leads to the counterparts of dissipated processes of viscous loss and thermal conduction loss, and vice versa, (2) literature review on the development of the constitutive models necessary for describing the time-dependent, nonlinear deformation for the tin-based solder alloys, such as lead-contained and lead-free, concentrated on the unified visc oplasticity theories based on internal variables (back or equilibrium stress and drag stress and (3) constitutive modeling with the selected viscoplasticity theories and finite element simulations for the selected examples of miniature solder systems under specific cyclic fatigue loads due to thermo-mechanical interactions.

NANOSTRUCTURED HYBRID MATERIALS: Characterization

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Physical Metallurgy Committee

Program Organizers: Gan-Moog Chow, National University of Singapore, Dept. of Mats. Sci., Kent Ridge, Singapore 117600 Yeukuang Hwu, Institute of Physics, Academia Sinica, Nankang, Taipei Taiwan; Sara Majetich, Carnegie Mellon University, Dept. of Phy., Pittsburgh, PA 15213 USA; Luz Martinez-Miranda, University of Maryland, Dept. of Mats.& Nuclear Eng., College Park, MD 20742-2115 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899 USA

Wednesday PM Room: 16A
March 3, 1999 Location: Convention Center

Session Chair: Sara A. Majetich, Carnegie Mellon University, Dept. of Phys., Pittsburgh, PA 15213 USA

2:00 PM INVITED PAPER
STRUCTURAL, CALORIMETRIC, AND MAGNETIC STUDIES OF AMORPHOUS AND NANOCRYSTALLINE Co SUBSTITUTED Fe-Se-B-Nb-Cu AND Fe-Zr-B-Bu SOFT MAGNETIC MATERIALS: *Karl M. Unruh*[1]; [1]University of Delaware, Dept. of Phys. and Astronomy, Newark, DE 19711 USA

Co substituted Fe-Se-Nb-Cu and Fe-Zr-B-Cu alloys have been prepared by rapid quenching from the melt over a wide range of Fe/Co ratios. Subsequent thermal treatments have transformed the initially amorphous materials into a nanocrystalline sate characterized by a typical grain size of about 10 nm. The evolution of the magnetic properties of both the amorphous and nanocrystalline alloys have been studied form room temperature to above 800ϒC, and the saturation magnetization, Curie temperature, and coercivity determined as a function of the Fe/Co ratio. X-ray diffraction, electron microscopy, and calorimetric measurements have also been carried out and the microstructural information obtained from these studies has been correlated with the magnetic properties. The results of these measurements indicate that Co has indeed been incorporated into the nanocrystalline phase. The observation and the relative stability of the magnetically soft nanophases for temperatures near 400ϒC indicate that these alloys may be potentially useful as high temperature soft magnetic materials.

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DOMAIN BEHAVIOR IN MAGNETIC NANOSTRUCTURES AS REVEALED BY MOIF OBSERVATIONS: *Robert D. Shull*[1]; Alexander J. Shapiro[1]; Valerian I. Nikitenko[2]; Vladimir S. Gornakov[2]; [1]National Institute of Standards & Technology, Metallurgy Division, Bldg. 223, Rm. B152, Gaithersburg, MD 20899 USA; [2]Institute of Solid State Physics, RAS, Chernogolovka, Moscow Region Russia

A magneto-optical indicator film (MOIF) technique has been used for imaging magnetic domains and applied to magnetic nanostructures, including granular metals, magnetic multilayers, and antiferromagnet (AF)/ferromagnet (FM) bilayers. In this technique, the sample domains are imaged by their effect on a garnet film with in-plane magnetization located immediately above the sample. In addition to static domain structures, dynamic information has been obtained by monitoring the domain pattern evolution upon the application of an external magnetic field. Fractal type domain walls were observed in Co/Ag granular metals with a two-step remagnetization process, non-homogeneous nucleation processes were observed in AF/FM bilayers with remagnetization behavior dependent upon field direction, and non-collinear spin configurations were detected in Cu/Co multilayers (electrodeposited on Si substrates) displaying giant magnetoresistance (GMR) effects during the remagnetization process. In these latter samples, the GMR magnitude was correlated with the spin reorientation mechanism. In all samples the effects of crystal lattice defects on the remagnetization process was documented, and found to be significant. The MOIF technique was also found to be capable of detecting not only the domain structure of the surface layer, but also that of subsurface layers in a multilayer morphology. In this presentation, a review of the domain statics and dynamics which have been observed in a variety of nanostructured material types will be discussed. Particular attention will be given to the origin of enhanced coercivity in a bilayer system with unidirectional anisotropy.

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A DEPTH PROFILE STUDY OF THE STRUCTURE OF CHEMICALLY GROWN Cu FILMS ON AlN, AND ITS RELATION TO FILM PROCESSING : *Luz J. Martinez-Miranda*[1]; Yiqun Li[1]; Lynn K. Kurihara[2]; Gan Moog Chow[3]; [1]University of Maryland, Dept. of Mats. and Nuclear Eng., Bldg. 090 Rm 2135, College Park, MD 20742-2115 USA; [2]Naval Research Laboratory/Potomac Research International, Washington, D.C. 20375 USA; [3]Naval Research Laboratory, Washington, D.C. 20375 USA

We have studied Cu coatings grown on AlN substrates via a polyol deposition method using grazing incidence X-ray (GIXS) techniques and small angle scattering techniques to determine the dependence of film structure on depth as well as the presence of nanometer size structures in the films. Small angle measurements indicate the presence of ordered structures in the order of 4 nm close to the surface of the films. Depth studies using different X-ray energies in combination with the GIXS technique suggest the first 20 to 60 nm of the film correspond to a textured region, with strains ranging between +0.1% to -0.6%. The azimuthal ordering in the plane of the films depends on the sample deposition time and the substrate orientation. This work was supported partially by a NSF grant No. ECS-9710789, and by NRL and ONR nanostructured materials programs. X-ray studies were performed at the National Synchrotron Light Source at Brookhaven National Laboratory, which is supported by the U. S. Department of Energy.

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INTERDIFFUSION IN GRADED TiC-Ti MULTILAYERS.: Moshe Peter Dariel[1]; *Itzhak Dahan*[1]; Uri Admon[1]; Joseph Sariel[1]; Naum Frage[1]; [1]Ben-Gurion University, Mats. Eng., Beer-Sheva 84105 Israel

A functionally graded transition zone between a hard TiC coating and a ferrous metal substrate, e.g., a tool steel, can be formed by taking advantage of the stability range of the titanium carbide phase that extends from TiC0.5 to TiC. The transition zone is formed by sputter deposition of a multilayer stack of nanometric TiC and Ti layers. The composition gradient within the carbide layer is generated by varying the relative thickness of the as-deposited Ti and TiC layers within the stack. A subsequent short diffusion treatment eliminates the interfaces between the adjacent layers yet maintains an overall carbon composition gradient across the thickness of the coating. The relative thickness of the individual sputtered layers is adjusted to yield a low carbon composition in the coating close to the substrate and a stoichiometric composition near the external surface. The composition profile within the coating can be further optimized with respect to residual stresses that arise during cooling after the diffusion anneal. In this approach, interdiffusion between adjacent TiC and Ti layers is a crucial processing step. The design of the diffusion treatment parameters requires solving the diffusion equations in the graded multilayer. The relevant diffusion parameters were obtained by x-ray diffraction study of the structural evolution in a multilayer stack with uniform layer thickness, as a function of the temperature and the duration of the diffusion anneal. The effects of interdiffusion between the adjacent layers superpose on recrystallization processes occurring in the as-deposited layers. Noteworthy is also the large departure of the interdiffusion parameters in the sputter-deposited nanometric layers from values extrapolated from high temperature measurements in bulk material.

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4:00 PM INVITED PAPER
MECHANICAL AND TRIBOLOGICAL PROPERTIES OF SPRAY-COATED CONVENTIONAL AND NANOSTRUCTURED WC/Co

COMPOSITES: *Trautgott E. Fischer*[1]; L. Liu[1]; Y. Qiao[1]; [1]Stevens Institute of Technology, MSE, Castle Point on Hudson, Hoboken, NJ 07030-5991 USA

We review the hardness, toughness, adhesive and abrasive wear resistance of WC/Co composites. We briefly review these properties for sintered bulk cermets and the effect of the WC grain size and the benefit of nano structured materials. The hardness of cermets increases and their toughness decreases with decreasing cobalt mean free path. Nanostructured cermets (WC size 70 nm) further increase the hardness without detriment to toughness. Abrasive and sliding wear resistance increase with the hardness of the material, but nanocomposites show an additional increase in abrasion resistance. Coatings applied with the thermal spray method have lower hardness and wear resistance than bulk material because of the lower adhesion between splats. The wear mechanisms are also different, consisting of polishing in sintered materials and removal of splats in the coatings.

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NANOINDENTATION MEASURE OF ADHESION IN LAYERED NANOSTRUCTURES: *Natalia Tymiak*[1]; Nagaraja Rao[2]; Steven Girshick[2]; Joachim Heberlein[2]; William Gerberich[1]; [1]University of Minnesota, Chem. Eng. and Mats. Sci., 151 Amundson Hall, 421 Washington Ave. SE, Minneapolis, MN 55455 USA; [2]University of Minnesota, Mechanical Engineering, 111 Church St. SE, Minneapolis, MN 55455 USA

Processing-structure-adhesion relationships have been established for nanostructured SiC films on Mo. These films have been produced with the novel method of Hypersonic Plasma Particle Deposition (HPPD) which produces a nanostructured compact consisting of 10 nm to 50 nm size particles. In addition, application of the nanoscale thick W overlayers have been incorporated to enhance mechanical properties of the films thereby creating a metal-nanostructured ceramic composite. A bi-layer elastic analysis has been utilized to assess adhesion strength from the indentation induced delamination measurements. Constitutive properties of the SiC films required for the analysis have been evaluated with the nanoindentation method. Before-test and after-test imaging of an indented area was possible with a Hysitron nanoindenter. Combined with the SEM results, this allowed correlation of the film structure with the observed mechanical property gradients across the film thickness. Measured interfacial fracture toughness has been correlated to the near-substrate film structure as revealed by SEM cross-sectional analysis. Adhesion strength have been found to increase with the increasing substrate temperature which was identified as the most critical deposition parameter.

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NANOSTRUCTURED Ni COATINGS DEPOSITED BY THE POLYOL PROCESS: *Gan Moog Chow*[1]; S. H. Lawrence[2]; C. R. Feng[2]; [1]National University of Singapore, Mats. Sci., Kent Ridge 119260 Singapore; [2]Naval Research Laboratory, Mater. Sci. and Technol., Code 6323, Washington, DC 20375 USA

Nanostructured nickel coatings were deposited on copper substrates by reducing nickel acetate in refluxing ethylene glycol. This process has been used to prepare nanostructured composite powders and films. The Ni coatings were studied using x-ray diffraction, scanning electron microscopy, transmission electron microscopy, high resolution transmission electron microscopy, energy dispersive x-ray fluorescence spectrometry, and Vickers hardness testing. The average crystallite size and the morphology of the coatings were studied as a function of deposition time at a fixed concentration. The effects of heterogeneous nucleating aids were also investigated. The crystallite size was found to initially increase with deposition time, and then decrease at a longer deposition time. The crystallite size was reduced when the nucleating aids were employed. Microhardness data revealed softening for small crystallite size.

5:10 PM

CONTINUUM BASED MODELS OF INTERFACES IN NANOSTRUCTURED HYBRID COATINGS: *I. A. Ovid'ko*[1]; [1]Russian Academy of Sciences, Laboratory for Theory of Defects in Materials, Institute for Problems of Mechanical Engineering, Bolshoj 61, Vas. Ostrov, St. Petersburg 199178 Russia

This report reviews continuum based models of interfaces - intergranular and interphase boundaries - in nanostructured materials with the special attention being paid to specific peculiarities of interfaces in metallic and ceramic phases in nanostructured hybrid films and coatings. Interfacial defect structures as well stress-field and energetic characteristics of interfaces are considered. The nano-scale grain size effect in nanostructured hybrid films and coatings is discussed.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Solid Freeform Fabrication

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee; Shaping and Forming Committee; Structural Materials Division, Structural Materials 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

Wednesday PM Room: 7B
March 3, 1999 Location: Convention Center

Session Chairs: D. B. Chrisey, Naval Research Laboratory, Washington, D.C. USA; J. C. Arnault, Groue Surfaces-Inerfaces, Instut de Physique et Chimie de Straabourg France

2:00 PM

LASER ENGINEERED DIRECT FABRICATION/MANUFACTURING OF METALS: *John E. Smugeresky*[1]; D. M. Kelcher[2]; [1]Sandia National Laboratories, Livermore, CA 87123 USA; [2]Optomec Design Company, Albuquerque, NM 87123 USA

Direct Fabrication Technology, which utilizes computer aided design (CAD) solid models to automatically produce functional piece parts, is rapidly gaining popularity as a means to significantly reduce the time to market of new products. Since the introduction of stereolithography in 1982, several Rapid Prototyping (RP) technologies have evolved. These RP technologies rely almost exclusively on the use of surrogate rather than actual materials of construction to create models as an intermediary step to impact the manufacturing process. These models provide a means for designers to quickly realize their designs and also provide reasonable patterns for processes such as castings. Unfortunately, these models produced on RP systems are typically constructed from polymers or other low strength materials that do not allow them function in very demanding situations. Based on the successes of these early RP methods, researchers have more recently begun to develop laser-based methods to obtain fully dense metallic components directly from a CAD solid model thus eliminating intermediary processing steps. These laser-based methods have demonstrated that near-net-shaped components can be fabricated directly from a CAD solid model in a variety of useful materials. Furthermore, components fabricated using these techniques exhibit material properties equal, or superior to those of similar composition components fabricated using conventional methods. Many of these methods hold a great deal of promise for revolutionizing the approach to manufacturing. Although systems to apply this technology are currently research based, efforts are underway to bring this technology to the commercial manufacturing floor. In this article, an overview of the current state of Laser Engineered Direct Fabrication/Manufacturing technologies is given, demonstrating we may be closer to acceptance of these technologies as a preferred method of manufacture than is commonly believed.

2:20 PM

LASER ENGINEERED NET SHAPING (LENS♦) OF INTERFACES OF FUNCTIONALLY GRADED MATERIALS: *J. E. Smugeresky*[1]; D. M. Keicher[2]; J. A. Romero[3]; M. L. Griffith[3]; [1]Sandia National Laboratories, Livermore, CA 94551 USA; [2]Optomec Design Company, Albuquerque, NM 87185 USA; [3]Sandia National Laboratories, Albuquerque, NM 87185 USA

This paper describes recent developments in LENS® (Laser Engineered Net Shaping) technology, to design the interfaces of functionally graded materials in order to provide electrical isolation in components built directly from CAD solid models. The current LENS® process uses a moving laser beam to fuse solid material particles together by creating a small liquid puddle on a substrate. The solid particles are then injected into this puddle where they dissolve, increasing the volume of the puddle to create the deposited layer by resolidification as the laser beam is moved away. For electrical isolation, depositing liquid particles onto a solid surface may be required. In this work, we examine the mixing of two materials and identify the processing conditions that allow an abrupt material transition to occur at the interface without first melting the substrate. A simple model was developed to predict conditions for melting the particles as they are propelled through the high power laser beam. Statistically designed experiments were used to identify the key process variables critical for achieving good material properties and an abrupt interface transition. The bend tests, optical and transmission electron microscopy for microstructure evaluation, and composition profiles used to assess the integrity of the bonded interfaces are discussed. These results are compared to conventional LENS® processed homogeneous materials. Work supported by the U. S. Department of Energy under contract DE-AC04-94AL85000, and Ballistic Missile Defense Organization SBIR under contract DASG60-97-M-0107

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SLIP CASTING OF RAPID INJECTION MOULDING TOOLS: *Christopher Charles Ainsley*[1]; [1]Nanyang Technological University, School of Mechanical & Production Engineering, Nanyang Avenue, Singapore 639798

This paper discusses how the slip casting process can be used as a forming process for injection moulding tools. Descriptions of the process and the effects of the slip casting parameters on the final casts are given with particular reference to the dimensional tolerances of the final castings. The results show that the slip casting process is suitable for the formation of small injection moulding tools with a 0.12% dimensional tolerance and low distortion. Also shown is the effect casting rate has on the shrinkage of the green parts as well as surface finish. Finally a discussion of the possible befits the slip casting process has over other tooling methods is given.

3:00 PM INVITED PAPER

ORIENTED SHORT FIBER COMPOSITES BY EXTRUSION FREEFORM FABRICATION: Paul Calvert[1]; *Jiong Peng*[1]; [1]AML, 4715 E. Fort Lowell Rd., Tucson, AZ 85712 USA

Extrusion freeform fabrication can be used to build parts from epoxy resins. The resin is blended with a hydrophobic fumed silica to give it the rheology of toothpaste. The part is built and subsequently cured. The same approach can be used to form composite materials with carbon or glass fibers up to about 1mm in length. Moduli up to 3 times that of the base resin can be obtained at a fiber volume fraction of 20%. During the forming process the fibers become closely oriented with the direction of motion of the write-head. This allows fiber orientation to be varied local within the part. Elastic modulus and strength have been measured and are very dependent on the angle between the fiber direction and stress direction. The use of mixed reinforcing fillers to maximize modulus and the combination of soft and tough layers to improve strength will also be discussed.

3:30 PM BREAK

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COMPLEX STRUCTURAL COMPOSITES FOR BIOMEDICAL APPLICATIONS: *Robert Sinclair Crockett*[1]; Lisa Milkowski[1]; Vito Gervasi[1]; [1]Milwaukee School of Engineering, Rapid Prototyping Center, 1025 N. Broadway, Milwaukee, WI 53202 USA

Solid Freeform Fabrication techniques have been used to produce extremely complex composites that are well-suited for replicating the non-homogeneous mechanical properties of bone. The process begins with Stereolithography patterns consisting of open cellular structures inside a surface shell. Geometry for major structures (e.g. outside shape and regions of various bone densities) are obtained from CT or MRI medical images. Composites are created by using the SFF pattern as a host for filler materials; regions within a single object may be separated by thin barriers, allowing filling with different matrix materials to create regions of differing local properties. The internal structure can also be continually gradiated in thickness to produce composites with properties ranging from that of the filler material to that of the Stereolithography epoxy. Current fillers include epoxy matricies loaded with glass microspheres, as well as hydraulically-bonded ceramic mixtures with bulk properties matched to various bone structures. Results of initial experiments on composites with both discrete and continually gradient properties are presented.

