TUESDAY AM

Daily Personal Schedule - Tuesday - March 14

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
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11:00 am				
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6:00 pm				
6:30 pm				

Tutorial Luncheon Lecture
"Ancient Arts of Sword Making"
Daniel Eylon
12:00noon-1:30pm
Convention Center, Canal B

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EPD Luncheon
"Bridging the Gap Between Technology Development
& Commercial Applications"
Edward Dowling
12:00noon
Convention Center, Presidential Ballroom - Adams A

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EPD Distinguished Lecturer
"Aspects of Technology Transfer"
Derek Fray
1:45pm
Convention Center, Presidential Ballroom - Jefferson A

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 1

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

Tuesday AM — Room: Memphis A
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Hideki Minagawa, Hokkaido National Industrial Research Institute, Materials Division, Toyohira-ku, Sapporo 062-8517 Japan

8:30 AM Welcome and Introductions
R. A. Schiffman, R.S. Research Inc., Crystal Lake, Barton, VT 05822 USA

8:50 AM
Halting Convection during Solidification Experiments by Application of a Susceptibility Dependent Magnetic Body Force: *Chris Seybert*[1]; J. W. Evans[1]; W. K. Jones[2]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., Berkeley, CA 94720 USA; [2]Motorola Inc., 8000 W. Sunrise, Plantation, FL 33324 USA

The role of convection in transporting solute during solidification is well known. Ground-based experiments where such convection is eliminated, e.g. to study solidification in the diffusive limit, have not been successful and several solidification experiments in Earth orbit have been carried out with NASA support. The paper describes an experimental investigation with associated mathematical modeling, that is intended to eliminate convection in ground-based experiments, or to further still convection in microgravity. This is achieved by exploiting a magnetic body force that is dependent on the magnetic susceptibility of the material under investigation and the fact that susceptibilities are temperature dependent (or concentration dependent in the case of solutions). In a sufficient magnetic field (gradient), the reduction in gravitational body force resulting from expansion (or a concentration change) can be balanced by an opposite change in the magnetic body force. Sufficient fields require superconducting magnets on Earth but simple solenoids may suffice in space. Experiments have been conducted in a superconducting magnet at Marshall Space Flight Center to measure velocities in convecting solutions as a function of field strength using particle image velocimetry. A cell with a hot wall at one end and a cold at the other (the other walls being glass) was position a little below the magnetic center of the magnet. A particular current has been identified at which convection is halted, to within the precision of the measurements. Initial results have been on experiments without solidification but similar results with solidification should be available at the time of the conference. Results are interpreted in terms of mathematical models for the magnetic field and the flow. Research supported by NASA.

9:10 AM
The Influence of Gravity on Particle Pushing and Agglomeration during the Freezing of Reinforced Aluminum Alloys: *Reginald W. Smith*[1]; [1]Queen's University, Dept. of Matls. and Metallu. Eng., Kingston K7L 3N6 Canada

Particle reinforced metal matrix composites (PRMMCs) appear to have significant industrial potential, however there are still processing problems associated with them. The main obstacle to good MMC processing is the limited ability to control particle pushing, particle settling, and particle clustering. Experimental results given in the literature have been inconclusive as to estimating the critical velocity, a term which describes the freezing rate above which a particle is engulfed rather than pushed, and for which calculated rates appear to vary over a range of 10^4 when applied to the Al + SiC particle system. A series of experiments were performed on the Russian Space Station MIR in an attempt to further understand these problems in PRMMC processing. A semi-automated furnace apparatus was used on MIR for the particle pushing experiments. The samples consisted of three different PRMMC systems processed at growth rates varying from 1μm/sec to 100μm/sec. Some particle pushing was observed under all of the growth conditions examined. However, during cellular growth, most of the pushing was done laterally so that the reinforcement eventually became trapped between cellular projections. The effects of G-Jitter were also examined and found to increase the critical velocity for engulfment. These results will be discussed in the light of current theory for the interaction of inclusions suspended in the liquid with themselves and advancing solid/liquid interface.

9:30 AM
Containerless Solidification of Zr63Al10Ni11Cu16 Alloy Using a Short Drop Tube: *Takahiro Suwa*[1]; Toshio Suzuki[1]; Kazuto Tokumitsu[1]; Katuhisa Nagayama[2]; [1]The University of Tokyo, Dept. of Metallu., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]Shibaura Institute of Technology, Dept. of Matls. Sci., 3-9-14, Minato-ku, Tokyo 108-8548 Japan

By ejecting the molten alloy from the nozzle of a silica crucible into a short drop tube filled with helium gas, the small droplets of Zr63Al10Ni11Cu16 alloy were containerlessly solidified. By measuring the crystallization heat of droplets the amorphous fractions were determined for droplets within certain diameter ranges. The obtained relationship between the amorphous fraction and the droplet diameter was compared with those obtained from the classical nucleation theory. The result showed the homogeneous nucleation was attained in the experiments. The fine particles of TiB2, TiC and SiC were added in the alloy and the same kinds of experiments were carried out. The heterogeneous nucleation by the addition of the particles was confirmed.

9:50 AM
Thermodynamic Properties of Bulk Metallic Glass Forming Alloys in the Stable and Undercooled Melt: *R. K. Wunderlich*[1]; H.-J. Fecht[2]; D. S. Lee[3]; S. Glade[3]; W. L. Johnson[3]; [1]Technical University of Berlin, Instit. of Metals Rsch., Hardenbergstr. 36, PN 2-3, Berlin

10623 Germany; [2]University of Ulm, Dept. of Eng., Albert-Einstein-Allee 47, Ulm 89081 Germany; [3]California Institute of Technology, Keck Lab. for Eng. Matls., 138-78, Pasadena, CA 91125 USA

The thermophysical properties of the metallic glass forming alloys $Zr_{65}Al_{7.5}Cu_{7.5}Ni_{10}$, $Zr_{60}Al_{10}Cu_{18}Ni_9Co_3$, $Ti_{34}Zr_{11}Cu_{47}Ni_8$ and, $Zr_{57}Nb_5Cu_{1_{5.4}}Al_{10}$ have been investigated in the stable and undercooled melt in a reduced gravity environment. The specimen were processed in the electromagnetic containerless processing device TEMPUS on board spacelab. Specific heat measurements were performed by the method of non contact accalorimetry. Combined with c_p data obtained by heating a glass prepared in earth laboratory by rapid quenching, the thermodynamic functions of these alloys have been evaluated over a large range in temperature allowing a systematic comparison of the thermodynamic factors determining glass forming ability of these alloys. These data are combined with results from ground based viscosity measurements to model temperature-time-transformation curves with surface tension as a free parameter. Comparison with temperature-time-transformation curves measured in a electrostatic levitation device allows estimates of the interfacial tension and the influence of heterogeneous nucleants on the glass forming ability in these alloys.

10:10 AM Break

10:30 AM

Gravitational Effects on Combustion Synthesis of Advanced Porous Materials: *X. Zhang*[1]; J. J. Moore[1]; F. D. Schowengerdt[1]; D. P. Johnson[1]; [1]Colorado School of Mines, Ctr. for Commercial App. of Combustion in Space, Golden, CO 80401-1887 USA

Combustion of self-propagating high-temperature synthesis (SHS) of the B_4C-Al_2O_3, Ti-TiB_x and NiTi-TiB_x systems has been studied with respect to their sensitivity to the SHS reaction parameters of stoichiometry, green density, gasifying agents, ambient pressure, diluents and microgravity in an effort to engineer the required porosity and mechanical properties in these composite materials to meet the requirements of a consumer, such as for the application of bone replacement materials. Gravity serves to restrict the gas expansion and the liquid movement during SHS reaction, as a result, it affects the microstructure and properties of the SHS products. Microgravity testing in KC135 has extended the ability to form porous products. This paper will emphasize the gravitational effects on the SHS process, the microstructure and properties of those porous composites, by comparing the combustion synthesis of porous materials under microgravity (~Og), normal gravity (1g), and higher gravity (~2g).