4:05 PM

DIRECT LASER FABRICATION OF HIGH PERFORMANCE METAL COMPONENTS VIA SLS/HIP: *Suman Das*[1]; Joseph J. Beaman[1]; Martin Wohlert[1]; David L. Bourell[1]; [1]University of Texas at Austin, Laboratory for Freeform Fabrication, Mechanical Engineering Dept., ETC 5.160 C2200, Austin, TX 78712-1063 USA

This paper focuses on recent advances in direct freeform fabrication of high performance metal components via selective laser sintering (SLS). The application, known as SLS/HIP, is a low cost manufacturing technique that combines the strengths of selective laser sintering and hot isostatic pressing (HIP) to rapidly produce low volume or "one of a kind" high performance metal components. Direct selective laser sintering is a rapid manufacturing technique that can produce high density metal parts of complex geometry with an integral, gas impermeable skin. These parts can then be directly post-processed by containerless HIP. The advantages of in-situ encapsulation include elimination of a secondary canning step and container material, no container-powder interaction, reduced pre-processing time, a short HIP cycle and reduction in post-processing steps compared to HIP of canned parts. SLS/HIP is currently being developed for INCONEL 625 superalloy and Ti-6Al-4V under a DARPA/ONR program. Microstructure and m echanical properties of material processed by SLS/HIP are comparable to conventionally processed material. The potential of SLS/HIP technology has been demonstrated by fabricating a Titanium guidance section housing for the AIM-9 Sidewinder missile.

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DIRECT LASER FABRICATION OF NICKEL SUPERALLOY CERMET TURBINE ENGINE COMPONENTS: *Suman Das*[1]; Timothy P. Fuesting[2]; Joseph J. Beaman[1]; David L. Bourell[1]; [1]University of Texas at Austin, Laboratory for Freeform Fabrication, Mechanical Engineering Dept., ETC 5.160 C2200, Austin, TX 78712-1063 USA; [2]Allison Engine Company, 2001 South Tibbs, Speed Code W08, Indianapolis, IN 46241 USA

This paper presents the development of a new technique for the production of cermet abrasive turbine blade tips by direct laser processing. These components form part of the low pressure turbine sealing system in an IHPTET demonstrator engine being developed at Allison Engine Company. The influence of laser processing parameters on resulting solidification microstructure and mechanical properties will be presented. Direct laser fabrication of a prototype lot of 100 blade tips was completed successfully. The direct laser fabrication technique results in superior performance and 67% cost reduction over the currently employed production technique. This is the first instance of a direct SFF method applied to the production of functional engine hardware. The next stage of this research is focusing on directly laser fabricating abrasive blade tips onto turbine blades.

4:45 PM

UNDERSTANDING THERMAL BEHAVIOR IN LENS PROCESSING OF STRUCTURAL MATERIALS: *Michelle Griffith*[1]; Lane Harwell[1]; M. Eric Schlienger[1]; John Smugeresky[2]; William Hofmeister[3]; [1]Sandia National Labs, P.O. Box 5800, MS 0958, Albuquerque, NM 87185 USA; [2]Sandia National Labs, P.O. Box 969, Livermore, CA 94551

USA; [3]Vanderbilt University, Dept. of Chemical Engineering, P.O. Box 1604 Station B, Nashville, TN 37235 USA

In direct laser metal deposition technologies, such as the LENS process, it is important to understand and control the thermal behavior during fabrication. Without control of the thermal behavior, components cannot be reliably fabricated with sound material properties. This talk will describe the use of contact and imaging techniques to monitor the thermal signature and history during LENS processing. Recent results show a direct correlation between thermal history and resulting material properties. Microstructural evolution, mechanical properties, and residual stress results will be shown. Development of an understanding of solidification behavior, residual stress, and microstructural evolution with respect to thermal behavior through modeling will be discussed.

SYNTHESIS OF LIGHTWEIGHT METALS III: Aluminum

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. G. McCormick, University of Western Australia, Res. Ctr for Adv. Min. & Mats. Proc., Nedlands, W.A. 6907 Australia

Wednesday PM Room: 10
March 3, 1999 Location: Convention Center

Session Chairs: M. Amateau, Penn State University, Applied Research Laboratory, State College, PA 16804-0030 USA; E.G. Baburaj, University of Idaho, IMAP, Moscow, ID 83844-3026 USA

2:00 PM INVITED PAPER

OVERVIEW OF ALUMINUM - PART I: *John Liu*[1]; F.H. (Sam) Froes[2]; [1]Aluminum Company of America, Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The current status of aluminum science, technology and applications will be discussed. This will cover both modifications which have been made to conventional ingot alloys and the development of new families of ingot alloys. It will also include production of aluminum based materials by nonconventional techniques such as Rapid Solidification, Mechanical Alloying and Vapor Deposition. Consideration will also be given to composite concepts including particulate, fiber and lamellae reinforcements.

2:20 PM INVITED PAPER

OVERVIEW OF ALUMINUM - PART II: *John Liu*[1]; F. H. (Sam) Froes[2]; [1]Aluminum Company of America, Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The current status of aluminum science, technology and applications will be discussed. This will cover both modifications which have been made to conventional ingot alloys and the development of new families of ingot alloys. It will also include production of aluminum based materials by nonconventional techniques such as Rapid Solidification, Mechanical Alloying and Vapor Deposition. Consideration will also be given to composite concepts including particulate, fiber and lamellae reinforcements.

2:40 PM

SPRAY METAL PROCESSING OF Al-Ce-Co-Cr ALLOYS: *Maurice F. Amateau*[1]; Timothy J. Eden[1]; Michael J. Kaufman[2]; Jay M. Galbraith[1]; Eric J. Fodran[1]; Raymond S. Baker[1]; [1]The Pennsylvania State University, Applied Research Laboratory, Mats. Sci. Division, P.O. Box 30, State College, PA 16804-0030 USA; [2]University of Florida, Dept. of Mats. Sci. and Eng., 132 Rhines Hall, P.O. Box 116400, Gainesville, FL 32611-6400 USA

Al-Ce-Cr-Co alloys processed by rapid solidification rate (RSR) methods exhibit great potential for high strength and high temperature applications. In this work the processing of these alloys by spray metal deposition was investigated to determine if microstructure and mechanical properties similar to RSR processing could be achieved. The effect of processing variables including superheat temperature, gas-to-metal ratio and second particle injection on the resulting microstructure and properties were examined. Processing variables were found to have a pronounce influence on droplet size, dispersoid size, composition and volume fraction. Strength properties to 315ϒC were determined and discussed in terms of microstructure and phase characteristics.

3:00 PM

A NEW APPROACH IN HARDENING OF Al-Fe ALLOYS: *O. N. Senkov*[1]; F. H. (Sam) Froes[1]; V. Stolyarov[2]; R. Z. Valiev[2]; M.D.S. Pirzada[1]; J. Liu[3]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Ufa State Aviation Technical University, Institute of Physics and Advanced Materials, K. Marks 12, Ufa, 450000 Russia; [3]Aluminum Company of America, ALCOA Technical Center, 100 Technical Drive, Alcoa Center, PA 15069-0001 USA

Aluminum-iron alloys are attractive for engine application. Unfortunately, the equilibrium solubility of iron in the aluminum lattice is very low even at high temperatures, and these alloys cannot be dispersion-strengthened with the use of conventional thermal treatments. In the present work, the severe plastic deformation approach has been used to extend the iron solubility in a submicrocrystalline aluminum matrix. This allowed aging of the aluminum-iron alloys (5 to 16 wt.% Fe), which resulted in a very high hardness and strength. The effect of aging time and temperature on microhardness of the alloys was also studied.

3:20 PM BREAK

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EFFECT OF MODIFIERS ON THE AS CAST MICROSTRUCTURE OF A390: *E. G. Baburaj*[1]; Jeff Hill[2]; Robert Osborne[2]; *F. H. (Sam) Froes*[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm., 321, Moscow, ID 83844-3026 USA; [2]LA Aluminum Casting, W. 1905 Miles Avenue, P.O. Box 250, Hayden Lake, ID 83835 USA

The hyper-eutectic alloy A390 (Al-17.5Si-0.5Fe-4.5Cu-0.1Mn-0.5Mg-0.1Zn) is the potential choice material for casting automobile cylinders because of its good fluidity, solidification range, strength, rigidity, thermal conductivity, abrasion, wear, and high temperature corrosion resistance along with low thermal expansion coefficient. The extensive use of this alloy is limited by its low ductility. The present work is an attempt to improve the ductility through the control of microstructural features of A390 by the addition of trace elements.

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SECONDARY PHASE SELECTION IN 1XXX ALUMINUM ALLOYS: AN OVERVIEW: *C. M. Allen*[1]; K. A. Q. O' Reilly[1]; B. Cantor[1]; P. V. Evans[2]; [1]University of Oxford, Dept. of Mats., Oxford Centre for Advanced Materials and Composites, Parks Rd., Oxford OX1 3 PH UK; [2]Alcan International, Ltd., Banbury Laboratory, Southam Rd., Banbury, Oxon OX16 7SP UK

1xxx series Al alloys are used in a wide variety of wrought applications, including packaging foil, anodised sheet for architectural use and lithographic printing sheet. These products are commonly direct chill (DC) cast to form ingots, which are then heat treated and rolled to the required final gauge. Alloying additions and impurities together constitute typically <1wt% of the alloy composition. Consequently the as-solidified microstructure contains only ~1vol% of secondary phases, in the form of Fe and Si based aluminides. These aluminides however influence material properties such as strength, resistance to fracture, ductility and surface electrochemistry, which in turn affect downstream

processing requirements and final bulk and surface properties. Model 1xxx Al alloys have been rapidly solidified to produce a dispersion of submicron phases entrained in an Al matrix. On heating these phases melt eutectically to produce a highly dispersed liquid in a solid Al matrix. On subsequent cooling therefore the nucleation requirements for solidification are much exaggerated. The solidification of the dispersed liquid can be studied calorimetrically, which provide valuable information on the change of phase content with changes in alloy trace chemistry and solidification rate. The dispersion technique has shown that <100ppm of V impurity in combination with Al-Ti-B grain refiner addition promotes the formation of a metastable phase, responsible for the well know 'fir-tree' surface defect in DC castings. This unequivocally demonstrates that certain impurities and grain refiners have a potentially strong influence on the nucleation aspects of secondary phase selection during conventional solidification, which until now have largely been overlooked or poorly understood.

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NANOCRYSTALLINE Al-Ni-Y ALLOYS: *M. Gogebakan*[1]; P.J.S. Warren[1]; B. Cantor[1]; [1]University of Oxford, Dept. of Mats., Oxford Centre for Advanced Materials & Composites, Parks Road, Oxford OX1 3PH UK

This paper describes the manufacture of amorphous and nanocrystalline Al-Ni-Y alloys by a combination of melt spinning and heat treatment. The effects of alloy composition and quenching rate are discussed in detail. The crystallisation process has been investigated by a combination of differential scanning calorimetry, transmission electron microscopy, X-ray diffractometry and kinetic analysis.

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

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: Robert Schiffman, R.S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica, Bologna I-40126 Italy

Thursday AM Room: 15B
March 4, 1999 Location: Convention Center

Session Chair: Michael Wargo, NASA Headquarders, Washington, DC USA

8:30 AM
OVERVIEW OF ALTERNATE CONTAINERLESS TECHNIQUES: *Rick Weber*[1]; [1]Containerless Research, Inc., 906 University Place, Evanston, IL 60201-3149 USA

Containerless processing methods provide a high-purity environment to study high-temperature materials and non-equilibrium states of matter. This talk will identify and review containerless processing techniques which are based on technologies not represented by ELF, TEMPUS, or Space-DRUMS. The technology issues and research capabilities associated with each will be assessed.

8:50 AM
OPTICAL DIAGNOSTIC TOOLS: *Shankar Krishnan*[1]; [1]Containerless Research, Inc., 906 University Place, Evanston, IL 60201-3149 USA

Optical diagnostic tools represent a key technology required for containerless processing. Optical pyrometry provides temperature measurement. Optical position sensing devices are required for controlling specimen location during processing. High-performance cameras provide data for thermophysical property measurement.

9:10 AM
LESSONS LEARNED FROM CONTAINERLESS PROCESSING FACILITIES: *Jan Rogers*[1]; [1]NASA Marshall Space Flight Center, Mail Code ES76, MSFC, AL USA

Containerless processing experiments have been performed in several reduced gravity settings and in ground-based laboratories. Lessons learned from acoustic, electromagnetic, and electrostatic levitation devices are valuable for future containerless research. There will be a panel discussion on this topic.

9:30 AM
DEFINITION OF TARGETS FOR SCIENTIFIC RESEARCH USING LEVITATION TECHNIQUES: *Michael Wargo*[1]; [1]NASA Headquarters, Code UG, Washington, DC 20546 USA

Targets identified will include materials of interest, scientific data which can be obtained, and technical requirements for levitation. There will be a panel discussion on this topic.

9:50 AM BREAK

10:10 AM
DEFINITION OF SPECIFIC EXPERIMENT TYPES FOR EACH ISS LEVITATION FACILITY: *Shinichi Yoda*[1]; [1]NASDA, Space Utilization Research Center, Sengen, 2-1-1, Tsukuba-shi, Ibaraki-ken 305 Japan

Each levitation facility will provide unique environments for performing containerless research. An attempt will be made to categorize experiments which are best performed in each of the planned facilities. There will be a panel discussion on this topic.

10:40 AM
IDENTIFICATION OF DIAGNOSTIC TOOLS FOR ISS CONTAINERLESS RESEARCH AND COMMON REQUIREMENTS: *Rainer Kuhl*[1]; [1]DLR, Koenigswinterer4, Str. 522-52, Bonn 53227 Germany

To take advantage of the full capabilities of containerless facilities, a number of diagnostic tools must be developed and added. A discussion of the existing and desired diagnostic techniques will be included. There will be a panel discussion on this topic.

11:10 AM
RECOMMENDATIONS FOR FACILITY MODIFICATIONS TO ENHANCE SCIENCE: *Philip Gregory*[1]; [1]Canadian Space Agency, 6767 Route de l'Aeroport, St-Huber, Quebec J3Y 8Y9 Canada

Facility modifications may be necessary to accomplish on-going research in the planned containerless facilities. A discussion of the compatibility and desired modifications will be included. There will be a panel discussion on this topic.

11:40 AM
IDENTIFICATION OF LEVEL OF INTEREST IN SPECIFIC TECHNIQUES OR EXPERIMENT TYPES: *Ivan Egry*[1]; [1]DLR, WB-RS, Institut fuer Raumsimulation, Cologne, 0551140 Germany

Since the planned containerless facilities will service a wide ranging international community, a discussion of the level of interest for each facility as appropriate to each research group will be included. There will be a panel discussion on this topic.

ALUMINUM REDUCTION TECHNOLOGY: Fundamental Studies

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

Thursday AM Room: 6F
March 4, 1999 Location: Convention Center

Session Chair: Jean-Paul Huni, Alcan International, Ltd., Arvida Research & Development Centre, Jonquiere, Quebec G7S 4K8 Canada

8:30 AM
A TWO ELECTRON PROCESS PRODUCING CO AND A FOUR ELECTRON PROCESS PRODUCING CO_2 DURING ALUMINIUM ELECTROLYSIS: *T. S. Sorensen*[1]; S. Kjelstrup[2]; [1]DTH, Physical Chemistry, Modeling and Thermodynamics, Norager Plads 3/DTH, Copenhagen-Vanlose, Sjaelland DK2720 Denmark; [2]NTNU, Physical Chemistry, S. Saelands vei 14, Trondheim, Troendelag N-7034 Norway

Earlier measurements of anode potentials in the Hall-Heroult cell, have been re-analyzed in terms of two parallel Butler-Volmer processes. The RI-corrected electric potential differences between a working car-

bon anode and a non-working Al reference electrode were measured. The current density is found as the sum of Butler-Volmer expressions for a two electron process (producing CO) and a four electron process (producing CO_2). Each process is associated with its own overpotential. We propose that the $Al_2O_2F_4)^{2-}$ ion is responsible for the CO_2 production. This ion forms an activated complex with an O-atom placed on each side of a reactive surface carbon atom. The negative, effective activation energy for the n=4 process is explained by the thermodynamic behaviour of this species in the acid NaF-AlF_3-CaF_2-Al_2O_3 melt. The Pearson-Waddington relation overestimates the percentage of CO_2 by disregarding electrolytically produced CO which dominates at lower potentials (current densities).

8:55 AM

THE KINETICS AND MECHANISM OF THE ELECTRODE REACTIONS IN ALUMINIUM ELECTROLYSIS: *A. Kisza*[1]; J. Kazmierczak[1]; J. Thonstad[2]; T. Eidet[2]; J. Hives[3]; [1]University of Wroclaw, Faculty of Chemistry, 14 Joliot, Curie, Wroclaw 50383 Poland; [2]NTNU, Dept. of Electrochemistry, 7034 Trondhelm Nowway; [3]SUT, Dept. of Inorganic Technology, Bratislava 81237 Slovakia

With the use of electrochemical techniques, the kinetics and mechanism of the cathodic and anodic reactions in aluminium electrolysis was determined at several electrolyte compositions, and in particular in a typical industrial bath, i.e. cryolite with 11 wt% AlF_3, 3 to 5 w% CaF_2 and variable alumina concentrations at 1000℃. A three step electrode process was observed for the cathodic reaction, comprising a preceeding chemical reaction followed by two charge transfer steps. The exchange current density of the cathodic reaction was found to be dependent upon the concentration of the aluminium fluoride ionic species. Also the anodic reaction was found to proceed according to a three step mechanism involving a preceding chemical reaction and two charge transfer steps with intermediate adsorption. The experimental data gave the double layer capacitance, electrode coverage, effective rate constants, the charge needed for the coverage of the electrode by a monolayer and coefficients of the Tafel equation. The role of the two additives, AlF_3 andCaF_2 was evaluated.

9:20 AM

VISCOSITY OF OXYFLUORIDE MELTS RELATED TO THE DETERIORATION OF REFRACTORY LININGS IN ALUMINUM REDUCTION CELLS: *T. Grande*[1]; J. Rutlin[1]; [1]Norwegian University of Science and Technology, Inorganic Chemistry, Trondheim N-7034 Norway

Molten fluoride attack on refractory pot linings increases the energy consumption and may in extreme cases terminate the pot life. The diffusion of molten fluorides down into refractory pot linings is determined by the viscosity of the melt which penetrates/reacts with the refractory lining. In the present paper estimated viscosities of molten mixtures of fluorides and sodium aluminum silicates are reported. The viscosity is estimated by use of a semi-empirical theory which relates viscosity to heat capacity of melts in the glass transition region. The viscosity of the fluorides is increasing several orders of magnitude when albite and nepheline are dissolved in molten fluorides. The present findings demonstrate that the penetration of molten fluorides into traditional fireclay refractories is strongly retarded due to the formation of a viscous oxyfluoride layer between the cathode and the refractory lining. Finally, the formation of the viscous layer is discussed in terms of the silica-content in the refractory lining.

9:45 AM

LABORATORY CHARACTERIZATION OF THE INTERACTIONS BETWEEN CRYOLITIC BATHS AND REFRACTORIES: *G. Oprea*[1]; [1]University of British Columbia, Metals and Materials Engineering, 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Refractories in the SiO_2 - Al_2O_3 system were investigated using a testing method with six different preparation procedures for bricks, mortars and dry-barrier powders and various testing conditions. The corrosion results were correlated with chemical and mineralogical compositions, at the refractory - melt interface, for couples refractory - bath, ranging from 20 to 50% Al_2O_3 for refractories and 1.2 to 2.5 bath ratio by weight for the cryolitic bath. A "corrosion criterion" was defined based on individual corrosion parameters and used in evaluating the performance of a refractory material in contact with molten cryolitic baths.

10:10 AM BREAK

10:30 AM

ACTIVITY OF ALUMINA IN THE SYSTEM NAF - AlF_3 - Al_2O_3 AT NaF/AlF_3 MOLAR RATIOS RANGING FROM 1.4 TO 3: *A. Solheim*[1]; Å. Sterten[2]; [1]SINTEF, Materials Technology, Trondheim N-7034 Norway; [2]Norwegian University of Science and Technology, Dept. of Electrochemistry, Trondheim N-7034 Norway

The activities in the ternary liquid system NaF - AlF_3 - Al_2O_3 were derived. The results are presented in the form of two sets of empirical equations; one set describing the activity of each component at 1300 K as a function of the melt composition, and the other set describing the temperature dependence of the activities. The derivation was based on mathematical functions attributed to each component, those functions being consistent with the Gibbs-Duhem equation. By varying the constants in the functions, finally good agreement was obtained with experimental data available in the literature, such as heat of dissolution, liquidus temperature, alumina solubility, vapour pressure, and emf of electrochemical concentration cells.

10:55 AM

LOWERING THE ANODIC OVERVOLTAGE BY DOPING THE CARBON ANODES IN ALUMINIUM ELECTROLYSIS: *J. Yang*[1]; Q. Zhang[1]; J. Thonstad[2]; Y. Liu[1]; [1]Central South University of Technology, Dept. of Metallurgy, Changsha, Hunan Province 410083 China; [2]Norwegian University of Science and Technology, Dept. of Electrochemistry, Trondheim N-7034 Norway

A laboratory study has shown that the anodic overvoltage in aluminium electrolysis can be lowered by adding doping agents to the carbon anodes. For anodes doped with 1 wt.% AlF3 or 0.5 wt.% MgAl2O4, the anodic overvoltage at 0.8 A/cm2 decreased by 50-70 mV compared with undoped anodes, in a melt with Na3AlF6 - 11 wt% AlF3 - 5 wt% CaF2 - Al2O3 (sat.) at 970°C. An improved current interruption technique and a special cell configuration were used. A substantial increase in anodic overvoltage was observed by bubbling CO through the melt. The air and CO2 reactivities of the doped anodes were markedly lower than that of identical undoped anodes.

11:20 AM

ACTIVITIES IN THE SYSTEM KF-AlF_3: *Ø. T Gustavsen*[1]; Terje Østvold[1]; [1]NTNU, Dept. of Inorganic Chemistry, Sem Sælandsv. 12, Trondheim, 7034 Norway

Activities in the system KF-AlF_3 have been measured. In the acidic melts (CR<3) activities of $KAlF_4$ were calculated from vapour pressures and vapour composition data. In the basic part of the diagram (CR>3), the vapour pressures are, however, too low to give accurate pressure data. For this range a model which give vapour pressures of $KAlF_4$ from composition data of the vapour over the melts and the standard vapour pressure of KF has been developed. Activities in the basic region can be obtained from these calculated pressures. The activities of KF are obtained from the activities of $KAlF_4$ using the Gibbs-Duhem equation. Thereafter, mole fraction based activity coefficients of $KAlF_4$ and KF are calculated as gamma(i)=a(i)/x(i) , where x(i) are the real mole fractions as obtained by Raman spectroscopy.

11:40 AM

DISSOLUTION OF ALUMINA IN CRYOLITE BATH: *Z. Qiu*[1]; Z. Yang[1]; Z. Wang[1]; W. Li[1]; B. Kao[1]; X. Sun[1]; [1]Northeastern University, Dept. of Non-ferrous Metallurgy, Shenyand 110006 China

In this paper we report our experimental results of alumina dissolution in molten cryolite. The dissolution process was observed in a laboratory see-through cell. The dissolution process consists of several stages: (1) Floating of cold alumina on the surface of molten bath, (2) Disintegration of the floating alumina layer into fine alumina particles and flakes of alumina-cryolite agglomerates, (3) Dissolution of alumina in the bulk of the molten bath during its settling from the floating layer, and at the cell bottom after it settles down there. The dissolution time and behaviour of alumina were recorded with a video camera.

CAST SHOP TECHNOLOGY: Equipment & Handling

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

Thursday AM Room: 6C
March 4, 1999 Location: Convention Center

Session Chair: Edmund Hemmersbach, Commonwealth Aluminum Corp., Lewisport, KY 42351-0480 USA

8:30 AM
THE ANATOMY OF A CAST SHOP PROBLEM SOLUTION: *George J. Binczewski*; [1]SC Systems, P.O. Box 6154, Moraga, CA 99570 USA

Changing environmental concerns together with their inevitable regulations and restrictions often present complex and vexing problems to the cast shop practitioner. Invariably, this requires process and procedural modifications which almost always result in cost increases. By referencing a series of seemingly unrelated cast shop technological evolvements spanning a period of twenty years, an innovative method was developed. It utilized the application of a common material to solve a serious concern. Importantly, the material exhibits characteristics which offer significant potential for other cast shop operations.

8:55 AM
MOLTEN METAL CONTACT TESTING OF MORTAR JOINTED FURNACE BRICKS: *A. G. Furness*[1]; A. J. Forde[1]; [1]Alteck Limited, 2 Becton Mead, Becton Lane, Barton on Sea, New Milton, Hants BH25 7DL UK

In the aluminium industry it is common knowledge that the conversion of bricked furnace linings to a-AI2O3 is frequently initiated at the mortar joints. The question of how best to assess the behavior of mortar joints in molten aluminium, initiated a review of published techniques for the liquid metal testing of refractories and from these, the method to be utilized was finally selected. That most appropriate to mortar jointed samples was the Immersed Finger Test (IFT), in which individual 25 X 25 X 225mm refractory samples are partially immersed in a 1 kg crucible of the preferred alloy, at a selected temperature for no less than 7 days. Daily metal sampling of each crucible is carried out to determine if any alloy contamination had taken place. A novel method was employed to maintain permanent joints between the bricks/mortar sandwich. This enabled 42%, 80% Al2O3 and an aluminium resistant brick to be tested, at 900°C, each with two different mortars both in A.A 6063, the Worlds largest tonnage alloy and A.A7075, known to be particularly aggressive to refractories. The results demonstrated that in most cases the 42 & 80% Al2O3 bricks suffered significant a-AI2O3 conversion in contact with both alloys, leaving the mortar largely unaffected. The aluminium resistant brick showed minor conversion against 7075. The integrity of brick/mortar joints was maintained during and after all tests, also no significant difference existed in the degree of conversion relative to the 'as made' or cut faces of the test samples.

9:20 AM
OPTIMIZING BURNERS FOR ALUMINUM MELTING AND HOLDING FURNACES WITH ULTRA LOW NO_X DESIGNS: *Frank L. Beichner*[1]; [1]Bloom Engineering Company, Inc., Sales Engineer - Non-Ferrous Products, 5460 Horning Rd., Pittsburgh, PA 15236 USA

Recent EPA restrictions on POC emissions from aluminum melters and holders have necessitated burner designs with extremely low NOx and CO emission levels along with flame patterns to provide optimum melting. Recent breakthroughs in burner technology has led to the development of the recuperative LumiFlameä design that uses two modes of operation based on the furnace operating and melting requirements. This design provides a high intensity low NOx burner design operating mode for melting the initial charge, along with a LumiFlame ultra low NOx mode to be used when raising the molten bath to pour temperature. With this design there is no compromise with furnace performance when extremely low NOx emissions are required.

9:45 AM
VORTEX CHARGE WELL WITH GAS LIFT PUMP AND GRAVITY FURNACE CHARGER: *Larry D. Areaux*[1]; [1]Premelt Pump, Inc., 2205 Miller Road, Kalamazoo, MI 49001 USA

This paper presents the latest technological development in high capacity aluminum scrap melting in a reverberatory furnace with melt yields above 96% when charging aluminum chips, foil or UBC. The process combines submerged charging through a self-leveling Charge Well Cover, a Vortex Charge Well and a nitrogen operated, Gas Lift Molten Metal Circulating Pump. Employed to produce the Vortex stirring action while circulating several thousand pounds of molten aluminum per minute throughout the main chamber of the melt furnace. This development creates an oxygen deprived environment in which aluminum scrap in its lightest fraction can be continuously submerged into a bath of circulating molten metal for rapid conversion into its liquid state. The inert gas being discharged from the Molten Metal Circulating Pump below the Charge Well Cover, provides additional benefits in achieving the exceptionally high metal quality and melt yield.

10:10 AM BREAK

10:30 AM
CONTINUOUS IN-LINE MONITORING OF Mg CONTENT IN ALUMINUM MELTS: *Johan Vangrunderbeek*[1]; Pieter Lens[1]; Paul Verstreken[2]; Cees Castelijns[3]; [1]VITO, Process Technology, Boeretang 200, Mol B-2400 Belgium; [2]Heraeus Electro-Nite Int. NV, R&D, Centrum Zuid 1105 B1, Houtalen B-3530 Belgium; [3]Hoogovens Aluminium NV, Quality Dept., A. Stocletlaan 87, Duffel B-2570 Belgium

Magnesium is an important alloy addition to aluminum for can stock and automotive components in particular. The successful development of magnesium sensors for aluminum alloys has made continuous real-time and in-line measurement of magnesium content in the cast possible. Plant evaluation indicated that the sensor possessed sufficient accuracy and stability for process monitoring and control. The working mechanism of the magnesium sensor is briefly described and the results of sensor applications in laboratory experiments as well as in plant trials are presented.

10:55 AM
MINIMIZING METAL LOSS AND ENERGY DURING MELTING AND HANDLING OF MOLTEN ALUMINIUM AND UTILIZATION OF VALUABLE DROSS TO SAVE A FORTUNE: *Wolfram S. Ruff*[1]; [1]Consultant, 21680 Stade, Hempbarge Germany

Millions of tons of Aluminium are produced every year and a certain percentage of this amount is lost by conversion to Alumina during process. To minimise metal loss and amount of dross, one has to take care about the mechanism of oxidation during every step of operation. Oxidation depends on five parameters which are affecting metal loss or vice versa savings, If optimised. From scrap or tapped metal, during charging, melting, alloying, metal treatment, skimming down to casting, all parameters are discussed and improvements are shown. But even with the very best technology there will be dross, which has to be cooled immediately after skimming and processed to get as much valuable metal back as possible. My Credo for total metal handling and secondary dross processing: Use the best available technology, with minimum metal loss, smallest amount of residues, most economic treatment of said residues and then YOU ARE ABLE TO SAVE A FORTUNE!

CAST SHOP TECHNOLOGY: Molten Metal Processing/Filtration

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

Thursday AM Room: 6D
March 4, 1999 Location: Convention Center

Session Chair: C. William McCormick, McCormick & Associates, Sydney, NSW 2075, Australia

8:30 AM

STAGED FILTRATION EVALUATION AT AN AIRCRAFT PLATE AND SHEET MANUFACTURER: *Michael M. Niedzinski*[1]; David D. Smith[1]; Leonard S. Aubrey[2]; [1]McCook Metals, Chief Metallurgist, 1st Ave. & 47th St., McCook, IL 60425 USA; [2]SELEE Corporation, 700 Shepherd St., Hendersonville, NC 29792 USA

SELEE Corporation has recently developed a staged filtration system, utilizing two standard ceramic foam filters (CFF) in series, which improves metal filtration capabilities beyond that of standard single element system, yet retains all the advantages of single element ceramic foam filtration system. Some of the advantages retained include operator acceptance and alloy flexibility. McCook Metals recently evaluated this system on 7XXX series alloys using both LiMCA II and LAIS techniques. Additionally an attempt was made to correlate the LiMCA II results with ultrasonic plate recovery. In addition LiMCA data was obtained on a standard single filter element system and a filter containing a coarse/fine duplex pore size structure. LiMCA results indicated that with the staged filtration system average N20 inclusion contents down to 100 particles per kilogram (0.1 k/kg) were obtainable. These excellent results were followed by other combinations including the duplex filter and then by a standard single ceramic foam filter element. Comments on potential future applications and benefits will be reviewed.

8:55 AM

EVALUATION OF THE EFFICIENCY OF FINE PORE CERAMIC FOAM FILTERS: *Neil J. Keegan*[1]; Wolfgang Schneider[2]; Hans-Peter Krug[2]; [1]Foseco International, Ltd., Aluminium, P.O. Box 5516, Tamworth, Staffs B78 3XQ UK; [2]VAW Aluminium AG, R&D, Georg-v. Boeselager-Strasse 25, Bonn D-53117 Germany

Melt treatment leading to inclusion removal is an important process step in the production of d.c. cast ingots so as to limit defects during the subsequent processing of these ingots into the finished product. A range of in-line treatment methods are available for their removal. Currently the most widely used method is by filtration of the liquid aluminium using a porous media, namely ceramic foam. With the objective of investigating the performance characteristics of a range of in-line filtration systems, in particular that of ceramic foam filters, a joint programme of work has been ongoing between Foseco and VAW - R&D since 1995. The latest phase of this programme has investigated the use of fine and ultrafine pore ceramic foam filters with cell sizes down to 650 microns. This extends the earlier ceramic foam filter trials by investigating CFF's with cell sizes which are closer to the mean pore diameters of the tube filters trialed earlier. Trials under carefully controlled plant conditions were undertaken on 50ppi, 65ppi and 80ppi porosity filters in addition to those of coarser 30ppi CFF's Their performance has been monitored using LiMCA II, PoDFA and LAIS. This paper presents a comparison of the relative performance of these filters and compares these results with those of other in-line filtration systems. It also considers both the physical characterisation of the CFF's and some of its important structural characteristics along with metallographic evaluation of the spent ceramic foam filters themselves.

9:20 AM

RECENT IMPROVEMENTS IN CERAMIC FOAM FILTER BOWL DESIGN BY COUPLED HEAT AND FLUID FLOW MODELLING: *Gerd Ulrich Gruen*[1]; Wolfgang Schneider[1]; Steven F. Ray[2]; Jan-Olaf Marthinusen[2]; [1]VAW Aluminium AG, Research & Development, Georg-von-Boeselager-Strasse 25, Bonn D-53117 Germany; [2]Foseco International, Ltd., P.O. Box 5516, Tamworth, Staffordshire B78 3XQ England

Increasing demands on metal quality and ongoing improvements in the application of ceramic foam filters have secured the use of this technique to treat molten aluminium. It now offers a simple, reliable and cost effective method to remove inclusions from liquid aluminium which is an important part of meeting the quality demands of specific product applications of DC cast aluminium ingots. The importance of metal cleanliness to a modern casthouse led Foseco and VAW to jointly examine the design of current filter bowls. The flow patterns in the bowls were optimised by coupled heat transfer and fluid flow modelling for the initial and stationary phase. The paper demonstrates the design principles used and some of the problems with the traditional bowl designs with particular regard to the flow field in the outlet area. A stepwise approach was used to improve the design. The heat and fluid flow modelling results at each step were calculated and the benefits are reviewed. Finally, the fluid flow in a standard bowl is compared to a fully optimised bowl.

9:45 AM

DEVELOPMENT OF A COMPACT DEEP BED FILTER FOR ALUMINIUM: *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

A new deep bed filter has been developed and tested in production runs at Elkem Aluminium ANS, Lista, Norway. The filter is cylindrical and the melt moves axially downwards. It thus has a more compact design and utilises the filter media more efficiently than conventional deep bed filters. Objectives were to investigate the mechanisms of inclusion removal and "ageing" of the deep bed filter. During operation, samples of the melt were taken before and after the filter. Samples were taken both at the start-up, in the middle and towards the end of the test period. The samples were studied metallographically using automatic image analysis. Size distributions of inclusions in and out of the filter were obtained and filtration efficiencies were determined. Removal may be explained in terms of an interception mechanism. When the efficiency dropped only 2% of the void volume was occupied by inclusions.

10:10 AM BREAK

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PRODUCTION SCALE EVALUATION OF NEW DESIGN CERAMIC FOAM FILTER: *Jennifer Black*[1]; Gary Parker[2]; Tabb Williams[2]; [1]Vesuvius Hi-Tech Ceramics, P.O. Box 788, Alfred, NY 14802 USA; [2]Reynolds Metals Company, 13203 N. Enon Church Rd., Chester, VA 23831 USA

The aluminum industry has widely accepted the standard flat surface ceramic foam filter for use in a wide range of product quality requirements. Over the past 20 years, there have been relatively few revolutionary improvements in the basic flat filter design. Vesuvius Hi-tech Ceramics has now developed and patented a high surface area, ceramic foam filter for use in molten aluminum. A cooperative program between Reynolds Metals Corporation R&D and Vesuvius Hi-Tech Ceramics was established to investigate the performance of the Vesuvius "New Wave" high surface area filter versus their commonly used, flat ceramic foam filter. The objective of the program was to compare the Vesuvius "New Wave" filter to their standard flat ceramic foam filter under actual production conditions. Production scale casting of DC 5182 ingot was conducted at the Reynolds Corporate R&D Casthouse. The flow rate per unit area of filter was made comparable to typical DC production facilities. The two filter types, standard and "New Wave", were evaluated as 40 ppi and 50 ppi filters producing four filter categories. A total of 12 casts were made to evaluate the four filter categories. Filtration efficiencies using LiMCAII results will be presented, along with LAIS results and operational differences for the flat and "New Wave" filters.