10:50 AM

The Measurement of Diffusion Coefficient of Molten Eutectic Alloys by Using the Shear Cell under Microgravity: *Tadahiko Masaki*[1]; Satoshi Matsumoto[1]; Shuji Munejiri[1]; Misako Uchida[1]; Hirokazu Kato[1]; Makoto Natsuisaka[1]; Shinichi Yoda[1]; Tomihisa Nakamura[1]; Yasuhiro Nakamura[1]; Naokiyo Koshikawa[1]; Minoru Kaneko[1]; Tomoharu Fukazawa[1]; Yoshito Arai[1]; Kyoichi Kinoshita[1]; Toshio Itami[1]; [1]National Space Development Agency of Japan, Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

The measurement of diffusion coefficient of high temperature melts is one of the most important subjects for the utilization of microgravity environment. The shear cell is an advanced and capable method for the diffusion experiment. The shear cell for the high temperature melts was developed and tested due to the sounding rocket TR-IA 7th. The shear cell was applied to the diffusion experiment of molten Ag-Cu alloys and InGaAs semiconductors. The six furnaces were boarded on the sounding rocket. Ten or five different diffusion couples were contained within each furnace. The microgravity experiment was performed successfully in November 1998. The isotope diffusion and interdiffusion coefficient of molten Ag-Cu alloys were determined in the 6 different compositions at three different temperatures. Interdiffusion coefficients of molten InGaAs semiconductor at three different temperatures were also determined. The correlation between the experimental results under microgravity and on ground is discussed.

11:10 AM

Interfacial Temperature Measurements using Seebeck Effect during Directional Solidification of Bi-Sn Alloys: Y. Lian[1]; F. Chen[1]; *R. Abbaschian*[1]; H. C. de Groh[2]; [1]University of Florida, Matls. Sci. and Eng., P.O. Box 116400, Gainesville, FL 32611 USA; [2]NASA, Matls. Div., Processing Sci. and Tech. Brnch., M/S 105-1, Cleveland, OH 44135 USA

The Seebeck technique was used to measure the solid/liquid interfacial undercooling during directional solidification of Bi-lat%Sn. The measurements were conducted under microgravity conditions during the STS-87 flight of the space shuttle Columbia using the MEPHISTO directional solidification facility. The results show that the Seebeck signals depend not only on the interface velocity and growth distance, but also on the structure of the solid behind the interface. The latter, termed the structural Seebeck, is due to the translation of the solidified structure through the temperature gradient zone behind the interface. The structural Seebeck contributions to the solidification measurements were determined by the post-flight analysis of the space-grown samples.

11:30 AM

A Unique Sample Quench Technology for Microgravity Furnaces: *Michael R. Fiske*[1]; April M. Heaton[1]; Kerry D. Moody[1]; Daniel Popok[2]; [1]Morgan Research Corporation, Matls. Sci., 2707 Artie St., Ste. 17, Huntsville, AL 35805 USA; [2]Popok Analytical Consulting & Engineering Inc., 6302 Homestead Rd., N.E., Huntsville, AL 35811 USA

NASA's Microgravity Materials Science research programs require rapid cooling of samples to enhance microstructural analysis, evaluate concentration/impurity profiles, and preserve the geometry of the solid-liquid interface. A sample quench rate of up to 100°C/sec is desired which has been shown to be unattainable using traditional quench systems and Sample Ampoule Cartridge Assembly (SACA) designs. This paper will present the results of a Phase I SBIR project aimed specifically at solving this problem. A unique "Quench-in-Cartridge" (QIC) system has been designed for SACAs in which the quench media (water and liquid gallium have been evaluated) is delivered internal to a metallic containment cartridge. This concept reduces furnace design complexity and allows it to be kept independent of SACA design while achieving significantly higher quench rates. None of the existing or planned quench system designs/methodologies incorporated in either terrestrial or microgravity furnaces offer the simplicity and overwhelming advantages provided by the QIC concept.

Advanced Technologies for Superalloy Affordability: Affordability Technology for Casting

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Tuesday AM — Room: Canal C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: David Furrer, Ladish Company, Cudahy, WI 53110 USA; Gerhard Fuchs, University of Florida, Gainesville, FL 32611 USA

8:30 AM Invited

Effect of Trace Elements on Directional Solidification of Superalloys: Yaoxiao Zhu[1]; Langhong Lou[1]; Baiyun Tong[1]; *Changxu Shi*[1]; [1]National Natural Science Foundation of China, 35 Huayuan Beilu, E. Gate, Beijing 100083 China

Technology of low segregation superalloy controlled by trace elements was developed two decades ago at Institute of Metal Research in

China. The principle and experimental effects will be described. The Low segregation technology used for superalloy development is very effective in the following way. (1) New alloys could be used 20-25°C higher then conventional ones at similar composition. (2) Nickel base superalloy that can substitute cobalt base superalloy X-40 with superion properties has been developed by low segregation technology. (3) Low segregation technology can make more superalloys suitable for directional solidification with pronounced improvement of properties at elevated temperatures. (4) The properties of DS superalloy Rene'125 can be improved with low segregation technology without addition of Hf. Experimental facts with explanation will be presented.

8:55 AM Invited

Large Diameter Superalloy Ingots: *Richard L. Kennedy*[1]; Laurence A. Jackman[1]; A. Stewart Ballantyne[1]; Betsy J. Bond[1]; [1]Allvac, An Allegheny Teledyne Co., 2020 Ashcraft Ave., Monroe, NC 28110 USA

In recent years, there has been a very substantial increase in the size and operating temperature of turbine engines for both aircraft and land base power generation. This has led to the demand for larger superalloy components. Processing and current size capabilities are reviewed for several superalloys with reference to metallurgical defects, which can result from the melting process. These can include freckles, dirty white spots and oxide-nitride inclusions. Many of these defects may be detected as ultrasonic indications in finished forgings. Smaller components, made from small diameter forged billet, allow for an in-process inspection and selective removal of such defects at the billet stage. This is not possible for very large parts where the entire ingot is one part. With continuing improvements in the technology of melting superalloys, it has been possible to meet today's requirements for larger parts with the conventional and low cost cast-wrought approach.

9:20 AM Invited

Niobium Segregation in 718 VAR Ingots: *J. Brooks*[1]; J. Krafcik[1]; J. VanDenAvyle[1]; [1]Sandia National Laboratories, P.O. Box 969, MS 9402, Livermore, CA 94550 USA

Segregation of Nb can be a major issue in high quality rotating grade Alloy 718. The tight commercial specifications on Nb content, and on Nb containing macrosegregation defects can only be met if large ingots are melted under closely controlled conditions. To better understand the segregation of Nb and the formation of defects, ingots have been melted over a wide range of processing parameters. Ingots have been longitudinally sectioned and compositionally analyzed using x-ray fluorescence techniques to generate composition maps over the total ingot cross section, as well as in isolated regions at higher spatial resolution. The nature of Nb segregation, its relationship to melting conditions, and the mechanisms by which the segregation occurs will be discussed. This work was supported by the U.S. Department of Energy under contract DE-AC04-94AL85000, the Specialty Metals Processing Consortium.

9:45 AM

Modeling of Grain Movement and its Influence on Microstructure Evolution during Solidification of Superalloy IN718: *B. J. Yang*[1]; D. M. Stefanescu[1]; [1]The University of Alabama, Solid. Lab., P.O. Box 870202, Tuscaloosa, AL 35487 USA

An existing model was further developed to account for the effect of grain movement on the microstructure evolution and the final microstructure of alloys. The continuum model is used in the macro-transport calculation. It considers thermosolutal flow, solidification contraction. The micro-transport model is coupled to a solidification kinetics model that assumes continuous nucleation and calculates grain growth. A distribution function is introduced to handle the non-uniformity of grain size for each control volume. At this time only equiaxed dendrites are considered. The model was used to calculate the solidification of INCONEL 718. The results of calculation can be presented in terms of amount of Laves phase, grain distribution and final macro-segregation. It was demonstrated that grain movement during solidification has a significant effect on the final solidification microstructure.

10:10 AM Break

10:25 AM

Carbides and Grain Defect Formation in Directionally Solidified Nickel-Base Superalloys: *Sammy Tin*[1]; Tresa Pollock[1]; Wendy Murphy[2]; [1]Carnegie Mellon University, Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]General Electric Aircraft Engines, One Neumann Way, Cincinnati, OH 45215 USA

Demands for increased efficiency in turbine engines have led to the development of multi-component single crystal nickel-base superalloys. As levels of refractory alloying additions have gradually increased to improve high-temperature creep properties, grain defect formation during directional solidification has become an increasingly important problem. Recent solidification experiments have been conducted to investigate the relationship between alloy chemistry and the mechanism(s) which lead to grain defect formation. Carbon additions (up to 0.1wt. %) have a strong beneficial effect in reducing the number of grain defects. While it is known that carbon additions result in the precipitation of MC carbides, the interactions of these carbides with the defect generating mechanisms are not well understood. The role of carbides has been studied via segregation measurements coupled with DTA for a large set of alloys. The results of these experiments will be discussed with respect to the possible mechanisms by which carbon influences solidification.