10:55 AM

MODELING OF GROWTH AND SEPARATION OF INCLUSIONS IN METALS REFINING SYSTEMS: *Nagy El-Kaddah*[1]; David Godard[2]; Pierre-Yves Menet[3]; [1]The University of Alabama, Dept. of Metall. Eng., P.O. Box 870202, Tuscaloosa, AL 35487 USA; [2]Pechiney CRV, PB 27, Voreppe 38340 France; [3]Pechiney Rhenalu, Z.I. Biesheim, B.P. 49, Neuf-Brisach F-68600 France

Theoretical models of inclusion removal in aluminum melt treatment systems need to account for effects of flow and turbulence on transport, coalescence and separation of dispersed phases. Traditional models based on discrete particle flow or continuum species transport ignore some of these effects. This paper presents a population balance model for describing the dynamic behavior and separation inclusion particles in a stirred molten metal. It is based on the solution of a modified Smoluchowski population balance equation that allows for convective transport of inclusions in the melt. The procedure for solving coupled fluid flow and particle balance equations is also described. The model was tested by comparing model predictions against measured rates of removal of 10 mm SiC and Al2O3 particles in levitated aluminum droplets. The excellent agreement obtained between measurements and predictions regarding both the evolution of particle size distributions and the rate of inclusion removal shows a full validation of the technique employed.

11:20 AM

THE MEASUREMENT OF CONTROLLED SIZE PARTICLES IN MOLTEN ALUMINUM USING THE LIMCA TECHNIQUE: *C. Dupuis*[1]; [1]Alcan International, Ltd., Arvida Research and Development Center, Jonquiere, Quebec Canada

The LiMCA technique allows the measurement of nonmetallic particles suspended in molten aluminium based upon the resistive pulse principle. The size of the particles measured is based on their volume and is reported as the diameter of the equivalent sphere. In order to verify the calibration of the LiMCA, metal samples containing controlled size particles were prepared and analyzed using LiMCA. This paper presents the results obtained with the LiMCA in comparison with the measurements done by other techniques. It will be shown that for regular shape inclusions, the size distribution obtained from the LiMCA is in good agreement with the one obtained by other characterization methods. The effect of sample preparation and particle shape will also be discussed.

FLUID-FLOW PHENOMENA IN METALS PROCESSING: Metals Refining and Aqueous Processing

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, Dept. of Proc. Metall. & 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

Thursday AM Room: 2
March 4, 1999 Location: Convention Center

Session Chairs: Knut Halvard Bech, SINTEF, Materials Technology, Trondheim, N-7034 Norway; Ramana Reddy, The University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487-0202 USA

8:30 AM

MAGNETIC FIELD CONTROL OF THE MELT EXTRACTION PROCESS: A. Cramer[1]; *Gunter Gerbeth*[1]; [1]Research Center Rossendorf, Inc., P.O. Box 510119, Dresden D-01314 Germany

The production of highly porous metallic substrates requires an effective and well-controlled production of metallic fibres with diameters in the range of 20 to 200 microns. The liquid melt extraction, where a quickly rotating wheel extracts the fibres directly out of the inductively heated melt, is a promising technology for that purpose. Up to now instabilities caused by the rotating wheel and the temperature gradients in the melt did not allow a reproducable production of controlled fibre diameters. The idea is to suppress these instabilities by suitable external magnetic fields. We present model experiments on the influence of steady magnetic fields on the melt surface instabilities, the meniscus at the boundry between wheel and melt, and the diameter distribution of the finally produced fibres. The model experiments are performed with an eutectic tin-lead alloy. Real-scale results for NiAl and other high-temperature melts will also be given. The magnetic field has a serious stabilizing influence on the process.

8:50 AM

FLOW INSTABILITIES IN VAR: *P. A. Davidson*[1]; [1]University of Cambridge, Eng. Dept., Trumpington St., Cambridge CB2 1PZ UK

We discuss a well-known but much misunderstood instability which occurs in Vacuum Arc Remelting. In the absence of buoyancy, the dominant force on the pool arises from the interaction of the current flow and its self magnetic field. The resulting flow is poloidal. However, it is well known that very small, stray magnetic fields can induce an intense swirling motion. This is usually attributed to the breakdown of a self-similar solution for the poloidal flow. We show that this is not the case, and that the true explanation lies in the action of the Ekman pumping.

9:10 AM

CONVECTIVE HEAT TRANSFER DURING ELECTRON BEAM EVAPORATION OF LIQUID METALS: *Christian Karcher*[1]; R. Schaller[1]; A. Thess[1]; [1]Dresden University of Technology, Institute for Aerospace Engineering, Center for Physical Fluid Mechanics, Dresden D-01062 Germany

Electron beam evaporation of liquid metals is an innovative technology increasingly used in industry to produce very thin coatings of high purity. In this process the surface of a metal ingot, placed in a water-cooled copper crucible, is heated by bombarding it with a high-energy electron beam gun. The material melts, forming a free surface and eventually starting to evaporize. The rising vapor cloud condensates as thin film on a moving substrate. The strong energy input at the free surface gives rise to vigorous thermocapillary and buoyancy-driven convective motion within the melt leading to unwelcome heat losses. The present paper aims to show how these losses can be reduced by optimizing the geometry of the cruicible and by using external magnetic fields. We present both experimental and numerical results.

9:30 AM

OXYGEN-ENRICHMENT OF SIDE-WELL ALUMINUM FURNACES: *Charles E. Baukal*[1]; [1]Air Products and Chemicals, Inc., Global Applications Development, 7201 Hamilton Blvd., Allentown, PA 18195 USA

Reverberatory furnaces play a significant role in the aluminum melting industry. Optimizing the heat and mass transfer in these furnaces reduces the capital, operating, and maintenance costs. The use of oxygen-enriched air/fuel firing or pure oxy/fuel firing can reduce the melting cycle, fuel consumption, and total emissions from the furnace. Control of the oxygen content at the aluminum surface is also important for reducing metal losses which means increased yields. Improper use of oxygen-enriched or pure oxygen firing technology in aluminum reverb furnaces as practiced in the past has resulted in furnace damage and metal loss from local overheating. Many questions remain to be answered about new oxy/fuel technologies. Aluminum producers need to be convinced of the benefits of new technology involving oxygen enhanced combustion. To answer many of the questions and to try to optimize the heat and mass transfer in aluminum reverb furnaces, a 3-D computer model was developed which couples the combustion space and the molten aluminum bath. Prior models considered only the combustion space. This new model solves the mass and the energy transfer equations in both media simultaneously. Turbulent flow in the combustion space interacts with viscous laminar flow in the melt. Conduction, convection and radiation heat transfer mechanisms are included in the model. Improved understanding of the furnace operation is based on temperature, velocity, and species concentration in the combustion space and the molten bath. Typical results are presented for an aluminum side-well furnace with air/, air-oxy/, and oxy/fuel combustion systems. It will be shown that oxygen-enriched combustion has a significant advantage over air/fuel systems. A properly designed oxy/fuel system can increase furnace production and fuel savings while minimizing pollution emissions. The newly-developed model is a powerful tool for process and geometry optimization in aluminum reverb furnaces.

9:50 AM

VISCOSITY MEASUREMENT AND MODELING OF BORATE AND BOROSILICATE MELTS: *Zhijing Zhang*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., A129 Bevill Bldg., 126 Seventh Ave., P.O. Box 35487-0202, Tuscaloosa, AL 35487-0202 USA

The viscosity of molten oxides is one of the important physical properties since viscosity has a decisive influence on fluid flow. The knowledge of the viscosity of molten oxides is essential for modeling and controlling of metallurgical process. In the present study, viscosities of Na2O-SiO2-B2O3 ternary melts were measured as a function of temperature and composition. Experiments were carried out using rotary viscometer and graphite components. Our experimental results were compared with those in literature. Based on the current experimental data, the previously developed viscosity model was modified. The characteristic features of viscosities of these melts have been discussed. The predicted viscosity results are in excellent agreement with the present experimental data in the temperature range with molten state samples.

10:10 AM BREAK

10:30 AM

MATHEMATICAL MODELING OF THE FLUID FLOW FIELD IN AN ELECTROLYSIS CELL: *Andreas Filzwieser*[1]; Klaus Hein[1]; Peter Paschen[1]; Herwig Grogger[2]; [1]University of Leoben, Dept. of Nonferrous Metall., Franz-Josef-Straße 18, Leoben A-8700 Austria; [2]AVL LIST GmbH, Hans-List-Platz 1, Graz, Styria A-8020 Austria

The fluid flow in a copper refining cell is calculated with a closer look to the mass transport phenomena in front of the electrode surface, using the CFD-software package FIRE®. The simulation is based on different density values in the boundary layer at the electrode surface given by a density/concentration correlation. The copper concentration - linked with current density by the Faraday law - is solved by an additional transport equation. The thickness of diffusion layer and hydrodynamic boundary layer is calculated. The numerical solution of the fluid flow field is compared with results of LDA-measurements, which were done in a special cell parallel to the numerical calculation. The mathematically modeled copper concentration is also compared with experimental measurements. Furthermore, the limit current density is calculated for various values of the electrolyte circulation.

10:50 AM

A STUDY OF FLUID FLOW IN PACHUCA TANKS: D. A. Salinas G.[1]; *A. H. Castillejos E.*[1]; [1]Centro de Investigacion y Estudios Avanzados del IPN, Unidad Saltillo, Apdo. Postal 663, Saltillo, Coahila 25000 Mexico

Pachuca tanks are commonly used in the mineral processing industry for leaching operations. The velocity distribution in the liquid phase, resulting from the injection of gas into the draft tube, is important to achieve a complete suspension of the mineral particles and has a decisive influence in the mass transfer processes occurring in this reactor. This study investigated the effect of different design and operating characteristics on the liquid velocity field. Experiments were carried out in a laboratory-scale Pachuca tank. The transparent model was filled with water, and air was injected through the bottom to establish a recirculatory flow pattern in the absence of mineral particles. Liquid velocities for the whole reactor were measured under several experimental conditions using particle image velocimetry (PIV). This technique allowed to obtain a very detailed picture of the liquid motion even in the draft tube, when bubbles were absent from the measuring field of view. The experiments have shown that the liquid velocities increase with increasing the draft tube diameter to tank diameter ratio, the distance from the conical bottom to the draft tube entrance, or the gas superficial velocity. On the other hand, an increase in the water level with respect to the draft tube outlet causes a decrease of the liquid velocities in the tank.

11:10 AM

VISUALIZATION AND QUANTIFICATION OF THE FLUID FLOW IN A COPPER ELECTROWINNING CELL: *Gerhard Hanko*[1]; Klaus Hein[1]; Andreas Filzwieser[1]; [1]University of Leoben, Dept. of Nonferrous Metall., Franz-Josef-Straße 18, Leoben, Styria A-8700 Austria

Optical studies of the hydrodynamic flow in a copper electrowinning cell have been compared with experimentally determined velocity profiles. The experimental velocity measurements have been carried out in a laboratory cell using a laser Doppler anemometer. The relation between velocity profiles and the height measured from the lower edge of the electrodes has been examined for a constant current density. Moreover, the velocity distribution has been measured for a varied electrode interspace and different current densities. Each velocity profile has been evaluated with regard to the three different causes of the fluid flow: natural convection, forced convection by the electrolyte circulation and forced convection by the electrochemically induced gas stirring. The utilised software has made a calculation of the void fraction possible. Therefore, a rough estimation of the mass transfer coefficient through the prevailing gas bubble induced convection can be given, assuming influence of relative bubble volumina.

11:30 AM

OPERATION VARIABLES DESIGN ON SPIRAL HC 1870 AND HG 8/7 TO OPTIMIZE THE SEPARATION OF SILICE FROM IRON ORE THROUGH A MATHEMATICAL MODEL: *Mokka N. Rao*[1];

Asdrubal Serrano[1]; [1]Universidad Nacional Experimental De Guayana, U.N.E.G. Aptdo Postal 302, Puerto Ordaz 8015-A Venezuela S.A.

A mathematical model is developed which describes the movement of the minerals present where the vertical and angular velocities are related with the different operative parameters involved in the process of separation of silice from iron ore, which permit maximize the capacity of the spirals to obtain major productivity. The process is realized in two steps, of the rough dressing and the other cleaning. Through the results obtained for chemical analysis of the final products of each spiral and the equation of movement of the particles, it is proved the applicability and efficiency of mathematical model to operating parameters of the spirals. Moreover, it is determined through the recuperation of the final products of quality in the use of spirals HC 1870 and HG-8/7.

FUNDAMENTALS OF LEAD AND ZINC EXTRACTION AND RECYCLING: Recovery of Lead and Zinc From By-Products

Sponsored by: Extraction & Processing Division, Lead, Zinc, and Tin Committee

Program Organizers: A. Morris, San Diego, CA 92128 USA; Markus Reuter, Delft University of Technology, Netherlands

Thursday AM Room: 7A
March 4, 1999 Location: Convention Center

Session Chairs: Arthur E. Morris, Thermart Software, San Diego, CA 92128-2720 USA; Markus A. Reuter, Technical University Delft, Dept. of Raw Mats. Proc., Delft 2628 RX The Netherlands

8:30 AM

THE REDUCTION OF ZINC OXIDE IN CALCIUM FERRITE SLAGS: *Hurman Rauf Eric*[1]; E. A. Van Dijk[1]; Markus A. Reuter[1]; [1]Delft University of Technology, Faculty of Applied Earth Sciences, 120 Mijnbouwstraat, Delft 2628 RX The Netherlands

Dusts from steelmaking processes contain substantial amounts of zinc. Such dusts are also a hazardous waste and dumping them becomes unacceptable from an environmental point of view. Therefore processes that can recover zinc and possibly other metals from steelmaking dusts are being investigated. The aim of this paper is a contribution to the more fundamental kinetic aspects of zinc reduction from silica-free calcium ferrite slags. The effects of alumina, lime and magnesium oxide contents of the slag, the CO/CO_2 ratio and the effect of initial Fe_2O_3CaO ratio were studied experimentally at 1400°C. The results indicated that the reduction of zinc from molten slag was possibly a first order reaction. As expected higher CO/CO_2 ratios increased both the extent and rate of reduction. It was also observed that a higher iron content of the slag improved the reduction of zinc from calcium ferrite slags. This fact seems to support the proposed mechanism that zinc is partly reduced by ferrous iron to zinc vapour and that ferric iron is reduced by carbon monoxide. The reduction rate of zinc oxide in these slags was decreased by increasing alumina content of the slag. Alumina decreased the calculated optical basicity of the slag showing the fact that reduction of zinc oxide was more favorable in more basic slags. Probably the formation of complex ions by alumina and zinc oxide such as $ZnAl_2O_3^{2+}$ lowers the activity of Zn^{2+} ions in the slag and hence renders them more difficult to reduce.

8:50 AM

FUMES, FOGS AND MISTS, PART 2: OPERATIONAL EXPERIENCE IN PYROMETALLURGICALLY RECOVERING ZINC AND LEAD FROM STEEL MILL DUSTS: *Larry M. Southwick*[1]; [1]L.M. Southwick & Associates, Suite 306, 992 Marion Ave., Cincinnati, OH 45229 USA

Baghouse dusts generated by integrated steel mills and electric arc furnace minimills contain economically interesting quantities of zinc and lead. A number of pyrometallurgical processes have been attempted in the past and new ones are being proposed to fume the heavy metals and recover them as liquid metals in a splash condenser. While splash condensers have long been demonstrated commercially in the zinc industry, their application to powdery, halide-containing steel mill dusts have uniformly met with difficulties. Part 1 of this paper reviewed theoretical and practical considerations in the design and application of splash condensers to this service. This paper, Part 2 of the series, will sumarize actual operation of two of these units and present a technical analysis of results and performance. The focus will be on condensation and collection efficiency of the units and an analysis of operating problems with the units. Of particular interest is recovery and recycling of heavy metals not collected i n the condenser, how that activity influenced earlier data analysis and the design of possible improvements, and an evaluation of those new designs in the light of these earlier operations. These specific results will also be related to the more general analysis presented in Part 1 of the study.

8:50 AM

RECYCLING OF GALVANIZED STEEL SCRAP USING CHLORINATION: *James K. S. Tee*[1]; Derek J. Fray[1]; [1]University of Cam bridge, Department of Materials Science and Metallurgy, Pembroke St., Cambridge, Cambridgeshire CB2 3QZ UK

The steel industry is facing a daunting task. It has become increasingly difficult to obtain clean scrap for the manufacture of steel. This problem has been accentuated by the use of galvanized steel. Present methods such as caustic leaching used for removal of zinc from steel scrap are not only tedious and costly but also unsuitable when zinc is present as an alloy. Based on a thermodynamics study, it is apparent that zinc chloride is more stable than zinc oxide while the reverse applies to the iron compounds. Described here, a novel separation route using air and chlorine mixtures has been proposed. Chlorine is both cheap and readily available; volatile chlorides are easy to separate, and better surface contact between gas and scrap can be achieved. This gives improved zinc removal over a shorter duration, with less preparation. Special attention, however, is required in handling chlorine due to its corrosive nature. The chlorination of galvanized steel in air and chlorine mixtures has been investigated in the laboratory by using thermogravimetry. Scanning electron microscopy, atomic absorption spectroscopy and x-ray diffraction were used to characterise the reaction products. The results demonstrated that it is feasible to separate zinc from steel at 800℃, using a ratio of 10: 1 with respect to air and chlorine. A separation of up to 97% zinc is achieved in 10 minutes. The chlorination of galvanized steel is based upon selective chlorination of zinc instead of iron. While zinc undergoes chlorination producing a volatile zinc chloride, the oxidation of iron provides a protective oxide layer of hematite that resists the attack of chlorine, and thus the carryover of iron as a chloride. This provides an attractive method for the steel related industries to recycle galvanized steel scrap.