10:50 AM

The Hot Deformation Modeling of As-Cast High-Strengthened Superalloys: *Zhengdong Long*[1]; Jingyun Zhuang[1]; Bo Deng[1]; Ping Lin[1]; *Zengyong Zhong*[1]; [1]Central Iron & Steel Research Institute, Dept. of Superalloys, 76 Xueyuan Nan Rd., Haidian District, Beijing 100081 PRC

The high-strengthened superalloys have harsh hot workability because of their high flow stress, low deformation ductility and narrow available deformation temperature range. So, the controlling of hot deformation parameters is very difficult and important. In the conventional manufacturing routes, a great amounts of trial-and-error steps must be taken to get suitable processing parameters, so, it is very worthy to develop a effective hot deformation modeling to predict the deformation behavior. In this paper, the hot deformation behaviors were examined, and the hot deformation modeling was built up based on the test results. First, the mathematical relationship between flow stress and deformation parameters, especially the peak flow stress vs hot deformation parameters were built up. Second, the mathematical relationship between deformation ductility and deformation parameters was also built up. Generally, these equations not only fit to the experimental results accurately but also reflect the physical phenomena occurring during the hot deformation.

11:10 AM

A New Technology of Superalloy Surface Metallurgy–Double Glow Plasma Surface Alloying: *Xishan Xie*[1]; Xu Zhang[1]; Jianxin Dong[1]; Zhong Xu[2]; [1]University of Science and Technology, Dept. of Matls. Sci. and Eng., 30 Xueyuan Lu, Beijing 100083 China; [2]Taiyuan University of Technology, Taiyuan 030024 China

The Double Glow Plasma Surface Alloying Technology (DGPSA) is an advanced new surface metallurgy technology. It was invented in China by Zhong Xu and patented in China, Canada, Japan, United Kingdom, United States and so on. The premium quality plasma alloying layers with special properties can be formed on the common material surfaces. In this paper the surface alloying layer similar to nickel-base superalloy Inconel625 is obtained on the surface of three kind steels (low carbon steel, pure iron and stainless steel 304) by using DGPSA. The chemical composition and microstructure of surface layers were analyzed by SEM, EDAX and XRD and show that the alloying layers consist of matrix and several precipitates which depends on the local chemical composition. The corrosion tests in 20%H2SO4 and 20%HCl solution show that the corrosion rates of surface alloying layer on pure iron are equal to that of nickel-base superalloy Inconel625 and one order of magnitude lower than that of AISI 304 stainless steel.

11:30 AM
Multizone Sonic Inspection of Ingot Metallurgy Superalloy Billet Material: *T. Reay*[1]; H. Waldal[1]; B. Zirbel[1]; D. Furrer[1]; [1]Ladish Company Inc., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

Quality level and flaw content are key factors for design requirements and component lifting issues for aircraft turbine engine manufacturers. Cast and wrought nickel-base superalloys are used for many demanding applications in turbine engines, such as the low and high pressure turbine disks. Inspection of these critical components is of significant importance. An effort has been undertaken to develop a new method of ultrasonic inspecting cast and wrought superalloy material to allow for a more complete assessment of input material in the billet stage prior to extensive value-added operations being performed. The utilization of multizone ultrasonic inspection has proven to be effective and beneficial for improving ultrasonic inspection of cast and wrought superalloy billet material.

Aluminum Reduction Technology: Process Control/Anodic Phenomena

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland New Zealand; Georges J. Kipouros, Dalhousie University, Department of Mining and Metallurgical Engineering, Halifax, NS B3J2X4 Canada

Tuesday AM — Room: Sewanee
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Bernd Rolofs, Hoogovens Aluminium Huttenwerk GmbH, Voerde D-46549 Germany

8:30 AM Invited
Overview of Process Control in Reduction Cells and Potlines: *Pierre Homsi*[1]; Jean-Michel Peyneau[1]; Michel Reverdy[1]; [1]Aluminium Pechiney, LRF-BP 114, Saint-Jean-de Maurienne 73303 France

Hardware and software developments have enabled the control of reduction cells to evolve dramatically over the last two decades. Process computers allow the control of cells as well as the technical management of modern potlines. The objectives of cell automatic process control are reviewed. They normally encompass alumina feeding, cell resistance regulation and bath chemistry and cell heat balance control. Routine operations on the cells are also accounted for. Tools are made available to assist potline supervisors in the optimization of operations, quick diagnosis of process changes, correction of abnormal and exceptional situations on individual cells or potlines as well as scheduling of work on the cells. New developments in cell and potline control provided by the ALPSYS system are presented.

9:00 AM
The Effects of Process Operations on Smelter Cell Top Heat Losses: *M. Derek Gadd*[1]; Barry J. Welch[1]; Tony D. Ackland[2]; [1]The University of Auckland, Chem. and Matls. Eng., Private Bag 92019, Auckland New Zealand; [2]New Zealand Aluminium Smelters Limited, Tech. Dev., Private Bag 90110, Invercargill, New Zealand

With modern smelting cells operating to a fine heat balance the impact of various operating and cell condition changes on the heat balance needs to be examined more closely. A study of the changes in top heat losses has been made by monitoring duct gas flow rate, duct gas and hood temperatures while regularly measuring ambient air and electrolyte temperature. With a continuous recording of cell process event messages, correlations between heat loss and the various routine operations and process changes have been possible. Particular attention has been placed on anode changing, metal tapping, alumina feeding and cell anode effects. The differences between individual cells have also been studied and it has been found these differences have a direct effect on the individual cell conditions.

9:25 AM
Anodic Phenomena–Observations of Anode Overvoltage and Gas Bubbling during Aluminium Electrolysis: *Xiangwen Wang*[1]; Alton T. Tabereaux[1]; [1]Smelter Technology Laboratory, Corp. Rsrch. and Dev., Reynolds Metals Co., 4276 Second St., Muscle Shoals, AL 35661-1625 USA

Anode overvoltage and the gas formation-release process (gas bubbling) are two important anodic phenomena during the electrolytic production of aluminum in Hall cells. Comprehensive electrolysis studies and physical modeling of these anodic phenomena including bubble formation and release observations, overvoltage, and their relations to anode size and configuration have been reported in the literature. But, these anode bubbling studies have been conducted mainly with two extreme anode scales; 1) extremely small (low amperage) anodes in laboratory cells 2) large industrial (high amperage) anodes in production cells. This paper reports on electrolysis studies conducted with an intermediate scale (15.2 cm diameter) anode in a laboratory cell to provide experimental data and explanations to bridge the results obtained from previous studies using the two extreme anode scale cases with bubble formation. Information on bubble size and the formation-release processes were obtained by measuring the bubble formation-release process frequency as well as the voltage magnitude from bubble growth, coalescence and release from the anode during a continuous 72 electrolysis test. The impact of gas bubbles on increasing the cell resistance, the anode overvoltage and gas bubble phenomena as a function of anode consumption (shape change) were observed and recorded. Variation in anode current density was investigated. The results provide an insight in understanding the relationship between bubbling phenomena and anode overvoltage variations during aluminium production.

9:50 AM
On the Anode Effect in Aluminium Electrolysis: *Jomar Thonstad*[1]; Torstein A. Utigard[2]; Helmut Vogt[3]; [1]Norwegian University of Science & Technology, Dept. of Matls. Tech. & Electrochem., Trondheim 7491 Norway; [2]University of Toronto, Dept. of Matls. Sci., 184 College St., Toronto, Ontario M5S3E4 Canada; [3]University of Applied Sciences, Berlin D-133353 Germany

Anode effects are detrimental in that they result in reduced energy efficiency and cause emissions of CF4 and C2F6. With prospects of future CO2, taxes, the emissions of these greenhouse gases may become costly. With a CO2 tax of 15 US$ per tonne, each anode effect minute per day per cell will increase the production cost by about 1.2%. Research work related to anode effects has been reviewed and analyzed. Although it is well known that anode effects occur when the alumina content becomes too low to maintain normal electrolysis, the mechanism of the initiation of the anode effect as well as the current distribution and gas behavior in industrial cells are still not fully understood. The paper concludes by analyzing various methods which may be used to decrease the frequency and duration of anode effects in prebake as well as Soderberg cells.