9:10 AM

THERMAL TREATMENT OF JAROSITE: *I. Gaballah*[1]; A. Bonazebi[1]; N. Kanari[1]; [1]ENSG-LEM, Mineral Processing and Environmental Engineering, Rue du Doyen Marcel Roubault, BP 40, Vandoevre 54501 France

Jarosite contains up to 15% of heavy metals. The current waste disposal methods will not be allowed in the near future due to environmental regulations and pollution risks. For these reasons, thermal treatments of raw or decomposed jarosite, in controlled atmospheres, were investigated. The aim of these treatments is to decontaminate the jarosite and/or to recover the heavy metal compounds for recycling. According to physico-chemical characteristics of the samples, the thermal treatments under controlled atmospheres (N_2), (CO_2 + N_2) and (Cl_2 + air) lead to extraction extents that vary from 64 to 94%, from 89 to 98% and 95 to 99% respectively. Heavy metal compounds are separated by cooling the gaseous phase. The treatments' residue is almost free from heavy metal compounds. Between 700°C and 800°C, the chrlorination treatment was successful in eliminating more than 97% of heavy metal compounds contained in four different samples.

9:30 AM BREAK

9:50 AM

SCALE-UP OF BIOLOGICAL REACTORS FOR SULFATE REDUCTION AND SULFIDE OXIDATION: *C. J. N. Buisman*[1]; G. H. R. Janssen[1]; H. Dijkman[1]; S. H. J. Vellinga[2]; [1]PAQUES Bio Systems B.V., P.O. Box 52, Balk 8560 AB The Netherlands; [2]PAQUES B.V., P.O. Box 52, Balk 8560 AB The Netherlands

THIOPAQ technology developed and marketed by PAQUES Bio Systems of Balk, Netherlands, has been successfully used at commercial scale at the Budelco zinc refinery in the Netherlands for the treatment of contaminated groundwater since 1992. In essence, THIOPAQ technology consists of two biological process steps in series: sulfate reduction to hydrogen sulfide (anaerobic) and sulfide oxidation to elemental sulfur (aerobic). The biogenic sulfide produced can be employed for the chemical precipitation of metals in solution either inside the anaerobic reactor or in a separate vessel. Since the solubilities of most metal sulfides are much lower than of their respective hydroxides, considerably lower effluent metal concentrations can be achieved with THIOPAQ systems than in neutralization processes which immobilize metals predominantly by hydrolytic precipitation. In recent years, PAQUES has made significant advances in the design and operation of aerobic and anaerobic bioreactors for metal-sulfur systems. Moreover, the company has increased its technology portfolio to allow the development of more process oriented applications of THIOPAQ technology. A new PAQUES designed plant at Budelco treating both the zinc electrowinning bleed stream as well as the acid plant blowdown will be described in the present paper. Special emphasis is put on the scale-up of the gas lift loop bioreactors as selected for this Budelco project.

10:10 AM

REMOVAL OF LEAD FROM PROCESS SOLUTIONS: *M. K. Mohan*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Department of Metallurgical and Materials Engineering, P.O. Box 870202, A-129 Bevill Building, Tuscaloosa, AL 35487 USA

Processing of lead from secondary sources is of primary importance not only for the conservation of resources but also clean environment. In the present investigation, studies were conducted on the removal of Pb from process solutions using zeolites. Effects of pH, time and type of zeolite viz., chabazite, mordenite, erionite, and clinptilolite on the removal of Pb from solutions was investigated. Rate of sorption was found to depend on the pH of the solution and about 99pct. recovery was observed at pH 6. Optimal conditions were established for the maximum recovery of lead from process solutions. Possible sorption mechanisms were proposed. Based on the theoretical and experimental observations a conceptual flow sheet was proposed for the recovery of Pb from process solutions.

10:50 AM

RECOVERY OF HEAVY METALS FROM ACID MINE DRAINAGE WASTEWATER - AN INTEGRATED PROCESS: Paulo F.M.M. Correia[1]; Celina M.L. Santos[1]; M. Teresa A. Reis[1]; Jorge M.R. de Carvalho[1]; [1]Instituto Superior Tecnico; Department of Chemical Engineering, Av. Rovisco Pais - 1096 Lisboa Codex, Portugal

Acid drainage waste water from abandoned mines (namely the ones processing complex sulphides ores) have often high contents of heavy metals, such as iron, zinc, copper, cadmium, manganese, lead and nickel due to natural leaching of the mines exhausted ores. The leaching is increased by the atmospheric air oxidation of the sulphides to sulphates with consequent formation of sulphuric acid. If iron is present in the wastewater, Thiobacillus ferrooxidans bacteria help to catalyze the process. Further oxidation of the heavy metals present in the ores also helps to decrease wastewater pH and to increase mine waste water leaching power. These leachates end up contaminating ground waters and consequently rivers, lakes and wells, being a threat to the environment. Physical means of curtailing mine drainage formation and discharge, e.g. ground water diversion and mine sealing, have not met with unqualified success. Currently, treatment of acid mine drainage effluents to protect receiving streams appears to offer the best environmental protecting measure. Alkaline chemical neutralization followed by air oxidation has been the most popular method of mine water treatment. However considering the world wide continuous lacking of natural resources, new treatment processes that allow to concentrate selectively the metals present in the acid mine drainage wastewaters in order to allow their recovery are urgently needed. In this paper an integrated process involving cementation, leaching, emulsion liquid membranes, biosorption and electrolysis is proposed to treat the acid mine drainage wastewater of Algares mine (Aljustrel region, Portugal). This effluent has the following average composition: iron (891 ppm), zinc (500 ppm), manganese (98 ppm), copper (49 ppm), cadmium (1.25 ppm) and lead (1.11 ppm). Each of these heavy metals content is above the maximum permitted by the Portuguese environmental law. Through the application of the proposed integrated process a selective recovery of zinc, lead, copper and manganese with enough purity to allow their commercialization can be achieved. From the process an aqueous stream with low heavy metal content enough to be discharged in the environment and a sludge containing iron hydroxide and manganese result. This sludge can be disposed in a landfill according to the Portuguese environmental law.

INTERNATIONAL SYMPOSIUM ON GAMMA TITANIUM ALUMINIDES: TiAl Alloys: Creep

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. 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

Thursday AM — Room: 8
March 4, 1999 — Location: Convention Center

Session Chairs: Michael J. Mills, The Ohio State University, Columbus, OH 43210 USA; Birgir Karlsson, Chalmers University, Dept. of Eng. Metals, Goteberg SE-412 97 Sweden

8:30 AM INVITED PAPER

MICROMECHANISMS OF CREEP OF GAMMA-BASE TITANIUM ALUMINDES: *Fritz Appel*[1]; Michael Oehring[1]; P. J. Ennis[2]; [1]GKSS Research Center, Materials Research, Max Planck St., Geesthacht D-21502 Germany; [2]Forschungszentrum Juelich, IWE, Institut fuer Werkstofforsch, Juelich D-52425 Germany

Creep strength and rupture life are critical issues regarding high temperature applications of gamma base titanium aluminides. The mechanisms controlling these properties are not yet fully understood, partly due to the wide variety and complexity of microstructures. In addition, the long-term creep behaviour is not well characterized at modest stresses and temperatures even though these conditions are close to the intended design requirements. Therefore, long-term creep tests were performed on different two-phase TiAl alloys. Deformation processes were investigated by TEM on specimens crept at T = 700ϒ and sigma = 80 - 140 MPa for 6.000 - 10.000 hours. Accordingly the creep strength seems to be limited by significant structural changes of the lamellar constituents due to the emission of dislocations and the propagation of structural ledges. Potential metallurgical techniques to improve the creep strength will be discussed.

9:00 AM

CREEP MECHANISMS IN A NEAR-GAMMA TiAl-ALLOY WITH DUPLEX MICROSTRUCTURE : *Birgit Skrotzki*[1]; T. Rudolf[1]; G. Eggeler[1]; [1]Ruhr-University Bochum, Dept of Mech. Eng., Institute for Materials, Bochum 44780 Germany

Intermetallic near-g TiAl-alloys are candidate materials for high temperature applications due to their attractive properties (i.e. low density, high strength, good oxidation resistance). Single-phase g-alloys and fully lamellar alloys have been intensively studied in the last 10 years. Duplex alloys have received less attention although they show a

good compromise of properties such as strength, ductility, fracture toughness and creep resistance. The present work studies the creep behavior of a TiAl-alloy with duplex microstructure. In addition to the measurement and evaluation of creep data for component design, special emphasis was given to the microstructural evolution during creep of a near-g TiAl-alloy with duplex microstructure. The following basic processes have been identified as key elements of the overall creep deformation mechanism: (i) ordinary dislocation plasticity, (ii) twinning and (iii) dynamic recrystallization. These processes are coupled and jointly control the creep behavior of the material.

9:20 AM

CREEP DEFORMATION OF TiAl-Si ALLOYS WITH ALIGNED LAMELLAR MICROSTRUCTURES: *David Ray Johnson*[1]; Yosihiro Masuda[1]; Takamitsu Yamanaka[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 materials from a model system where the orientation and lamellar spacing were both controlled. Ingots of Ti-43Al-3Si with an aligned gamma/alpha2 lamellar microstructure were grown from an appropriately oriented seed by the floating zone technique. The as-processed microstructure consisted of eutectic silicide particles embedded in a PST matrix. Post processing heat treatments were then used to produce materials with either a fine or coarse lamellar spacing. Tensile creep tests were conducted at 1023 K (180 to 240 MPa) and 1073 K (120 MPa) on the as-processed and heat treated materials. The results clearly indicate the beneficial effect of decreasing the lamellar spacing as an order of magnitude difference in the secondary creep rate was found between the two microstructures. In addition to the lamellar spacing, the results are also discussed in terms of the microstructural stability of the lamellar microstructure.

9:40 AM

CREEP BEHAVIOUR OF A FULLY LAMELLAR Ti-47Al-2Nb-2Cr ALLOY: *Marc Thomas*[1]; [1]ONERA, Dept. Materiaux Metalliques et Procedes, BP 72, Chatillon, Cedex 92322 France

The creep behaviour of a fully lamellar Ti-47Al-2Nb-2Cr alloy has been studied over the temperature range from 650ϒC to 900ϒC and with the initial stress range between 69 and 400 MPa. Moderate variations in the activation energy Q and in the stress exponent n have been observed as a function of the creep conditions. The initial ingot which was supplied by the Austrian company Böhler was found to exhibit a highly textured structure. Our aim was then to assess the effect of texture by using typically five different orientations of the lamellar grains with respect to the deformation axis. Depending on this orientation, different plastic modes have been observed by means of tensile and compressive tests. Creep properties are therefore very sensitive to the orientation of the columnar grains and of the lamellae. Attempts to correlate the deformation anisotropy either to the orientation of the columnar grains or to the orientation of the lamellae are presented.

10:00 AM

NEUTRON DIFFRACTION MEASUREMENTS OF INTERNAL STRAINS IN TiAl-BASED ALLOYS: *Bimal Kad*[1]; Hahn Choo[2]; Mark Bourke[2]; [1]University of California-San Diego, AMES-0085, UCSD, La Jolla, CA 92093-0085 USA; [2]LANSCE, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Recent advances in computational methodologies can now predict the microstructure sensitive development of intrinsic strains and stresses inside thick anisotropic Ti-Al based composites [1]. However, such predictions are largely uncorroborated because of the lack of experimental means to probe at sub surface levels (i.e., deeper than 100μm). Towards this end, neutron sources present the unique capability of probing deep within dense structural materials and composites, to provide a reliable calibrating measure for the numerical predictions. Currently, a combination of experimental -theoretical -computational tools is being employed for directly measuring and interpreting the development of internal stresses in two-phase TiAl+Ti3Al low symmetry composite materials. The low symmetry results in severe internal constraints and stresses which affect both constitutive response and material toughness (i.e. ductility and fracture resistance). We will present our initial results on the evolution of internal stresses during i) thermo-mechanical processing induced phase transformations and ii) deformation and subsequent in-service loading in two-phase TiAl+Ti3Al alloys. [1] B.K. Kad, M. Dao and R.J. Asaro, (1995) Philos Mag, 71, p.567-604.

10:20 AM INVITED PAPER

IMPORTANCE OF MICROSTRUCTURAL STABILITY TO CREEP STRENGTH OF FULLY LAMELLAR TiAl ALLOYS: Kohei Mizoguchi[1]; Ryuichi Yamamoto[1]; Gerhard Wegmann[1]; *Kouichi Maruyama*[1]; [1]Tohoku University, Dept. of Mats. Sci., Aoba-yama 02, Sendai 980-8579 Japan

In principle, refinement of lamellar spacing should improve creep strength of fully lamellar TiAl based alloys. However, fine lamellar materials do not always give high creep strength, probably due to instability of fine lamellar structure during creep. High temperature creep of fully lamellar Ti-42mol%Al alloys with 0.1 and 1.5 micron meter in lamellar spacing was studied to examine effects of microstructural instability on creep strength. Microstructural degradation was more significant in the fine lamellar material at low stresses. Some specimens with the fine lamellar spacing were annealed at high temperatures to stabilize their lamellar structure. This heat treatment suppressed the microstructural degradation during creep, and improved creep strength of the fine lamellar material at low stresses. On the basis of these findings, it will be proposed that stable lamellar structure as well as fine lamellar spacing is highly important to improve long-term creep strength of fully lamellar TiAl alloys at low stresses.

10:50 AM

ON THE ROLE OF INTERFACE STRENGTHENING IN LAMELLAR MICROSTRUCTURES AT CREEP TEMPERATURES IN Ti-(47-48)Al BASED ALLOYS.: *Gopal Babu Viswanathan*[1]; Perena I. Gouma[1]; Subramanian Karthikeyan[1]; *Young-Won Kim*[2]; *Michael J Mills*[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]UES, Materials & Processes Division, 4401 Daton-Xenia Rd., Dayton, OH 45432 USA

Lamellar microstructures have traditionally shown higher creep resistance in g-TiAl based alloys. The strengthening in these microstructures comes from the presence of numerous g/g and g/a2 interfaces providing resistance to slip both in soft mode and hard mode type deformation. In this study, the presence of such interfaces in large numbers or in other words the decrease in inter-lamellar spacing has been observed to moderately decrease both the primary and secondary the creep rates in the temperature range 700-815ϒC, in polycrystalline binary Ti-48Al alloy studied. On the other hand, the creep rates are further lowered in Ti-47Al alloy containing C and Si. The strengthening here comes from the carbide and silicide precipitates at lamellar interfaces. Such lamellar interfaces have also been investigated in prior-to and post creep deformed samples. Defect analysis through weak beam dark field imaging was carried out to elucidate the dislocation mechanisms at the lamellar interfaces. Observations indicate that both 1/2[110] unit and <011] and [112] superdislocations are active, that the deformation is highly anisotropic and that the precipitates at these lamellar interfaces obstruct slip both in soft mode and hard mode orientations. A relative high density of mobile dislocations at the lamellar interfaces seem to suggest that the creep strain is controlled by the soft mode type deformation. Consequently, the presence of these precipitates play a major role in obstructing such interfacial slip by providing Orowan type strengthening at lower temperatures and/or providing climb barrier resistance at higher temperatures, similar to that of dispersion strengthened materials. However, these advantages of interface strengthening could be compromised by virtue of the instability of these interfaces caused by the dissolution of a2 during creep that has been seen to occur in this study. The posterior changes in the morphology of the interfaces and associated deformation structures have been addressed.

11:10 AM

THE EFFECTS OF MINOR ALLOYING ADDITIONS AND MICROSTRUCTURE ON THE CREEP BEHAVIOR OF Ti-47Al: *Margaret Keller*[1]; D. Eylon[1]; [1]University of Dayton, 300 College Park, Dayton, OH 45469 USA

Microstructure and certain minor alloying additions have been found to significantly influence the creep behavior of TiAl alloys. However their effects have not been isolated. This paper presents the results of a

systematic study investigating the effect of lamellar lath spacings and minor alloying additions of Si and C on the creep behavior of Ti-47Al. For each composition heat treatments were developed to produce two fully lamellar alloys with two different lamellar spacings. The effects of alloying additions and lamellar lath spacing on primary and secondary creep were measured at 760ϒC and 207MPa. Preliminary results indicate that additions of carbon as well as a refinement in lamellar spacing have beneficial effects on the creep behavior of this alloy.

11:30 AM

EFFECT OF FULLY LAMELLAR MORPHOLOGY ON THE CREEP OF A NEAR GAMMA TiAl INTERMETALLIC: Xiaomei Du[1]; *Jonathan Beddoes*[2]; Linruo Zhao[1]; [1]National Research Council of Canada, Structures, Materials & Propulsion Lab., Institute For Aerospace Research, Montreal Rd., M-13, Ottawa, Ontario K1A 0R6 Canada; [2]Carleton University, Dept. of Mech. & Aero. Eng., 1125 Colonel By Drive, Ottawa, Ontario K1S 5B6 Canada

The creep of Ti-48%Al with a fully lamellar microstructure is presented as a function of lamellar interface spacing and grain boundary morphology. The fully lamellar micro-structure is varied by altering the cooling from heat treatment in the a phase field, which included: furnace cooling, air cooling and an innovative two step cooling process consisting of furnace cooling followed by air cooling or water quenching. The lamellar interface spacing and extent of interlocked lamellae at grain boundaries increase with decreasing cooling rate. However, the two step process provides improved control of these microstructural features. Creep at 760ϒC/240 MPa indicates that the minimum creep strain rate decreases with decreasing lamellar spacing. Interlocked grain boundaries increase tertiary creep. The improved microstructural control available via the two step process allows the creep properties to be varied in terms of minimum creep strain rate and tertiary creep strain, providing a unique combination of creep properties.