10:15 AM Break

10:25 AM
An Electroanalytical Study of Electrode Reactions on Carbon Anodes during Electrolytic Production of Aluminium: Hongmin Zhu[1]; *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. Matls. Sci. and Eng., 77 Massachusetts Ave., Rm. 8-109, Cambridge, MA 02139-4307 USA

The electrode reactions occurring on carbon anodes during the electrolytic production of aluminum are being studied in a laboratory-scale cell. Electroanalytical techniques such as voltammetry and electrochemical impedance spectroscopy are being used to determine the mechanisms associated with the electrochemical reactions at the anode. The work is motivated by the belief that an understanding of the elementary reactions occurring under a variety of process conditions will enable the development of strategies for reducing the intensity of perfluorocarbon (PFC) emission. The research is sponsored jointly by the Aluminum Association and the U.S. Environmental Protection Agency.

10:50 AM

Analysis of Excess AlF_3-Harmonization in Hydro Aluminium: *Lorentz Petter Lossius*[1]; Helge Hoie[1]; Hanne Hoel Pedersen[1]; Trygve Foosnaes[1]; [1]Hydro Aluminium Technology Centre Ardal, P.O. Box 303, Ovre-Ardal N-6881 Norway

When comparing process data from different aluminium reduction plants it is important to ensure that the results really are comparable. This presentation describes the harmonisation of the analysis of bath acidity in Hydro Aluminium Metal Products (HAMP). Each step from sampling to instrument calibration and analysis was scrutinised and the critical steps identified. The most complex part was establishing new bath standards, and some problems connected with the accuracy of the standards are addressed. The presentation also outlines how the individual steps in the analysis contribute to the standard deviation in the reported results and the effect of using different equipment in the participating laboratories. It is shown that, although the instrument precision can be less than O.O5wt%, other steps in the analysis contribute to a real standard deviation in the reported results of 0.20 to 0.45wt% depending on equipment and methods. At present round robins are conducted annually to ensure that the harmonisation is preserved. After each round robin the results are analysed and used as feedback to improve the next round.

11:15 AM

Distribution Aspects in Reduction Line Control Systems: *Carsten M. Ritter*[1]; Luiz F. R. Neves[1]; Leonel V. M. Ivo[1]; José H. S. Trigueiro[1]; [1]ATAN Automation Systems, Aluminium Div., Rua Pernambuco 353, Belo Horizonte, Minas Gerais 30130-150 Brazil

This paper analyzes the different degrees of distribution adopted by aluminium reduction line control systems. The concepts, approach, and results to implement centralized, semi-distributed and totally distributed automation topologies are described. The pros and cons for some architectures are discussed. A framework for topologies comparison is then presented, providing a valuable decision tool when implementing, replacing, expanding or revamping the reduction line control system.

Automotive Alloys 2000: Applied

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh K. Das, University of Kentucky College of Engineering, Center for Aluminum Technology, Lexington, KY 40506-0043 USA

Tuesday AM — Room: Knoxville A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Subodh K. Das, University of Kentucky, College of Eng., Lexington, KY 40232 USA; Andy M. Sherman, Ford Motor Company, Dearborn, MI 48121-2053 USA

8:30 AM

Aluminum Tailor-Welded Blanks for Automotive Applications: *P. A. Friedman*[1]; G. T. Kridli[2]; [1]Ford Research Laboratory, Manufact. Sys. Dept., P.O. Box 2053, MD 3135/SRL, Dearborn, MI 48121-2053 USA; [2]University of Michigan-Dearborn, Dept. of Indust. and Manufact. Sys. Eng., 2300 Eng. Complex, Dearborn, MI 48128 USA

The push to manufacture lighter-weight vehicles has forced the auto industry to look to alternative materials than steel for vehicle body structures. Aluminum is one such material that can greatly decrease the weight of vehicle body structures and is also consistent with existing manufacturing processes. As in steel structures, cost and weight can be saved in aluminum structures with the use of tailored blanks. These blanks consist of two or more sheets of dissimilar thicknesses and/or properties joined together through some type of welding process. This enables the design engineer to “tailor” the blank to meet the exact needs of a specific part. Cost savings can be gained by the elimination of reinforcement parts and the stamping dies used to manufacture them. Weight savings can be attained based on the fact that one thicker piece is more efficient than a welded structure and therefore can allow for down-gauging of parts. While TWBs offer both potential weight and cost benefits, the continuous weld-line and thickness differential in TWBs can often result in difficulty in stamping. This problem is more severe in aluminum because of its limited formability as compared with typical drawing-quality steels. Additionally, welding of steel TWBs tends to increase the strength of the weld material which helps prevent failure in the weld during forming. Aluminum TWBs do not experience this increase in strength and therefore have a greater tendency to fail in the weld. In this study, several aspects of TWBs manufactured from 6111-T4, 5754-O and 5182-O aluminum alloys were analyzed and compared with those of a more conventional steel TWB. The effect of gauge mismatch on the formability of these blanks is discussed as well as the overall potential of these blanks for automotive applications.

8:50 AM

Surface Roughness Development in 6022 and 6016 Aluminum Alloy Sheet and its Relation to Microstructure Analyzed with the Disorientation Correlation Function: *Anthony D. Rollett*[1]; Paul S. Lee[1]; Brent L. Adams[2]; Henry R. Piehler[1]; Hasso Weiland[3]; [1]Carnegie Mellon University, Dept. Matls. Sci. & Eng., 3327 Wean Hall, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]Brigham Young University, Dept. Mech. Eng., Provo, UT USA; [3]Alcoa Technical Center, PA 15069 USA

An important aspect of sheet forming is the development of surface roughness as plastic strain accumulates. Such roughness may decrease the usefulness of the formed part or even limit the formability. Taking the crystallographic nature of slip as a basis for investigation, orientation imaging microscopy has been used to characterize sections through samples of 6022 and 6016, both in the T4 condition, deformed in plane strain tension. In order to quantify strain heterogeneities and correlations in texture that are not readily apparent in conventional images, the disorientation correlation function (DCF) has been used. The DCF calculates the average misorientation as a function of distance and direction. The results indicate the presence of long-range orientation correlations extending over more than five grain diameters. Substantial changes in the DCF are observed as a function of strain level. At large strains, for example, grains lying along the extension direction are more highly correlated than in any other direction. When bands observed in the DCF maps are compared with surface ridging, similar spacings are found. Also, a comparison of sheets with different surface roughening susceptibilities suggested that stronger texture correlations are observed in materials with greater roughening tendencies. These observations of long range orientation correlation may have implications for strain localization in the deformation of sheets. In samples of 6016 treated to develop the ridging phenomenon, the DCF reveals a complex banded structure. An interesting result of the sheet tensile tests is that the fracture angle follows the Hill theory as the angle from the rolling direction increases except that, when the tensile axis is within a few degrees of the transverse direction, the fracture path becomes parallel to the ridges, i.e. the rolling direction. This work was primarily supported by the Office of Energy Research, US Department of Energy under grant number DE-FG02-96ER45601.

9:10 AM

Precipitation Behavior in Grain Boundary Regions of Modified 5083 Aluminum Alloys: *M. C. Carroll*[1]; P. I. Gouma[1]; M. J. Mills[1]; G. S. Daehn[1]; B. R. Dunbar[2]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43201 USA; [2]Century Aluminum Corporation, Ravenswood Operations, P.O. Box 98, Ravenswood, WV 26164 USA

The susceptibility of 5000 series alloys to stress corrosion cracking (SCC) has been largely attributed to the precipitation of Mg (beta phase) along grain boundaries. The alloys of this series that contain levels of Mg higher than about 3.5 wt% have been specifically singled out as the most susceptible stress corrosion cracking as a result of Mg enrichment and subsequent Mg-based phase formation in grain boundary regions. The precipitation behavior of various alloys with near-5083 composition will be evaluated. Emphasis will be placed on additions of elements that produce a precipitate microstructure that may be less susceptible to SCC as a result of limiting beta phase (Al_8Mg_5)

formation. Particular attention will be paid to analyzing local precipitation changes in the regions adjacent to grain and subgrain boundaries. Information will be presented based on transmission electron microscopy (TEM) images as well as fine-probe energy dispersive spectroscopy (EDS) composition profiles.

9:30 AM

Finite Element Modeling of Creep Relaxation in Bolted Joints of Magnesium Castings: *Ken I. Johnson*[1]; Russell H. Jones[2]; Stan G. Pitman[3]; Eric A. Nyberg[3]; [1]Pacific Northwest National Laboratory, Energy Div./Appl. Mech. Grp., P.O. Box 999, Richland, WA 99352 USA; [2]Pacific Northwest National Laboratory, Matls. Resources/Structural Matls. Dev., P.O. Box 999, Richland, WA 99352 USA; [3]Pacific Northwest National Laboratory, Matls. Resources/Matls. Processing, P.O. Box 999, Richland, WA 99352 USA

Magnesium die-castings are of interest for automotive transmission housings because of the potential to save up to 30% of the weight of traditional aluminum castings. However, a major drawback is the poor creep resistance of magnesium casting alloys at elevated-temperatures. This can result in loss of bolt load retention (BLR) and subsequent leakage of bolted flange joints. There is a current need to develop modeling tools that correlate fundamental creep data with the BLR of actual joint designs. This stems from the ease of creep testing and the relative abundance of creep data compared with BLR tests. Both constant load tensile creep tests and simplified BLR tests are currently performed. This paper describes material testing and finite element modeling that was performed to demonstrate the correlation between these different forms of creep data for ZAC 8506. The goal of this research is to develop an analytical tool with which to optimize bolted joints in magnesium castings. Material constitutive models based on constant load creep tests where input to the MARC finite element code to simulate actual bolt load retention tests. The models include the effects of bolt preload, surface contact, creep relaxation, thermal expansion, and yielding due to thermal softening. Results are presented that compare the model predictions with the results of actual bolt load retention tests. The models are also used to show the relative importance of initial preload, creep, and yielding on the retained bolt load.

9:50 AM

High Temperature-Creep Resistant Magnesium Alloys: Advances in the Use of Thixomolding for Automotive Components: *Eric Arthur Nyberg*[1]; Russell H. Jones[1]; Stan G. Pitman[1]; Daniel J. Edwards[1]; Robert D. Carnahan[2]; Raymond F. Decker[2]; [1]Pacific Northwest National Laboratory, Matls. Resources, Battelle Blvd., P.O. Box 999, Richland, WA 99352 USA; [2]Thixomat Inc., 620 Technology Dr., Ann Arbor, MI 48108 USA

High temperature ZAC alloys (Mg-Zn-Al-Ca) have attracted interest due to their reasonable combination of cost, forming/processing characteristics and mechanical properties. In particular, the improved high temperature creep resistance of these alloys makes them candidates for automotive applications where the creep strength of magnesium is limited because the operating temperatures exceed approximately 125°C. Most research on the ZAC alloys has focused on comparing the die cast properties of the ZAC alloys to other alloys currently used in die cast production (AZ91D, AM50A, AM60B, AS21 and AS41). This project is part of the Northwest Alliance for Transportation Technologies (NATT) program which is funded through DOE's Partnership for New Generaton Vehicles (PNGV) program. The aim of the NATT program is to address specific transportation goals through cooperative R&D with suppliers. In this study, Pacific Northwest National Laboratory and Thixomat Inc. have partnered to investigate the high temperature properties of ZAC8506 (8%Zn-5%Al-0.06%Ca) comparing standard die casting and the semi-solid forming process referred to as Thixomolding. The objective of the work was to better understand the influence of lower part forming temperatures, from the semi-solid, laminar flow process (Thixomolding), on void/pore fraction, tensile strength and high temperature creep behavior as compared to die casting. As has been shown for other alloy systems, the fraction of pores is reduced and the mechanical properties are improved when formed by Thixomolding. This work is continuing to evaluate alloys produced by Thixomolding that are traditionally not die cast, such as magnesium metal matrix composites and the ZK alloys.

10:10 AM Break

10:20 AM

The Influence of Reversed Bending on the Formability of Aluminum Sheet: *Karole J. Lian*[1]; Armand J. Beaudoin[1]; Peter A. Friedman[2]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. and Industrial Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Ford Research Laboratory, Dept. of Manufacturing Sys., P.O. Box 2053, MD 3135/SRL, Dearborn, MI 48121-2053 USA

The forming limit diagram (FLD) provides a useful engineering tool for the development and analysis of stamping operations. It is predicated on the notion that the deformation history will follow a proportional strain path. To extend the utility of the FLD, recent efforts have studied the effect of pre-straining the sheet in a specific mode of deformation prior to FLD determination. In sheet forming practice there is often significant deformation imparted on the sheet before contact with the forming punch. Two specific examples of this type of deformation are reversed bending occurring during tension-leveling operations and in material traversing a drawbead. The objective of this current research is to study the effect of through-thickness straining on the subsequent formability of aluminum sheet. A test apparatus has been designed to impart reversed bending to sheet of width sufficient for extended limiting dome height tests. Experiments were performed on both AA5754 and AA6111 sheets after bending, and the results were compared to the original sheet stock to detect changes in formability. The magnitude of through-thickness plastic pre-strain is assessed through X-ray measurement of crystallographic texture and residual stress.

10:40 AM

Formability of Aluminum Extrusions during Hydroforming Applications: *Mark Tower Smith*[1]; Richard William Davies[1]; Mohammad A. Khaleel[1]; [1]Battelle PNNL, Matls. Processing, 902 Battelle Blvd., P.O. Box 999 MSIN:P8-35, Richland, WA 99352 USA

Tubular hydroforming of automotive components is receiving significant attention as a manufacturing method capable of reducing part count, component weight, and overall manufacturing costs. Hydroforming is extremely attractive as a method to produce complex shaped components from tradition tube and extrusion materials. U.S. automotive companies and suppliers are currently manufacturing many steel tubular hydroformed components. However introducing aluminum alloys to hydroforming applications is consistent with the current automotive company and government initiatives to reduce the overall weight and emissions of automobiles. The current work evaluates and presents the formability of aluminum alloys during hydroforming applications, including studies conducted to systematically vary the application of axial end feed and internal pressure during the hydroforming experiments. The current work also experimentally investigates the anisotropic yield locus of the extruded aluminum tubing, and theoretically evaluates the effects the manufacturing inherent anisotropy has on the formability of aluminum alloys extrusions.

11:00 AM

Thermo-Mechanical Fatigue of a Cast 319 Aluminum: *John Victor Lasecki*[1]; Huseyin Sehitoglu[2]; John E. Allison[1]; [1]Ford Motor Company, Matl. Sci. Dept., Scientific Rsch. Lab., MD 3182 SRL, Dearborn, MI 48121-2053 USA; [2]University of Illinois, Mech. and Industrial Eng., 144 Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

The majority of automotive engine cylinder heads are cast aluminum. The trend is for aluminum cylinder heads to be used in increasingly more demanding operational environments which necessitates a knowledge of the material behavior under cyclic thermo-mechanical loading conditions. The purpose of this study is to determine the extent to which existing constitutive and life models can be extended to loading situations in which the strains are predominantly compressive. TMF experiments were carried out under mechanical strain control on solid cylindrical specimens of 319 Al. The TMF cycle was selected so as to simulate the critical conditions that exist in the surface volume of a cylinder head during a start-up and shut down operation. Out of phase loading dominates the cylinder head duty cycle, therefore experiments were conducted with the maximum temperature coinciding with the maximum compressive strain. Tests were performed in air with the temperature cycled between 50 and 250°C. In this study the role of selected mean stresses and mechanical strain amplitudes were used to characterize the TMF behavior of cast aluminum. Results will be compared with recently developed constitutive and durability models for cast 319 Al. Fractography of failed samples was conducted to understand the crack initiation mechanism.

11:20 AM
Hot-Tearing Analysis of a Solidifying Die Cast Part: *Yimin Ruan*[1]; Jamal Righi[1]; [1]Alcoa Inc., Shaped Casting Platform, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

Aluminum alloys are susceptible to cracking during solidification (hot-tearing). The susceptibility largely depends on the alloy composition. Process conditions and the geometry of a casting also have significant influence on hot-tearing. Hot-tearing occurs in all castings, such as wrought ingot, die cast part, foundry cast part, etc. This paper presents a hot-tearing analysis of a solidifying die-cast part for an alloy which has a higher strength and more susceptible for hot-tearing. Due to the characteristics of the alloy, higher inelastic strains and stresses develop in the cast part and cause the cast material to crack. In this paper, the thermomechanical behavior of the alloy during solidification after the molten metal is injected into the die cavity is studied and the conditions to prevent cracking at the casting surface are determined. A hot-tearing criterion is also developed in this paper in order to reliably predict and prevent casting cracks during solidification.