11:50 AM

MICROSAMPLE TENSILE CREEP TESTING OF FULLY-LAMELLAR TiAl ALLOYS: *Carl J. Boehlert*[1]; *Mark Zupan*[1]; Dennis M Dimiduk[2]; Kevin J. Hemker[1]; [1]The Johns Hopkins University, Mech. Eng., 3400 North Charles St., Rm 122 Latrobe Hall, Baltimore, MD 21218 USA; [2]Materials and Manufacturing Directorate, Metals and Ceramics, Air Force Research Laboratory, Wright-Patterson AFB, OH 45433-7817 USA

Microsample Testing of single grains cut from polycrystalline metals has proven to be a valuable technique for understanding orientation effects and deformation behavior of titanium-aluminide alloys. An elevated-temperature microsample testing apparatus has recently been developed to better understand the creep performance of such alloys at targeted service temperatures. The present study focuses on the primary creep behavior of several TiAl alloys ranging in Al composition from 45-51(at.%). These alloys were thermomechanically processed and heat treated to obtain very large fully-lamellar grains containing different thicknesses of the alpha-2 and gamma phases. The objective of this work is to eliminate the effect of grain boundaries and characterize the primary creep deformation as a function of lamellar volume fraction, thickness, and orientation. The shapes of the primary transient will be modeled and, where possible, compared with microstructural observations of the underlying deformation mechanisms.

12:10 PM

MICRO- AND MACRO-MODELLING OF THE CREEP BEHAVIOUR OF TiAl ALLOYS: *Philippe Bastid*[1]; *Leo Christodoulou*[1]; *Ian Perrin*[2]; [1]Imperial College of Science, Technology and Medicine, Dept. of Mats., Prince Consort Rd., London SW7 2BP UK; [2]European Gas Turbine, Mechanical Engineering Centre, Whetstone, Leicestershire LE8 6LH UK

Creep tests and microstructural investigations carried out on TiAl intermetallic alloys at 750ϒC (Ti-48Al-2Nb-2Mn and XD Ti-47Al-2Nb-2Mn-0.8%TiB2) have shown different types of fracture behaviour depending on the stress applied (trans- and inter-lamellar at high stress and much finer "granular" fracture at low stress). The observed fracture morphologies have been related to the microstructural transformation occurring during creep, which involve local recrystallisation and void formation. This behaviour has been modelled using FEM analysis at both the micro- and macro-scales. Modelling at the micro-scale yields qualitative information concerning the strains and stresses appearing within a "basic representative cell" of the microstructure for given global stress and strain. The local strains predicted are sufficient to induce recrystallization as observed in the test specimens. The macro-creep behaviour of the alloys has been modelled using a modification of the Cumulative Damage Model of Dyson and McLean, that allows the description of the 3 stages of creep. Our model includes a recrystallisation term that allows the description of the change in the microstructure and its concomitant effect on creep behaviour. It is thus possible to predict lifetime for a wide range of stresses with good reliability.

LONG TERM STABILITY OF HIGH TEMPERATURE MATERIALS: Stability of Refractory Metals, Titanium and Stainless Steels

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

Program Organizers: Gerhard E. Fuchs, Lockheed Martin Corporation, Schenectady, NY 12301-1072 USA; Kathryn A. Dannemann, Southwest Research Institute, San Antonio, TX 7828-0510 USA; Todd C. Deragon, Special Metals Corporation, New Hartford, NY 13413-5392 USA

Thursday AM — Room: 9
March 4, 1999 — Location: Convention Center

Session Chair: Todd C. Deragon, Special Metals Corporation, New Hartford, NY 13413-5392 USA

8:30 AM INVITED PAPER

ELEVATED TEMPERATURE STABILITY OF THE REFRACTORY METALS: *R. W. Buckman*[1]; [1]Refractory Metals Technology, P.O. Box 10055, Pittsburgh, PA 15236-0415 USA

The refractory metals (Nb, Ta, Mo and W) and alloys were investigated extensively for space nuclear power systems applications which required exposures to elevated temperature, liquid alkali metals and vacuum for times up to 10 years. Substantial investigations were conducted which studied the effect of these test environments, for times up to 26,000 hours and temperatures up to 1700°C, on the mechanical behavior of these materials. This paper will discuss the results of these investigations and present examples of the changes in microstructure and mechanical behavior resulting from the test exposures.

9:00 AM

EFFECTS OF OXYGEN PRESSURE ON THE OXIDATION BEHAVIOR OF VANADIUM-CHROMIUM-TITANIUM ALLOYS AT 700°C: *M. Uz*[1]; K. Natesan[2]; C. Fullenwider[1]; [1]Lafayette College, Chem. Engr. Dept., 341 AHE, Easton, PA 18042 USA; [2]Argonne National Laboratory, Energy Technology Division, Argonne, IL 60439 USA

We estimated the effects of oxygen pressure and temperature on the oxidation kinetics and microstructure of V and V-base alloys containing (in wt.%) 4 Cr-4 Ti, 5 Cr-5 Ti, 10 Cr, 10 Cr-5 Ti, and 15 Cr-5 Ti. Samples were made from 1-mm-thick sheet stock of each alloy and were annealed for 1 h at 1050ϒC before any further oxidation. Experiments were performed in environments containing 760, 160, 0.1, 5 X 10^{-4} and 5 X 10^{-6} torr oxygen at three or more temperatures ranging from about 350 to 700ϒC. Oxidation behavior of each alloy was modeled by using the data of weight change with time. Microstructural features, including grain size and scale morphology and thickness, were determined from metallographic examination of the cross-sectional area of each sample across its thickness. Microhardness profiles across the sample thickness were used to determine the depth of oxygen penetration, and to estimate the parameters of diffusion of oxygen. These results will be presented, and the effects of oxygen pressure on the oxidation kinetics

and microstructure of V and V-Cr-Ti alloys will be compared. Work supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

9:20 AM

EFFECT OF LONG TERM AGING ON THE MICROSTRUCTURAL STABILITY AND MECHANICAL PROPERTIES OF Ti-6Al-2Sn-2Zr-2Mo-2Cr: *Xiao-Dong Zhang*[1]; William Baeslack[1]; Dan Evans[2]; Hamish Fraser[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Wright Patterson AFB, Materials Directorate, WL/MLLM, Dayton, OH 45433 USA

The microstructural development of Ti-6Al-2Mo-2Cr-2Sn-2Zr (Ti-6-22-22) alloys after long term aging heat treatment has been studied by scanning electron microscopy (SEM), conventional transmission electron microscopy (CTEM) and high resolution electron microscopy (HREM). It is found that secondary intermetallic precipitates, namely alpha 2 and silicites, are formed depending on the specific aging temperatures. Precipitation of these intermetallic compounds has strong effect on the fracture toughness and tensile properties of the alloys. Results on the macro and micro deformation modes associated with crack initiation and dislocation characteristics will be presented. The importance of these observations will be discussed in view of the fundamental understanding and further control of the structural stability of these type of a-b Ti alloys.

9:40 AM

PHASE STABILITY OF LAVES INTERMETALLICS IN STAINLESS STEEL-ZIRCONIUM ALLOYS: *Daniel P. Abraham*[1]; James W. Richardson[2]; [1]Argonne National Laboratory, Chemical Technology Division, Bldg. 205, Room A167, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave., Argonne, IL 60439 USA

Laves intermetallics in the stainless steel - 15 wt% zirconium (SS-15Zr) alloy are metastable and transform to the Zr6(Fe,Cr,Ni)23 intermetallic during high-temperature annealing. The growth of the Zr6(Fe,Cr,Ni)23 intermetallic has been studied by in-situ high-temperature neutron diffraction. The as-cast SS-15Zr alloy contains the stainless steel phases, ferrite and austenite, the Laves intermetallics C36 and C15 and small amounts of Zr6(Fe,Cr,Ni)23. Neutron diffraction patterns as a function of time have been obtained on SS-15Zr alloys that were held at various elevated temperatures. Phase transformation was very slow for temperatures < 1150ϒC. Alloys held at temperatures between 1200-1250ϒC showed diffraction peaks corresponding to ferrite, C15 and Zr6(Fe,Cr,Ni)23 after extended annealing times. We will discuss the results of results of neutron diffraction analysis and propose a mechanism for Zr6(Fe,Cr,Ni)23 formation.

10:00 AM

LONG TERM CREEP RUPTURE PROPERTIES AND PRECIPITATION IN TYPE 316 STAINLESS STEELS: *Takanori Nakazawa*[1]; Yasuo Otoguro[1]; Hidetaka Kimura[2]; Hitoshi Kaguchi[3]; Manfred Schirra[4]; [1]Gunma University, Mech. Syst. Eng., 1-5-1 Tenjin-cho, Kiryu, Gunma 376-8515 Japan; [2]Nippon Steel Corporation, Steel Research Labs., 20-1 Shintomi, Futtsu, Chiba 293-8511 Japan; [3]Mitsubishi Heavy Industries, Ltd., Nuclear Plant Engineering, 1-1 Wadasaki-cho, Hyogo-ku, Kobe, Hyogo 652-8585 Japan; [4]Forschungszentrum Karlsruhe, Institute fur Materialforschung 1, Postfach 3640, Karlsruhe D76021 Germany

The knowledge about the relationship between creep rupture properties and microstructure is required to estimate the long term creep behavior. The microstructure of Type 316 steels with different carbon and nitrogen contents tested in creep rupture at 550ϒC and 600ϒC up to 40000h has been examined with electron microscope. All steels had the same rupture strength at short rupture time. Low carbon medium nitrogen steel(LCMN) showed the highest strength at longer time and higher rupture ductility than the other two steels. In LCMN fine Laves phases precipitated on the grain boundaries(GB), while small amount of carbides and G phases precipitated on GB. Although medium carbon medium nitrogen steel(MCMN) showed the similar precipitation behavior on GB, the amount of carbides was a little larger than LCMN. Carbides and Laves also precipitated in the matrix of MCMN. In high carbon low nitrogen steel many carbides precipitated on GB and in the matrix whereas Laves appeared after longer period. The present results show that the creep rupture properties strongly depend on the precipitation behavior.

10:20 AM BREAK

10:30 AM

ELEVATED TEMPERATURE DIMENSIONAL STABILITY OF THIN GAUGE Fe-Cr-Al-RE (RARE EARTH) ALLOY FOR CATALYTIC CONVERTER SUBSTRATE APPLICATIONS: *Steve Chang*[1]; Bijendra Jha[1]; [1]Texas Instruments, Inc., Materials and Control, 34 Forest St., MS 4-14, Attleboro, MA 02703 USA

Honeycomb structures consisting of corrugated and flat ferritic Fe-20Cr-5Al-RE alloy foils have seen increased application as the substrate for stationary and automotive catalytic converters. The oxidation resistance of Fe-20Cr alloy is improved by the addition of Al which forms alumina scale to protect alloy substrate during elevated temperature exposure. The addition of rare earth elements, such as La and Ce, further improves cyclic oxidation resistance and enable the catalytic converter to satisfy the regulatory requirement for emission control. The major failure mode of honeycomb substrate during prolonged exposure at elevated temperature is the rupture of foil due to stretching. The necessary stress state in the honeycomb structure for stretching to occur is generally attributed to the dimensional change of alloy foils. This foil dimension has to remain constant in order for honeycomb structure to sustain long term, elevated temperature service. The elevated temperature dimensional change of Fe-20Cr-5Al-RE alloy foil was quantified by testing sub-size honeycomb samples. The effects of alloy chemistry on the oxidation rate and dimension change of Fe-20Cr-5Al-RE alloy foil are correlated and will be presented. A foil life prediction will be proposed and validated by testing foil with varying chemistry and gauges.

10:50 AM

ROLE OF REACTIVE ELEMENT ON LONG-TERM STABILITY OF ALUMINA SCALE FORMED ON Fe3Al DURING HIGH TEMPERATURE OXIDATION: *I. Kim*[1]; W. D. Cho[1]; [1]University of Utah, Dept. of Metall. Eng., Browning Bldg., Salt Lake City, UT 84112-1183 USA

A study on oxidation of iron aluminides was performed to investigate the effect of reactive element (yttrium) in terms of oxidation rate and oxidation adhesion at the temperature range of 800°-1100°C. At lower temperatures (<1000°C), oxidation rates of alloys, Fe_3Al and Fe_3Al-Y, was nearly same and alumina scale formed on the alloys displayed good adhesion. However, yttrium-added Fe_3Al alloy showed lower oxidation rate and much more improved oxide adhesion at higher temperatures. Microstructural examination of the oxidized Fe_3Al-Y alloy revealed the following: (1) formation of pegs at alumina scale/substrate interface, (2) formation of $Y_3Al_5O_{12}$ phase at the surface of the alumina scale, and (3) growth of alumina scale with preferred orientation. Adhesion improvement is believed to be due to the formation of pegs which increase the stability of the alumina scale. It was found that pegs were formed at grain boundary of the substrate near the interface because of the predominant diffusion of oxygen via grain boundary of lattice of alumina scale.

11:10 AM

REACTIONS BETWEEN THE COMPONENTS OF SOLID OXIDE FUEL CELLS AT 800-1000ϒC IN AIR: *Peter Majewski*[1]; Fritz Aldinger[1]; [1]Max-Planck-Institut für Metallforschung, PML, Heisenbergstr. 5, Stuttgart, 70569 Germany

Reactions between the different components of solid oxide fuel cells (electrolyte: ZrO2 or LaGaO3; cathode: LaMnO3; interconnector: LaCrO3) have been studied in detail for temperatures that are relevant for the application of the cells. A reaction between Y stabilized ZrO2 and Sr doped LaMnO3 has not been observed. However, diffusion of Mn into ZrO2 is significant. Sr doped LaMnO3 reacts with Sr as well as Mg doped LaGaO3 forming various products. Even a reaction between Sr doped LaMnO3 and Ca doped LaCrO3 has been observed for 1000ϒC in air. In addition, a decomposition of single phase Sr doped LaMnO3 into SrMnO3 and Sr poor LaMnO3 occurs at temperatures below about 1300ϒC in air.

11:30 AM

ISOTHERMAL OXIDATION BEHAVIOR OF TI-49AL ALLOY: *Xuerong Wen*[1]; *Ramana G. Reddy*[1]; [1]University of Alabama, Dept. of Metall. and Mats. Eng., Tuscaloosa, AL 35487 USA

The isothermal oxidation behavior of binary Ti-49Al intermetallic was investigated in pure oxygen over the temperatures of 750ϒC to 1000ϒC. The experiments have been carried out using TGA set up. The oxidation products were analyzed using X-ray diffraction, SEM and EDS. Parabolic rate constants were calculated. An effective activation energy of 404 kJ/mol was deduced. The oxidation products formed were identified as TiO2(rutile) and "-Al2O3 (alumina) at all experimental temperatures. The oxidation scale was more adherent compared to that of Ti-32Al. It was also observed that the layer structure was formed at 1000ϒC due to more rapid outward diffusion rate of Ti than that of Al. A physical model for layer structure formation was developed.

MATERIALS PROCESSING FUNDAMENTALS: Nonferrous

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

Program Organizers: W.D. Cho, University of Utah, Dept. of Metall. Eng., Salt Lake City, UT 84112 USA; Huimin Liu, UES, Inc., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Lab, P.O. Box 2008 Bldg. 4508, Oak Ridge, TN 37831-6083 USA

Thursday AM — Room: 5A
March 4, 1999 — Location: Convention Center

Session Chairs: Derek John Fray, University of Cambridge, Dept. of Mats. Sci. and Metall., Cambridge, Cambs CB23QZ England

8:30 AM

MECHANISM OF REFRACTORY WEAR BY CALCIUM FERRITE SLAG: *Hideya Sato*[1]; Fumito Tanaka[1]; Susumu Okabe[2]; [1]Mitsubishi Materials Corporation, Central Research Institute, 1-297 Kitabukuro-cho, Omiya, Saitama 330-8508 Japan; [2]Mitsubishi Materials Corporation, Naoshima Smelter and Refinery, 4049-1 Naoshima-cho, Kagawa-gun, Kagawa 761-3100 Japan

Refractory wear caused by melts is one of the major determining factors for the interval of shut-down repair of the metallurgical furnaces in general. Calcium ferrite slag, employed by Mitsubishi Continuous Copper Converter, shows high fluidity and corrosive characteristics against most of the refractories. In order to improve the corrosion resistance of the refractories, the mechanism of MgO-based refractory wear by the calcium ferrite slag was intensively studied. The dissolution behavior of the refractory into the molten slag and the penetration of the molten slag into the refractory were observed by crucible tests and newly developed oscillating furnace tests. It was found that the wear of MgO-based refractories by the calcium ferrite slag is mainly dominated by the internal structural failure caused by the penetration of CaO-Cu2O rich slag which is formed as a result of the preferential absorption of FeOx from original slag by the spinel of the refractory.

8:55 AM

A DYNAMIC ELECTROCHEMICAL TECHNIQUE FOR THE MEASUREMENT OF TRACE AMOUNTS OF ARSENIC AND ANTIMONY IN MOLTEN ZINC: *Derek J. Fray*[1]; Terry E. Warner[1]; [1]University of Cambridge, Materials Science and Metallurgy, Pembroke St., Cambridge, Cambs CB2 3QZ England

In order to detect arsenic and antimony in zinc various electrochemical experiments were performed involving the electrochemical introduction of sodium atoms into molten zinc using a solid electrolyte. Under high rates of the formation of the intermetallic phase NaZn13 was observed. Under more moderate injection rates employing a symmetric "zinc/zinc cell" configuration, a qualitative relationship between the current generated during cyclic voltammetry and the arsenic and antimony concentrations in molten zinc was observed.

9:20 AM

DYNAMIC PROCESS SIMULATION OF CONTINUOUS COPPER SMELTING FURNACE: *Osamu Inoue*[1]; Susumu Okabe[2]; [1]Mitsubishi Materials Corporation, Central Research Institute, 1-297 Kitabukuro-cho, Omiya, Saitama 330-8508 Japan; [2]Mitsubishi Materials Corporation, Naoshima Smelter and Refinery, 4049-1 Naoshima-cho, Kagawa-gun, Kagawa 761-3100 Japan

In Mitsubishi Continuous Copper Smelting and Converting Process, smelting, slag-matte separation and matte converting are continuously carried out in series in three furnaces connected by launders. And there influence of the change in operating condition in upper stream, such as variation of feeds in the smelting furnace, propagates to down stream with some delay. In order to achieve stable operation, process control algorithm based on the dynamic characteristics of each furnace would be very effective. In this study, the dynamic process simulation model was developed for the smelting furnace. The model consists of two mathematical modules, i.e. the thermodynamic module and the mixing module. The calculation results using operating data of charged materials, i.e. concentrates, fluxes, coal, oxygen, air, etc., obtained from Naoshima Smelter showed good agreement with the actually observed change in temperature, composition and flow rate of matte and slag at the outlet of the furnace. In addition, operation parameters such as reaction efficiency of oxygen and coal, dead volume of melts were estimated.