11:40 AM
Casting Simulations for Semi-Solid Metal Forming Process: Yie Zhao[1]; *Jamal Righi*[1]; [1]Alcoa Inc., Shaped Casting Platform, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

Forming alloys in the semi-solid state is a relatively new but potentially high rewarding method for producing near net-shaped castings. The production of high quality components requires detailed understanding of die filling and solidification pattern. To achieve this, numerical simulations have been adopted as a process design, development and optimization tool. This paper presents a filling analysis of a hat-shaped die, which is a demonstration part chosen by an Alcoa development program. Two software packages Flow-3D and APECS, using single phase simulation approach which employ governing equations for a Newtonian fluid and introduce rheological behavior for semi-solid slurries thought viscosity models, were used to perform the casting simulations. Simulation results showed the hat-shaped die could be filled without any difficulties from A356 slurry. The initial casting trials confirmed the simulation predictions. In addition, the effects of thixotropic behavior of material and process conditions on filling patterns were studied thoroughly.

Cast Shop Technology: Casting and Solidification

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Paul Crepeau, General Motors Corporation, GM Powertrain Group, Pontiac, MI 48340-2920 USA; James N. O'Donnell, Commonwealth Aluminum Corporation, Department of Engineering, Louisville, KY 40202-2823 USA

Tuesday AM — Room: Mississippi
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Martin J. Ekenes, Hycast, Otis Orchards, WA 99027-0603 USA

8:30 AM Introductory Remarks

8:35 AM
Automation & Data Acquisition: Tools for Safer & Easier Casting: *Patrick Pouly*[1]; Etienne Caloz[1]; [1]Alusuisse Technology & Management Limited, Tech. Ctr., Chippis 3965 Switzerland

Automation has proven to be a valuable tool since its introduction in the casthouse in the 1980's. One of the main arguments justifying its introduction is the drastic reduction in safety hazard, as most parameters can be kept under tight control. After a rapid presentation of the structure of a modern automation, this paper will show the potential lying within a powerful data acquisition system. Such a system can be linked to the automation and used for analyzing casting conditions. A series of practical examples will be given to illustrate how a data acquisition system can be used to localize the phenomenon that lead to a premature end of a cast.

9:00 AM
Chemical, Physical & Mechanical Properties of Today's Casting Tips for Aluminum Sheet Casting: *Jason M. Canon*[1]; [1]Thermatex Corporation, Rsch. & Dev., P.O. Box 125, Newton Falls, OH 44444 USA

A program has been instituted to measure pertinent properties of asbestos free casting tips currently used in the twin roll casting process and are now available in the global market. Casting tips are an integral part of the casting process and can directly affect the quality of the aluminum and impact the continuous casting operation. Measured properties included typical chemistry, shot content, thermal conductivity, density, modulus of rupture, compressive strength, flexural strength, shrinkage, water absorption and the measurement of organic/inorganic binders. Properties were also measured throughout the tip to determine the consistency of the product and it was determined variability exists within the casting tip. A method of improving the inconsistencies were evaluated and documented. Efforts were made to correlate calculated laboratory test results to product performance in the field.

9:25 AM
A Potential Casting Pit Coating Material: *George J. Binczewski*[1]; [1]S.C. Systems, P.O. Box 6154, Moraga, CA 94570 USA

Finding an acceptable replacement for the familiar, but environmentally unfriendly Tarset coating has been the object of an aluminum industry funded, and ongoing research project for several years. The need to enhance the safety performance of DC casting pits involving potential contact surfaces of molten aluminum and water is continuous. There are many favorable attributes associated with utilizing rubber sheeting, which is organic, as the coating material. These involve low costs, multiple availability, ease of application, exclusion of cure times, simplicity of repair, long service life, and vibration dampening characteristics. Experiences and performance evaluations of rubber sheeting involving situations of contact with molten aluminum and water are described and reported.

9:50 AM
Study of Hot-Tearing in Solidification of Aluminum Alloys Via an Acoustic Emission Technique: *Xiaojin Li*[1]; *Celil A. Aliravci*[1]; Mihriban O. Pekguleryuz[1]; Michel Bouchard[1]; [1]University of Quebec in Chicoutimi, Dept. Applied Sci., Alcan-UQAC Chair in Solidification and Metallu. of Al, 555 Univ. St., Chicoutimi, Quebec G7H 2B1 Canada

The hot-tearing tendency of AA1050 aluminum alloy solidifying in a ring-shaped test-casting mold was investigated in real-time and in situ via an acoustic emission (AE) and a temperature-data acquisition and analysis (TDA) techniques. In lab-scale experiments, AE signals were acquired simultaneously with temperature monitoring by the use of an inserted steel wave guide and a K-type thermocouple placed in the final freezing zone. These measurements have provided a time definition for AE signal characteristics and a definite time-temperature reference-frame for solidification and defect formation events. Hence, a technique of AE signal analysis combined with computer-aided cooling-curve analysis (CA-CCA) was developed. It was shown that the AE technique is effective in detecting hot-tearing occurrences, and can be further developed for mechanistic studies of hot-tearing, and possibly as a quality control tool for the casting industry. Grain refinement was carried out in AA1050 Al alloy to verify the effect of grain refinement on hot-tearing tendency. The experimental results showed that grain refinement can considerably reduce the hot-tearing tendency in the AA1050 alloy. Fracture surface analysis was conducted for all samples under a scanning electron microscope. Typical exposed hot tear surfaces with free dendritic structure were observed and analyzed.

10:15 Break

10:20 AM

Laboratory Study of Cast Surface Structure Evolution: I. Mold Contact Stage: *Douglas A. Weirauch*[1]; Lawrence J. Martonik[1]; Alvaro Giron[1]; Donald P. Ziegler[1]; Men Glenn Chu[1]; [1]Alcoa, Alcoa Tech. Ctr., 100 Tech. Dr., Alcoa Center, PA 15069-0001 USA

A small-scale, aluminum caster is described which permits the careful control of many of the important continuous casting process parameters which affect the early stages of solidification. The immersion caster is applied to high-purity aluminum as a first step in quantifying the effect of key process variables on cast surface features and subsurface microstructure. The observed characteristics of surface features are interpreted on the basis of heat transfer and shell growth kinetics. The effects of casting speed, melt superheat, mold surface roughness, and aluminum alloy solidification range are discussed.

10:45 AM

Laboratory Study of Cast Surface Structure Evolution II. Macro Air Gap Stage: *Makoto Morishita*[1]; Kiminori Nakayama[2]; Kenji Tokuda[3]; Katsuyuki Yoshikawa[1]; [1]Kobe Steel Limited, Process Tech. Rsch. Lab., 5-5, Takatsukadai 1-Chome, Nishi-ku, Kobe, Hyogo 651-2271 Japan; [2]Kobe Steel Limited, Mech. Eng. Rsch. Lab., 5-5, Takatsukadai 1-Chome, Nishi-ku, Kobe, Hyogo 651-2271 Japan; [3]Kobe Steel Limited, Aluminum Rsch. Dept., Moka Plant, 15 Kinugaoka, Moka, Tochigi 321-4367 Japan

At macro air gap stage of aluminum casting process, an exudation is occurred by surface remelting because of high thermal resistance of the air gap between solidification shell and mold. This exudation causes some problems such as segregation, uneven structure and so forth. The air gap tester, which is possible to create the air gap and the exudation on the surface experimentally, is applied to molten aluminum alloy in order to clarify the effect of casting variables on the exudation during solidification. The effects of grain size, melt superheat, mold surface roughness and aluminum alloy solidification range are discussed.

11:10 AM

Computer Simulation of Metal Feeding System Used in Twin Roll Casting: *Kemal Sarioglu*[1]; *Murat Dundar*[1]; *Gungor Yildizbayrak*[1]; [1]ASSAN Aluminum Plant, Tech. Coordination Div., Tuzla, Istanbul 81700 Turkey

Twin roll casting has been accepted worldwide as a cost-effective method of producing wide variety of Al products. This process converts molten aluminium alloys into coiled sheet. The uniform distribution of metal to the water cooled roll is accomplished with a ceramic fiber tip. Internal buffling configuration affects this uniform distribution causing defect free sheet production. In this study, the placement of the buffles in the tip having different widths has been investigated by using computer simulation. Uniform flow distribution at the exit of the tip was attained with proper arrangement of the buffles within the tip body. Practical applications verified that unperturbed flow distribution over the whole width of the tip prevented void formation at certain location of the sheet and insufficient crown formation. The goal of this research is also to find sufficient tip geometry that enables continuous metal feeding for thinner gauges.

11:35 AM

The Effect of Casting Parameters on Twin Roll Cast Strip Microstructure: A. Soner Akkurt[1]; *Murat Dundar*[1]; Seda Ertan[1]; Erol Ozden[1]; Kemal Sarioglu[1]; Gungor Yildizbayrak[2]; Shaun Hamer[3]; Chris Romanowski[3]; [1]ASSAN Aluminum Works, E-5 Karayolu 32.Km, Tuzla, Istanbul 81700 Turkey; [2]Kibar Holding A.S., E-5 Karayolu 32.Km, Tuzlal, Istanbul 81700 Turkey; [3]FATA Hunter Inc., 6147 River Crest Dr., Riverside, CA 92507 USA

Twin roll casting is used throughout the aluminum industry to produce reroll for a variety of fin and foil products. The recent trend has been to reduce the gauge at which these casters operate to <3mm. Difficulties in industrially processing this thin gauge cast strip have been reported; in particular, maintaining satisfactory final fin and foil product ductility. Assan Aluminum, in cooperation with FATA Hunter, recently completed an extensive series of casting trials to characterize the effects of casting parameters, including casting gauge, speed and tip setback on the microstructure of various fin and foil alloys. The samples were examined using optical and electron microscopy, in combination with mechanical properties, micro-hardness and resistivity measurements. This analysis was used to optimize the casting, rolling and annealing practice for each alloy. This paper presents the initial results from this study and describes general trends correlating casting parameters to microstructure.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Cyclic Deformation and Mechanism (II)

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Zhirui Wang, University of Toronto, Department of Metals and Materials Science, Toronto, Ontario Canada; Charles McMahon, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Pedro D. Peralta, Arizona State University, Department of Mechanical and Aerospace Engineering, Tempe, AZ 85287-6106 USA; J. K. Shang, University of Illinois, Department of Materials Science and Engineering, Urbana, IL 61801 USA

Tuesday AM — Room: Canal A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: J. D. Embury, McMaster University, Matls. Sci. & Eng., Hamilton, Ontario Canada; P. Peralta, Arizona State University, Dept. of Mech. & Aerospace Eng., Tempe, AZ 85287-6106 USA

8:30 AM

Constriction Energy in the Presence of an Interstitial Field: *Huseyin Sehitoglu*[1]; Scott Andrews[1]; Ibrahim Karaman[1]; [1]University of Illinois, Dept. of Mech. and Industrial Eng., Urbana, IL 61801 USA

It has been widely known that both solute concentration, ie. frictional effects, and stacking fault energy influence the degree of cross-slip and slip planarity in fcc alloys (for details see Hong and Laird, 1990). Cross-slip is preceded by constriction of two partial dislocations. A model is proposed for the energy required to form a constriction from two parallel partial dislocations as a function of stacking fault energy, interstitial concentration and atomic size misfit. In the analysis, the local stress fields are calculated due to either interstitial or substitutional solute concentrations around the partial dislocations. The cross-slip is curtailed due to interaction of interstitials with the edge components of the partials. The atomic size misfit influences the local interstitial concentration, local stresses which in turn decide the energy to form the constriction. The shape of partials and the energy to form the constriction was established for stacking fault energies in the range 10-100 mJ/m^2, misfit strains in the range of 0.1 to 0.5, and nominal interstitial concentration varying from 0 to 10 atomic %. In extreme cases, the constriction energy has been found to increase by four-fold compared to the interstitial-free case. The results are readily applicable to iron alloys with carbon and nitrogen interstitials, and copper alloys with manganese, aluminum or zinc as substitutional solute atoms. The results converge to the well known solution of Stroh in the limit of zero interstitial concentration.

8:55 AM

Stacking Faults on (001) in $MoSi_2$-WSi_2 Solid-Solutions with the $C11_b$ Structure and Their Influence on the Fracture Behavior: *H. Inui*[1]; K. Ito[1]; T. Nakamoto[1]; M. Yamaguchi[1]; [1]Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Stacking faults on (001) in $MoSi_2$-WSi_2 solid-solutions with the $C11_b$ structure have been characterized by transmission electron microscopy (TEM), using their single crystals grown by the floating-zone method. Although binary WSi_2 contains a high density of stacking faults, only a few faults are observed in binary $MoSi_2$. The density of these stacking faults increases with the increase in the WSi_2 content in the solid-solutions with the drastic increase occurring only near the WSi_2- end side. These (001) faults are characterized to be of the Frank-type in which two successive (001) Si layers are removed from the lattice, giving rise to a displacement vector parallel to [001]. When the displacement vector of faults is expressed in the form of R=1/n[001], however, their n values are slightly deviated from the exact value of 3, because of dilatation of the lattice in the direction perpendicular to the fault, which is caused by the repulsive interaction between Mo (W) layers above and below the fault. Matching of experimental high-resolution TEM images with calculated ones indicates Π values to be 3.12 + 0.10 and 3.34 + 0.10 for $MoSi_2$ and WSi_2 respectively. Fracture toughness has also been measured as a function of WSi_2 content with notched specimens by three point bending. When a crack propagates parallel to (001) fault planes, the value of fracture toughness is higher for $MoSi_2$ than for WSi_2 when a crack propagates parallel to (110) so that a crack is arrested by (001) faults if they exist. This may indicate that (001) faults are beneficial for increasing the fracture toughness of $MoSi_2$-WSi_2 solid-solutions.

9:20 AM

Cyclic Deformation and Strain Burst Behavior of Cu-7 at%Al and Cu-16at%Al Single Crystals with Different Orientations: *Zhong Guang Wang*[1]; Xi Mao Wu[1]; Guang Yi Li[1]; [1]Chinese Academy of Science, State Key Lab. for Fatigue and Fracture of Matls., Shenyang 110015 China

Compared to the cyclic deformation of wavy slip materials, our knowledge about the cyclic deformation of planar slip materials has been much less. The available reports up to date are not consistent and sometimes even controversial with each other. The materials used for these studies were usually copper alloys containing alloying elements such as Al or Zn in order to reduce the value of stacking fault energy (SFE). Abel et al. [1] studied the effect of aluminum content (2-16 at%) on the fatigue behavior of Cu-Al single crystals oriented for single slip. They found that the cyclic hardening behavior of the alloys with Al content less than 4 at% was similar to that of pure Cu single crystals showing clear saturation behavior. Alloys containing more than 11 at% Al did not show saturation behavior before failure. By using multistep tests, Yan et al. [2] reported the occurrence of saturation behavior and a regular cyclic stress-strain curve with a plateau in Cu-16at% Al single crystal oriented for single slip. However, Hong and Laird [3-5] investigated the cyclic deformation of the same alloy again under total strain control mode and this time for the entire range of strain amplitudes investigated stress saturation in cyclic hardening curves was not detected until the final failure.

9:45 AM

Internal Stresses in Heavily Deformed Materials and Their Influence on Mechanical Response: *Ke Han*[1]; J. D. Embury[2]; [1]Florida State University, Nat. High Mag. Field Lab., Tallahassee, FL 32310 USA; [2]McMaster University, Matls. Sci. & Eng., Hamilton, Canada

The deformation of two phase materials represents one very valuable method of producing extremely high strength materials. One aspect of these materials which is poorly understood is the pattern of residual stresses produced by codeformation. The current work examines a variety of methods of estimating these stresses and the influence of these stresses on both monotonic and cyclic deformation.

10:10 AM Break

10:35 AM

Influence of Thermo-Mechanical Treatment and Loading Frequency on the High Cycle Fatigue Properties of AlZnMgCul, 5 Aluminum Alloy: *H. Mayer*[1]; M. Papakyriacou[1]; R. Pippan[1]; S. Stanzl-Tschegg[1]; [1]University of Agricultural Sciences, Instit. of Meteorology & Physics, Vienna, Austria

Fatigue properties of age hardened AlZnMgCul, 5 aluminum alloy have been investigated in the high cycle/low crack growth rate regime. AlZnMgCul, 5 was tested in three different thermo-mechanical conditions: artificially aged (T6), cold worked and artificially aged to increase static strength (T66), and cold worked and artificially aged to increase ductility (T64). To observe an eventual influence of cycling frequency on the fatigue behaviour, S-N experiments were performed using conventional and ultrasonic fatigue testing equipment. The best fatigue properties in the S-N experiments were found for T6 hardening condition, whereas T66 as well as T64 thermo-mechanical treatment deteriorated the fatigue properties, especially at very high numbers of cycles. Similarly, the best fatigue crack growth properties were found for T6 condition. A threshold cyclic stress intensity to propagate a fatigue crack could be detected, whereas no endurance limit could be found in S-N experiments.