9:45 AM

THERMAL DECOMPOSITION OF ENARGITE: *R. Padilla*[1]; Y. Fan[1]; I. Wilkomirsky[1]; [1]University of Concepcion, Department of Metallurgical Engineering, Edmundo Larenas, Concepcion 270 Chile

In most of the Chilean copper and gold concentrates, arsenic is present mainly as enargite, Cu_3AsS_4 which at the conventional roasting or smelting temperature decomposes liberating arsenic compounds. The objective of this investigation was to study the decomposition reaction of energite in an inert atmosphere in the range of 530ϒC-750ϒC. The effect of the particle size on the decomposition reaction was also investigated. The results obtained indicated that the decomposition starts at about 550ϒC. Two distinct regions in the temperature dependence of the decomposition rate were observed; a low temperature region between 550ϒC to 650ϒC and a high temperature region between 560ϒ to 750ϒC. The X-ray diffraction analysis suggests that the decomposition of enargite occurs through the formation of intermediate tenantite, according to the following sequential reactions; $4Cu_3S_4(s)$ - Cu_{12} $As_4S_{13}(s)$ + 3 /$2S_2(g)$; $Cu_{12}As_4S_{13}(s)$ - $6Cu_2S(s)$ + $2As_2S_3(g)$ + $1/2S_2$(g. The kinetics of the decomposition was analyzed by using the model for a topochemical reaction of spherical particles. $1-(1-X)^{13}$-kt, which represented well the data. Apparent activation energies of 612 kJ/mol and 110.9 kJ/mol were found for the low and high temperature regions, respectively.

10:10 AM BREAK

10:20 AM

VIRCOSITY OF HIGH MAGNESIUM CONTENT FeO-Fe_3O_4-SiO_2-CaO-MgO SLAGS AT NICKEL SMELTING CONDITIONS: *Tan Pengfu*[1]; Zhang Chuanfu[1]; Zeng Dewen[1]; Li Zuogang[1]; Ari Jokilaakso[2]; Tapio Ahokainen[2]; [1]Central South University of Technology, Department of Nonferrous Metallurgy, Changsha 410083 China; [2]Helsinki University of Technology, Lab. of Materials Processing and Powder Metallurgy, FIN-02015, Hut, Espoo PB 6200 Finland

The viscosity of high magnesium content FeQ-Fe_3O_4-SiO_2-CaO-MgO slags at nickel smelting conditions was investigated using a commercial rotational viscometer. The studied parameters were composition of slag and temperature. The viscosity of these slags was measured as a function of CaO, MgO, SiO_2 Fe and Fe/SiO_2 in temperature range from 1473K to 1723K. These slags contained 5.81 to 10.59 pet MgO, 0 to 10.37 pct CaO, 28.21 to 36.21 pet SiO2, 39.40 to 55.42 pet Fe and 1.1 to 1.5 Fe/SiO_2.

10:45 AM

FLAME ATOMIC ABSORPTION ANALYSIS OF GOLD JEWELLERY: *M. A. Llavona*[1]; M. C. Crespo[1]; A. M. Fernandez[1]; J. L. Ibanez[1]; R. Zapico[1]; [1]University of Oviedo, Dept. Materials Science, U.S. of Mining and Topographhic Engineering, Reinerio garcia s/n, Mieres 33600 Spain

In Spain, as in many countries, the gold content of jewellery must be officially hallmarked. The official methods of assay are gravimetric cupellation (a method which is highly accurate but both time and sample consuming), potentiometry, gravimetry and volumetry. In this paper, a procedure for measuring gold by flame atomic absorption in jewellery samples is described. The method is based on the acid digestion of a 40 mg sample. The quantity of aqua regia necessary for dissolving the gold, the ideal weight of a sample in order that the error may be minimal, the effect of the acids employed, the type of material used for its storage, and the necessity of employing a buffer to avoid the ionization of the solution are described. The method proposed is simple, highly accurate and causes little damage to the jewellery.

11:10 AM

THERMODYNAMIC MODELING FOR THE EVALUATION OF THE CO-PRODUCTS BEHAVIOR IN THE NICKEL/COPPER SMELTING AND CONVERTING PROCESSES: *Florian Kongoli*[1]; [1]FLOGEN Technologies, 306-3325 Edouard Montpetit Montreal, Quebec H3T 1K4 Canada

The behavior of the co-products and byproducts in the Ni/Cu smelting and converting processes effects directly the quality of the final products as well as the revenues gained from the overall process. An effective way to evaluate and predict this important behavior is thermodynamic modeling. This work presents the thermodynamic modeling of various phases in Ni/Cu smelting and converting processes and shows how these can be used to predict and evaluate various aspects of this behavior such as the activities of different components in the matte phase, matte-slag distribution, metal-slag distribution, etc. Future developments are also discussed.

11:35 AM

PROGRESS IN UNDERSTANDING COPPER ANODE SLIMES SMELTING: S. Neven[1]; D.R. Swinbourne[2]; B. Blanpain[1]; [1]Department of Metallurgy & Materials Engineering, Catholic University of Leuven, De Croylaan 2, B-3001 Leuven, Belgium; [2]Department of Chemical & Metallurgical Engineering, RMIT University, P.O. Box 2476V, Melbourne, 3001, Australia

The smelting of copper anode slimes, a material which contains mostly lead, silver, copper and selenium, has been practiced for many years with a view to recovering the silver and associated small amounts of gold. However, a theoretical understanding of the process has been lacking and this handicaps efforts to improve and intensify smelting operations. In the past few years work has been done in this area and this work will be reviewed in the paper. The underlying thermodynamics of the oxidation of silver-copper selenide mattes has been developed and used as a basis for the construction of a computational thermodynamics model which attempts to predict the composition of all condensed phases during the course of smelting. The model will be outlined and its successes in matching experimental data, as well as its current deficiencies, will be considered. Supporting work on the thermodynamics of silver solubility in slags, on activity-composition relationships in the Ag-Se system and work in progress on the oxidation state of copper in the slags will be reviewed. These projects are designed to provide further detailed data which will improve the robustness of the thermodynamic model.

MICROMECHANICS AND MICROMECHANISMS OF DEFORMATION AND FRACTURE: A SYMPOSIUM IN HONOR OF PROFESSOR ALI S. ARGON: Ali S. Argon Symposium VII

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

Thursday AM — Room: 14B
March 4, 1999 — Location: Convention Center

Session Chair: K. Jimmy Hsia, University of Illinois at Urbana-Champaign, Dept. of Theor. & Appl. Mech., Urbana, IL 61801 USA

8:30 AM

MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF NOTCHED E-GLASS/VINYL ESTER COMPOSITE MATERIALS SUBJECTED TO THE ENVIRONMENT AND A SUSTAINED LOAD: *Stephanie E. Buck*[1]; David W. Lischer[1]; *Sia Nemat-Nasser*[1]; [1]University of California, San Diego, Center of Excellence for Advanced Materials, 9500 Gilman Drive, La Jolla, CA 92093-0416 USA

Flaws may be created in composite materials during manufacturing, machining, or use. Composites with such flaws, when subjected to adverse environmental conditions in the presence of a sustained load, may have dramatically reduced mechanical properties and changes in their microstructural properties up to failure. E-glass/vinyl ester composite coupons with a single edge flaw have been conditioned for periods up to 3000 hours at room temperature and at elevated temperatures, with and without a sustained load, to determine the changes in the tensile properties and damage mechanisms which occur with the introduction of the flaw. Several fiber architectures have been studied. The tensile strength has been found to decrease by at least fifteen percent for notched samples when compared with unnotched samples subjected to the same environmental and load conditions. The early damage mechanisms have been found to change from transverse matrix cracking, fiber breakages, and occasional edge delaminations for unnotched specimens to the growth of a significant matrix crack from the notch tip and well-defined internal delaminations for notched specimens. The notched specimens have been analyzed using a finite element model as well as existing constitutive relations.

8:50 AM

COMPACTION OF WOVEN FABRIC PREFORMS IN COMPOSITE MOLDING PROCESSES: *Tsu-Wei Chou*[1]; Baoxing Chen[1]; [1]University of Delaware, Center for Composite Materials and Department of Mechanical Engineering, Newark, DE 19716-3140 USA

Fiber composite materials based upon 2D and 3D textile preforms have received considerable attention in recent years. The merits of fabric composites include ease of fabrication, cost-effectiveness, and enhanced properties in the thickness direction. Among various composite manufacturing techniques, liquid composite molding processes, such as resin transfer molding, and resin film infusion process are of very high potential. While the preforming processes, such as weaving, knitting, braiding and stitching, orients the fibers into a skeleton of the actual part, the final fiber microstructure depends to a certain extent on the compaction of the preform to the desired thickness. The compaction of a preform due to tool closure and vacuum bagging flattens the

yarn bundles, reduces the pores and gaps among the fibers and yarns, and results in elastic deformation, nesting and inter-layer packing. All these factors enhanced the overall composite fiber volume fraction. This paper reports our recent research in the development of analytical models and methodologies for predicting the compaction behavior of woven fabric preforms in order to provide better guidance for engineering design and manufacturing of fabric composites by liquid composite molding and resin film infusion processes. The analysis focuses on the unit cell of an othogonal plain weave fibrous preform, which is composed of two sets of mutually orthogonal yarns of the same fiber (non-hybrid fabric). A 3D model of the unit cell is proposed with certain simplifying assumptions to predict the compressive behavior of yarns. Yarn nesting, yarn cross-section deformation, yarn flattening and yarn elastic deformation are considered in this analytical model. Based upon the proposed model and the beam theory in the mechanics of materials, analytical expressions for the relations among fiber volume fraction, applied compressive force and preform thickness reduction are established. Results of analytical predictions are compared with experiments. The implication of the present study on the understanding of preform permeability is also discussed.

9:10 AM

THE MONOTONIC AND FATIGUE BEHAVIOR OF CONTINUOUS FIBER-REINFORCED CERAMIC-MATRIX COMPOSITES (CFCCS) : *Naren Miriyala*[1]; Peter K. Liaw[2]; Carl J McHargue[2]; Lance L Snead[3]; [1]Solar Turbines, Inc., P.O. Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [2]The University of Tennessee, Materials Science and Engr., 434 Dougherty Engineering Building, Knoxville, TN 37996 USA; [3]Oak Ridge National Laboratory, Metals and Ceramics Division, P. O. Box 2008, MS 6087, Oak Ridge, TN 37831-6087 USA

Monotonic and fatigue tests were performed at room temperature in air, and at 1000ϒC in argon environment, on two continuous fiber reinforced ceramic matrix composites (CFCCs), namely, Nicalon/Alumina and Nicalon/SiC composites. Flexure specimens were used to perform the mechanical tests. The loads were applied either parallel or normal to the fabric plies to study the effects of fabric orientation to the loading axis on the mechanical behavior. The monotonic and fatigue behavior of the Nicalon/Alumina composite was significantly affected by the fabric orientation at room temperature and 1000ϒC, while the effects were insignificant in the Nicalon/SiC composite. The damage mechanisms responsible for the observed effects will be the focus of the paper. The results of finite element analysis (FEA) also will be presented to explain the effects of fabric orientation on the flexural behavior of laminate composites. Research supported by DOE under a subcontract from Lockheed Martin Energy Corporation (No. 11X-SV483V) to the University of Tennessee.

9:30 AM

FRACTURE TOUGHNESS AND HYDROGEN-ASSISTED CRACKING OF FORGED 304L AND 316L STAINLESS STEELS: *Brian P. Somerday*[1]; Neville R. Moody[1]; Ben C. Odegard[1]; Steve L. Robinson[1]; [1]Sandia National Laboratories, Materials Reliability Dept., P.O. Box 969, MS 9403, Livermore, CA 94551 USA

Severe localized deformation can develop during forging of stainless steel components into complex shapes, which produces microstructures characterized by elevated strength as well as elongated and recrystallized grains. The fracture behavior of such stainless steel microstructures formed from high deformation at elevated temperature has not been extensively investigated in as-forged components, particularly in hydrogen environments. This work characterizes the fracture toughness and hydrogen-assisted crack growth of 304L and 316L stainless steels that were forged to high strain levels (50% to 75%), which produced elevated-strength (greater than 500 MPa) microstructures that varied in degree of recrystallization (up to 100%) and grain aspect ratio (up to 4:1). Compact tension specimens were tested at room temperature in the as-forged condition and after charging in 140 MPa hydrogen gas at 300ϒC (equilibrium hydrogen concentrations between 0.4 - 2 at.%). Additional experiments involved constant-displacement loading of WOL specimens in 200 MPa hydrogen gas at room temperature. Mechanisms of fracture in the as-forged and hydrogen-exposed conditions are interpreted based on analysis of fracture surfaces coupled with models of ductile fracture and hydrogen embrittlement. Supported by the U.S. Dept of Energy under contract # DE-AC04-94L85000

9:50 AM BREAK

10:00 AM

DYNAMIC COMPRESSION FATIGUE OF IN-SITU REINFORCED SILICON NITRIDE AT ELEVATED TEMPERATURES - FAILURE MECHANISMS: *Gayathri Raghavendra*[1]; Sia Nemat-Nasser[1]; Mingqui Liu[1]; [1]Univeristy of California, San Diego, Center of Excellence for Advanced Materials, 9500 Gilman Drive, La Jolla, CA 92093-0416 USA

Silicon nitride is a promising candidate material for high temperature structural applications on account of its high strength and toughness up to elevated temperatures, good creep resistance, low density, low coefficient of thermal expansion and good corrosion and oxidation resistance. In order to use the material for high temperature structural applications, its quasi-static and dynamic fatigue properties need to be evaluated over a range of temperatures. This paper focuses on the dynamic compression fatigue of in-situ reinforced silicon nitride over a range of temperatures from room temperature to 1000ϒC. The unique experimental facilities that have been developed in order to conduct high strain rate tests at elevated temperatures will be described. Novel techniques that have been developed to accurately measure strains in hard ceramics [like silicon nitride] will also be presented. A detailed discussion of the experimental results obtained from high strain rate tests conducted using the Enhanced UCSD Hopkinson Bar Technique over a range of temperatures will be given. In addition, the results of microstructural analysis, including Scanning Electron Microscopy, Transmission Electron Microscopy, High Resolution Transmission Electron Microscopy and X-Ray Diffraction, on as-received material as well as tested material will be presented. Finally, the mechanisms of fatigue crack growth in in-situ reinforced silicon nitride and the toughening mechanisms will be discussed.

10:20 AM

DYNAMIC BEHAVIOR OF SiC UNDER UNIAXIAL COMPRESSION.: *Sai Sushilkumar V. Sarva*[1]; Sia Nemat-Nasser[1]; [1]University of California, San Diego, Center of Excellence for Advanced Materials, 9500 Gilman Drive, La Jolla, CA 92093-0416 USA

SiC is used in important impact related applications, such as integrated armor. This necessitates development of constitutive models which describe its mechanical behavior. Experimental results which relate the strain rate to the compressive strength of SiC under uniaxial loading are obtained. Quasi-static tests are conducted using a servo-hydraulic Instron universal testing machine. Dynamic tests are performed using a modified split-Hopkinson pressure bar. It is seen that there is a marked increase in compressive strength at strain-rates higher than 10^2/s. The failure modes are similar in specimens loaded at both low and high strain rates. At peak stress these specimens fail by axial splitting. This paper compares the experimental results to those predicted by a simple model of interacting, dynamically growing wing cracks which coalesce to cause failure of a brittle solid.

10:40 AM

MICROMECHANISM OF BRITTLE FRACTURE IN MEDIUM CARBON MICROALLOYED STEELS: *Djordje Drobnjak*[1]; Husain Hraam[1]; [1]University of Belgrade, Faculty of Tech & Metall, Karnegijeva 4, P.O. Box 3503, 11120 Belgrade Yugoslavia

The relationship between microstructural parameters and cleavage fracture has been studied in (950-1300 $_0$ C) air cooled V-microalloyed steels by means of impact testing, light microscopy and scanning electron microscopy. Large variation in impact energy are obtained as a function of test temperature and microstructure. The results show that acicular ferrite (AF) and a multi-phase structure consisting of ferrite-pearlite (FP) and 30-70% AF possess room temperature toughness superior to that of classical bainitic sheaves, (BS) as revealed by impact energy level. However, AF is superior to FP-AF in terms of energy transition temperature. At liquid nitrogen temperature, all steel grades show similar behavior. Transganular cleavage is an exclusive mode of fracture. Primary brittle nuclei, which control the critical tensile strength for fracture, σ_F, are found to be brittle TiN particles of diameter >2μm.

Large TiN particles are friendly, because the cracks are initiated by stress much smaller than that required for crack progagation. This means that cracks will become blunted out, what will make them inactive before the stress for crack propagation is achieved. Carbides and martensite/austenitc/carbide MAC consituent are tentatively identified as secondary brittle fracture nuclei, but they are of less significance. As the critical stage of brittle fracture is crack propagation through particle/matrix interface, the morphology and ferrite grain size play little role. Calculation of maximum tensile stress below the notch, σ max, and critical tensile strength for fracture, σ F, under assumption that the diameter of TiN particle, which are the primary nuclei, are equal to penny shaped crack size, have shown that the requirements for crack propagation through particle/matrix interface, $\sigma_{max} > \sigma_F$, is satisfied from the beginning of fracture process at liquid nitrogen temperature in all steels studied in this work. Room temperature behavior of these steels is considerably different. The dominant fracture mechanism is still transgranulur cleavage, but this preceded by a lower (BS steels) or higher (AF steels) degree of ductile fracture. Calculations have shown that at the beginning of fracture $\sigma_{max} < \sigma_F$. This means that the brittle crack initiated in brittle particles can not propagate; instead, the ductile crack will be initiated and propagated. During propagation, ductile crack is assumed to accelerate, what, in turn, increases the strain rate, and consequently, σ_Y and σ_{max}. At a critical ductile crack length, critical condition: $\sigma_{max} > \sigma_F$ is achieved, and brittle cracks, initiated in brittle particles ahead of the ductile crack tip are activated. These progagate across particle/matrix interface and cause the fracture. Higher toughness of steels with AF structure requires a longer ductile crack to be formed, than in steels with BS structure, before requirement for brittle fracture, $\sigma_{max} - \sigma_Y * n > \sigma_F$ is attained, because the strain hardening exponent (n) of the former steel is much lower and the product $\sigma_Y * n$ is smaller in spite of σ_Y is higher. The overall contribution of ductile fracture to the toughness is relatively small because the shear decohesion mechanism, which dominates ductile fracture in both steels, is characterized by a low expenditure of energy. In spite of TiN inclusions are present, the primary nuclei are assumed to be carbides, smaller than 1 μ m. The TiN-cracks are blunted out before the conditions for cleavage are attained. This can be ascribed to influence of a large plastic zone which is produced ahead of the ductile crack. In addition to being more resistant to brittle crack propagation across particle/matrix interface, the steels with AF structure show the susceptibility to cracks being arrested at the grain boundaries presumably AF plate boundaries. This feature is observed only in steels with AF and not with BS structure, providing thus additional burriers to crack propagation in the former.

SURFACE ENGINEERING: SCIENCE AND TECHNOLOGY I: Solid Freeform Fabrication

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee; Shaping and Forming Committee; Structural Materials Division, Structural Materials 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

Thursday AM Room: 7B
March 4, 1999 Location: Convention Center

Session Chairs: John L. Lombardi, Advanced Ceramic Research, Tuscon, AZ 85706-5013 USA; John E. Smugeresky, Sandia National Laboratory, Livermore, CA 94551 USA

8:30 AM
BALLISTIC TESTING OF LASER FREE-FORM FABRICATED Ti-6Al-4V: *William Herman*[1]; Eric Whitney[2]; [1]General Dynamics, Land Systems Division, 6000 East Seventeen Mile Rd., Sterling Heights, MI 48313-4500 USA; [2]Pennsylvania State University, Applied Research Laboratory, P.O. Box 30, State College, PA 16804 USA

Laser free form fabrication is a process to manufacture complex shapes without the use of molds or dies. The process utilizes a laser to melt and fuse powder or wire along a predefined computer controlled path. The fabrication of structural shapes differs from prototyping in that the material produced for structural applications must, naturally, be fit for use. One application for laser free form fabricated Ti-6Al-4V shapes is the manufacture of large, lightweight components for armored vehicles. This study compares the ballistic properties of Ti-6Al-4V material produced in a high power (14 kW CO2) laser free form process to conventionally prepared wrought and cast Ti-6Al-4V. Tensile and chemical tests were also prepared and compared to previously laser fabricated Ti-6Al-4V. Material was laser fabricated to meet the chemistry of MIL-T-9046 using gas atomized pre-alloyed powder. Ballistic testing using 20mm Fragment Simulated Projectiles and 0.50 caliber armor piecing rounds was conducted in accordance with MIL-A-46077.

8:50 AM
FABRICATION OF TOOL STEEL INSERTS FROM SFF PATTERNS USING THE RSP TOOLING PROCESS: *Kevin M. McHugh*[1]; [1]INEEL/Lockheed Martin, P.O. Box 1625, Idaho Falls, ID 83415-2050 USA

Rapid Solidification Process (RSP) Tooling is an alternative method for making tooling inserts which shows promise for reducing fabrication time and cost compared to conventional approaches. The approach combines rapid solidification of metals with net-shape materials processing in a single step. A molten tool forming alloy is atomized by the action of a high velocity gas jet to form very fine droplets that are entrained by the jet and deposited onto an SFF or other pattern. As the deposit builds up, it faithfully replicates the pattern's shape, surface texture and surface detail. Production rates are high, on the order of 500 lbs./hr using a bench scale system, allowing the inserts to be spray formed in minutes. This paper summarizes recent advances in processing and describes how material and microstructural properties transform during solidification and subsequent heat treatments.

9:10 AM

ESTIMATION OF LASER POWER FOR FREE-FORM FABRICATION: *Eric Whitney*[1]; [1]Pennsylvania State University, Applied Research Laboratory, P.O. Box 30, State College, PA 16804 USA

An energy balance approach for the estimation of the laser power needed to perform a laser free-form fabrication process is discussed. The approach takes into account the melting of the filler metal and a small portion of the substrate. The technique employed utilizes a modification of the energy balance approach taken for laser welding. Although a strict energy balance does not take into consideration other process requirements, such complete fusion between beads and layers and other metallurgical requirements, a simple set of rules can be used to impose practical limits on the energy balance. The approach is applied to commercially pure titanium and Ti-6Al-4V alloy.

9:30 AM

LASER FREE-FORM FABRICATION OF NICKEL-ALUMINUM-BRONZE: *Eric Whitney*[1]; Kenneth C. Meinert[1]; [1]Pennsylvania State University, Applied Research Laboratory, P.O. Box 30, State College, PA 16804 USA

The fabrication of the large structural shapes using laser free form fabrication techniques is of great practical interest because it offers the potential of significantly reducing costs, particularly for low volume production runs. Implementing laser free from fabrication technology can also reduce costs by eliminating the need to inventory a large quantity of specific tooling. This benefit is especially true for the procurement of replacement parts that are no longer manufactured as part of new production. Another benefit of laser free from fabrication process is the capability of making functionally graded compositions. In this paper the results of feasibility study on laser free from fabrication of a nickel-aluminum-bronze (NAB) alloy. Laser processing was performed using a 14 kW CO2 laser and a specially constructed environmental chamber. The environmental chamber was purged with oxygen such that the oxygen level in the chamber was less than 200 ppm. Chemical analysis showed that chemical composition between the precursor powder and the deposit was essentially unchanged by the laser deposition process. Mechanical properties were also tested and shown to be comparable to conventionally cast NAB. Finally, a functionally graded shape was made where the composition was initially that of Alloy 625 and graded to NAB.

9:50 AM

LASER-DEPOSITED HIGH TEMPERATURE MATERIALS: *Xiao-Dong Zhang*[1]; Richard Grylls[1]; Hamish Fraser[1]; [1]The Ohio State University, Materials Science and Engineering, 2041 College Rd., Columbus, OH 43210 USA

The objective of this work is to characterize the microstructure of high temperature materials produced by Laser Engineered Net Shaping (LENS®). LENS is a laser direct metal deposition process that combines laser cladding technologies with advanced rapid prototyping methods with the capability to directly manufacture complex three-dimensional components. Various alloys have been deposited including TiAl and Ni-based superalloys. Components were generated using a CW Nd: YAG laser operated at about 150-200 watts. Characterization was conducted using optical microscopy, SEM and TEM. Different microstructure can be obtained depended on the processing parameters (laser scan speed, powder feed rate and pressure, substrate subtract materials and post-heat- treatment). A number of high temperature metastable phases have been retained at room temperature in several of these alloys. By careful control of post- heat- treatment, optimum microstructures and mechanical properties may be obtained. This work will compare the microstructures of these laser-deposited materials with those typically seen in materials produced by more conventional means. The significance of the LENS® technique will be discussed in terms of near net-shape manufacture, microstructural control and potential applications.

10:10 AM BREAK

10:25 AM

ISSUES ASSOCIATED WITH THE DEVELOPMENT OF A WATER SOLUBLE SUPPORT FOR USE IN FREEFORMING: *John L. Lombardi*[1]; Gregory G. Artz[1]; Ranji Vaidyanathan[1]; [1]Advanced Ceramics Research, Inc., 3922 East Hemisphere Loop, Tuscon, AZ 85706-5013 USA

Freeforming are promising techniques for fabricating complex shaped prototype and low volume production components. Unfortunately many prototypes have complicated geometries (i.e. overhangs or internal features) such that they are not easily fabricated by these techniques without the assistance of a fugitive material to support the freeformed layers. Conventional freeforming techniques utilize a fugitive support material which must be removed from the completed part using either mechanical or thermal pyrolysis means. This paper discusses the development and characterization of a fugitive support which is soluble in aqueous solution.

10:45 AM INVITED PAPER

HIGH TEMPERATURE FILTERS MADE BY CERAPRINT♦: *Mark V. Parish*[1]; Andrew B. Jeffrey[1]; [1]Specific Surface Corporation, 101 Constitution Blvd., Franklin, MA 02038 USA

Ceramic and metal filters made using a manufacturing process called CeraPrint♦ will be discussed. CeraPrint♦ is based on Three Dimensional Printing♦ technology developed at MIT. Filters are made with controlled macro- and micro- structures using this manufacturing process that uses advanced liquid jetting technology and readily available powders. CeraPrint♦ allows a high degree of design and materials flexibility that is necessary in order for ceramic and metals to compete in the filtration market. Complex filter designs can be made without tooling or molds in a competitive manner, allowing for high surface area designs with uniform and controlled porosity. The process is scalable to virtually any size. Our CleanStac♦ filters, for example, have been designed for conventional filtration baghouses where operating temperatures of up to 600ϒC are desired. Operation in corrosive gas streams is also achieved with CleanStac♦. The presentation will include discussion of operations of CleanStac♦ filters in COHPAC I an II power generation pilot plants . In COHPAC I, CleanStac♦ rigid ceramic filters operated for 2,000 hours under a variety of conditions to test baghouse applications. Results have shown very good performance with low pressure drop and high filtration efficiency. CleanStac♦ filters were shown to be cleaned effectively using existing pulse jet technology.

11:15 AM

FUNCTIONALLY GRADIENT CERAMIC COMPOSITES VIA FUSED DEPOSITION PROCESS: *Raj Atisivan*[1]; Ashwin Hattiagadi[1]; M. Rubiela Diaz[1]; Susmita Bose[1]; [1]Washington State University, Amit Bandyopadhyad School of Mechanical and Materials Engineering, Pullman, WV 99164-2920 USA

Functionally gradient materials (FGM) show a variety of properties from one end of the material to the other end due to its compositional variation. Fused deposition modeling (FDM) process has been utilized to process functionally gradient ceramic-polymer and metal-ceramic composites. Porous ceramic preforms with a gradient in porosity from one end to the other are fabricated via indirect freeform fabrication route. The porous ceramic preforms are then infiltrated with polymers and metals to form a ceramic-polymer and metal-ceramic composites. Room temperature mechanical properties of these composites are tested with respect to the volume fraction ceramics content. In this presentation, functionally gradient alumina, mullite and silica ceramic composites will be discussed.

11:35 AM

EXTRUSION FREEFORM FABRICATION OF SILICON NITRIDE PARTS: *Ranji Vaidyanathan*[1]; John L. Lombardi[1]; Blake Tennison[1]; Sridhar Kasichainula[2]; P. Calvert[2]; [1]Advanced Ceramics Research, Inc., Tuscon, AZ 85706 USA; [2]University of Arizona, Arizona Materials Laboratories, Tuscon, AZ 85713 USA

Extrusion Freeforming (EFF) and Fused Deposition Modeling (FDM) processes are established freeforming techniques capable of fabricating complex shaped ceramic prototypes by the sequential deposition and solidification of green ceramic feedstock, layer by layer until the final part results. This paper will detail the development and characterization of a complicated ceramic prototype part using the EFF process and ACR's patented high-pressure extrusion head technology. Development of a suitable ceramic filled binder system for the high-pressure extrusion

head will be presented. Optimization of the binder burnout and sintering cycles, and characterization of the parts will also be discussed.

SYNTHESIS OF LIGHTWEIGHT METALS III: Magnesium

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. Centre, Farnborough, Hampshire GUI14OLX UK; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Neqev Israel; P. G. McCormick, University of W. Australia, Res. Centre for Adv. Min. & Mat. Proc., Nedlands, W.A. 6907 Australia

Thursday AM Room: 10
March 4, 1999 Location: Convention Center

Session Chairs: P. G. McCormick, University of Western Australia, Research Centre for Adv. Min. and Mats. Proc., Nedlands, Western Australia 6907; D. Eliezer, Ben Gurion University, Dept. of Mats. Eng., Beer-Sheva 84105 Israel

8:30 AM INVITED PAPER

OVERVIEW OF MAGNESIUM - PART I: *Dan Eliezer*[1]; Eli Aghion[2]; F. H. (Sam) Froes[3]; [1]Ben-Gurion University of the Negev, Dept. of Mats. Eng., P.O. Box 653, Beer-Sheva 84105 Israel; [2]Dead Sea Magnesium Works, Ltd., Potash House, P.O. Box 75, Beer Sheva 84100 Israel; [3]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The very low density and excellent castability of magnesium is leading to increased use in various applications, especially in automobiles, despite poor galvanic corrosion resistance and a higher cost than aluminum. Further expansion of the magnesium market should come from reduced cost, and increased design base, a better understanding of the scientific underpinning of magnesium alloys, improved protection systems, and the development of cost-affordable cast and wrought products. Magnesium is considered as an environmentally friendly material that can be easily recycled. The potential use of magnesium is strongly related to environmental conservation awareness.

8:50 AM INVITED PAPER

OVERVIEW OF MAGNESIUM - PART II: *Dan Eliezer*[1]; Eli Aghion[2]; F. H. (Sam) Froes[3]; [1]Ben-Gurion University of the Negev, Dept. of Mats. Eng., P.O. Box 653, Beer-Sheva 84105 Israel; [2]Dead Sea Magnesium Works, Ltd., Potash House, P.O. Box 75, Beer Sheva 84100 Israel; [3]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

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9:10 AM

SYNTHESIS OF Mg2Si BY MECHANICAL ALLOYING: *A. Alvarez*[1]; E.G. Baburaj[1]; Swati Ghosh[1]; F. H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Magnesium alloys containing Mg_2Si particles have the potential for applications as light weight alloys for relatively higher temperatures compared to other Mg alloys, because of their high melting point, low density, high hardness and low coefficient of thermal expansion. In the present work a combination of mechanical alloying and heat treatments has been employed to synthesize Mg_2Si. During mechanical alloying, powder particles fracture and re-weld resulting in the formation of lamellar structure. With increasing milling time lamellae thickness decreases, while defect concentration within the lamellar structure increases. Due to the large concentration of defects at the inter-lamellar boundaries diffusion becomes easier around these defect concentrations and hence continued milling results in the formation of Mg_2Si intermetallic compound. A set of experiments has also been carried out to ascertain the inter-dependence of milling time and heat treatments on the formation of the intermetallic. The compound formation temperature has been found to decrease with increase in milling time.

9:30 AM BREAK

9:45 AM

THE ROLE OF SECOND PHASES IS THE CORROSION MECHANISM OF MAGNESIUM AZ91 ALLOY: *P. Uzan*[1]; D. Eliezer[1]; E. Aghion[2]; [1]Ben-Gurion University of the Negev, Dept. of Mats. Eng., P.O. Box 653, Beer-Sheva 84105 Israel; [2]Dead Sea Magnesium, Ltd., Magnesium Research Division, P.O. Box 75, Beer-Sheva, 84100 Israel

Due to the light weight of magnesium alloys they have been continuously considered as structural materials in the transportation and electric industries. The most common Mg alloy in use is the AZ91D which has adequate corrosion resistance. However, one of the major causes for corrosion deterioration is the presence of impurities, non-metallic inclusions and intermetallic phases. Some of these phases may be more cathodic comparing to the Mg matrix and hence it is important to understand their effect on the corrosion mechanism of the magnesium alloys. The present study aims to evaluate the corrosion behavior of AZ91 alloy in view of the above phases. This paper will emphasize the effect of the second phases on the corrosion mechanism with the focus on the effect of the β ($Mg_{17}Al_{12}$) phase that will be discussed in detail. In order to understand the effect of the β phase, the corrosion performance of Mg AZ91 alloy in as cast (F), homogenized (T4) and artificially-aged (T6) conditions were evaluated under immersion test conditions and potentiodynamic polarization analysis in NaCl and $Mg(OH)_2$ electrolyte.

10:05 AM

MICROSTRUCTURE REFINEMENT OF Al-Mg-Si CAST INTERMETALLIC ALLOYS: *M. Bamberger*[1]; [1]Technion, Dept. of Mats. Eng., Israel Institute of Technology, Haifa 32000 Israel

Nano-crystalline powders added to molten metallic-alloys can modify the as-cast microstructure by inoculation, or by restricting grain growth of the primarily solidified intermetallic phase. The modification mechanism of the primarily solidified Mg_2Si, formed during the solidification of Mg_2Si- 50%wtAl intermetallic alloys, by nano-crystalline TiCN was investigated. Up to 10%wt of nano-crystalline TiCN powder (typical mean particle size 45-70nm) was added to molten Mg-Si-Al alloys. The microstructure of the modified and un-modified alloys was investigated using SEM/EDS, TEM/EDS, X-ray diffraction and optical microscopy. In addition, "model experiments", in which Mg-alloys were infiltrated into porous TiCN, were used to investigate their solidification sequence. Inoculation with TiCN leads to a decrease in the grain size of the primarily solidified Mg_2Si from 25-80μm to 0.1-0.8μm. The microhardness of the regions containing the ultrafine microstructure was 314-428Hv, as compared to 129-142Hv obtained from the unmodified structure. No cracks were detected after microhardness (1000gr) tests in modified regions, whereas cracks were found in the un-modified microstructure even after loading at only 100gr. Microstructural analysis revealed rejection of the TiCN from the intermetallic phase, which in turn limits growth of the Mg_2Si during solidification, and hence a fine microstructure is obtained by restricting grain growth mechanism.

10:25 AM

STRUCTURAL STABILITY AND CREEP PROPERTIES OF AZ91:

M. Regev[1]; A. Rosen[1]; M. Bamberger[1]; [1]Technion, Dept. of Mats. Eng., Israel Institute of Technology, Haifa 32000 Israel

Creep properties of AZ91D magnesium alloy (9%Al - 1%Zn) ingot castings having different microstructure were investigated in this research work within the temperature range of 120-180ϒC. The specimens of the ingot (taken from the equi-axed grains zone) are characterized by a relatively coarse grain size (~300μm) of magnesium matrix containing aluminum in solid solution and β($Mg_{17}Al_{12}$) precipitates. Aging study of the ingot casting, performed at the temperatures of 120ϒC and 180ϒC, yielded that the microstructure is thermally unstable at these temperatures. Aged specimens were investigated by optical and scanning electron microscopes and sub micron precipitates of β($Mg_{17}Al_{12}$) were detected near the matrix grain boundaries. Creep tests were performed on ingot specimens under three conditions: in the as cast condition, after solution heat treatment and after aging heat treatment.