11:00 AM

Interfacial Fatigue Fracture in Copper-Sapphire Bicrystals: *P. Peralta*[1]; U. Ramamurty[2]; S. Suresh[3]; G. H. Campbell[4]; W. E. King[4]; T. E. Mitchell[5]; [1]Arizona State University, Dept. of Mech. and Aerospace Eng., P.O. Box 876106, Tempe, AZ 85287-6106 USA; [2]Nanyang Technological University, School of Mech. and Prod. Eng., 639798 Singapore; [3]Massachusetts Institute of Technology, Dept. of Matls. Sci. and Eng., Cambridge, MA 02139-4307 USA; [4]Lawrence Livermore National Laboratory, MSL-356, Livermore, CA 94550 USA; [5]Los Alamos National Laboratory, MST-CMS, MSK765, Los Alamos, NM 87545 USA

Interfacial fatigue cracks were propagated in copper/sapphire bicrystals with the boundary perpendicular to the load axis and $(110)_{Cu}(1010)_{Al2O3}$/[001] $Cu[0001]_{Al2O3}$ to study the effect of crystallography and slip geometry in the fracture process. Compact tension (CT) specimens with two different crystallographic directions for crack growth were loaded in tension-tension with ΔK applied ≈2MPa.m$^{1/2}$. Cracks grew preferentially when the growth direction was $<110>_{Cu}$ and less favorably when the growth direction was along $<001>_{Cu}$, which also resulted in an inclined crack front. Striations, which did not coincide macroscopically with traces of available slip planes, could be observed on the copper fracture surface; however, large areas were also relatively free of features. Elastic analysis of the anistropic near-tip stress fields for the interfacial crack revealed that the preferential crack growth direction had the highest energy release rate, whereas the second crack direction had the minimum mode II mix. A model to account for the non-crystallographic striations observed is proposed.

11:25 AM

Cyclic Deformation Mechanisms in NiTi Alloys: *Huseyin Sehitoglu*[1]; Ken Gall[2]; I. Karaman[1]; R. Anderson[1]; Y. I. Chumlyakov[3]; [1]University of Illinois, Dept. of Mech. and Industrial Eng., Urbana, IL 61801 USA; [2]University of Colorado, Dept. of Mech. Eng., Boulder, CO 80309 USA; [3]Siberian Physical and Technical Institute, Physics of Plasticity and Strength of Matls. Lab., Tomsk 634050 Russia

Single crystal NiTi shape memory alloys exhibit considerable cyclic hardening under zero-tension and zero-compression strain control experiments. The stress amplitude under strain control can increase by as much as factor of 1.2 in tension and a factor of 2 in compression. The increase in stress amplitude is primarily from the increasing strain hardening modulus. The deformation is controlled by transformation from a parent to martensitic phase, and detwinning of the martensite. Both the transformation strains and detwinning strains are orientation and stress direction dependent. In the past, the cyclic deformation characteristics of NiTi have been primarily studied in polycrystalline alloys. In this presentation, the focus will be on single crystals. The strong advantage of single crystals is that the characteristics of the deformation mechanisms (martensite plate variants and slip systems) are a known function of the loading axis of the crystal. In addition, these materials are almost always used in aged condition to produce pseudoelastic stress-strain response. Consequently, the precipitate coherency and precipitate size influences the deformation resistance. The presentation will highlight some of the micro-mechanical modeling to predict the role of precipitates on the deformation resistance and explain the strong dependence of orientation and stress direction on the fatigue response.

Fundamentals of Lead and Zinc Extraction and Recycling: Session I

Sponsored by: Extraction & Processing Division, Lead, Zinc, and Tin Committee

Program Organizers: Markus Reuter, Delft University of Technology, Applied Earth Sciences, Delft 2628 RX The Netherlands; Eric Allain, University of Missouri, Rolla, MO 65409-1460 USA

Tuesday AM — Room: Bayou D
March 14, 2000 — Location: Opryland Convention Center

Session Chair: M. A. Reuter, Delft University of Technology, Appl. Earth Sci., Delft, RX 2628 The Netherlands

8:30 AM

Modeling of the Lead Blast Furnace: *Bart Blanpain*[1]; P. Verguts[1]; P. Wollants[1]; S. Brouwer[2]; Frank De Buyck[3]; [1]KU Leuven, Dept. of Metallu. and Matls. Eng., Leuven Belgium; [2]Union Minière, B.U. Precious Metals, A. Greinerstraat 14, Hoboken B-2660 Belgium; [3]Union Minière, Research, Kasteelstraat 7, Olen B-2250 Belgium

Union Minière, one of the major non-ferrous metal producers in the world with several industrial sites in Belgium, recently changed its copper-lead metallurgy flow sheet substantially. Before the change, lead-copper ore sinter and secondary materials were fed into a blast furnace. Now only secondary raw materials are smelted in the blast furnace. Since the porosity of this feed is very low in comparison with the former sintered ores, the gas-solid reactions occur very slowly. Because the process of the blast furnace has changed considerably, a mathematical model of the furnace is being constructed as a tool to study the phenomena occurring in the furnace. The model uses general Computational Fluid Dynamics together with models for porous zones, combustion and so on. The model will be used to study the influence of operating parameters on blast furnace phenomena in order to optimize blast furnace performance. An outline of the model, together with first results will be discussed.

8:50 AM

Production of an Ultra-Pure Fraction of ZnO by the Recycling of EAF Dust: Fernando García-Carcedo[1]; Nancy Ayala[1]; N. Goicoechea[2]; A. Hernández[1]; Enrique Ruiz-Ayúcar[1]; E. García-Ventosa[1]; I. Dañobeitia[2]; N. Cornejo[1]; *Eric G. Allain*[3]; [1]CENIM, Centro Nacional de Investigaciones Metalúrgicas, C/. Gregorio del Amo 8, Madrid 28040 Spain; [2]Compañía Industrial ASER, S.A. Carretera Bilbao-Plencia, Asúa-Erandio, Bilbao, Vizcaya 48950 Spain; [3]UMR, Dept. of Metallu., 210 Fulton Hall, Rolla, MO 65401 USA

This research aimed to develop a clean technology for the production of ZnO from residues generated by the steel making industry. The scientific and technologic parameters were both investigated in order to define an industrial methodology allowing the integral recycling of EAF dusts. A reengineering of the Wáelz process was performed, aiming to produce a clean fraction of ZnO, free of fluorine, as well as a slag enriched in metallic iron, which can be recycled to the electric arc furnace. The technical conditions used to produce the ultra pure fraction of ZnO are described. The magnetic separation of the EAF dust allowing the production of a phase high in iron and directly recyclable to the electric arc furnace is also discussed. Thermodynamic of the volatilization of all the elements contained in the EAF dust was performed in order to allow a better control of the thermal process in the rotary kiln. Tests were run using different reducing agents, weakly or highly reactive, to determine their specific efficiency.

9:10 AM

Zincproblem Integrated Steel Industries Gradually Alters to Source of Lead and Zinc with the Aid of Metallurgy: *Simon Honingh*[1]; [1]Hoogovens Staal BV, Environmental Control & Mgmt. Sys., P.O. Box 1000, IJmuiden 1970CA The Netherlands

Hoogovens has been investigating different options for the processing of their zinc/lead containing flue dust. Four methods have been applied on laboratory and/or pilot scale to process this flue dust viz. (i) pressure leaching and subsequent hydrometallurgical processing, (ii) processing of the flue dust together with spent pickling acid in a pyrohydrolyzer, (iii) pyrometallurgical treatment of the flue dust in an electric arc furnace/plasma furnace and (iv) processing of the flue dust in a cupola furnace. Various results obtained will be discussed and evaluated to establish which process is most suitable for processing of this flue dust.

9:30 AM

Comparative Study on Zinc Electrowinning from Sulfate and Chloride Solutions: Carla Lupi[1]; *Daniela Pilone*[1]; [1]University of Rome “La Sapienza”, Dept. ICMMPM, Via Eudossiana 18, Roma 00184 Italy

Zinc electrowinning from sulfate solutions is the traditional industrial method to produce SHG zinc. Over the past few years several efforts have been done to improve process efficiency in order to reach high productivity with low energy requirement. The latter aim can be pursued both by a hard purification of the electrolyte and by a reduction in the anodic voltage. In this work electrowinning tests on industrial electrolyte have been carried out by using Pb-Ag anode and by adding organic depolarizers such as ethanol, acetic acid or ethylene glycol. At the same time tests were performed on chloride solutions obtained from purification of zinc exhausted electrolyte: these experiments have been done in different operative conditions by using DSA anodes and by testing the effect of various additives such TEACl and TBACl. Comparing the best results obtained with the two methods it can be highlighted that a high quality zinc is electrowon in both cases, but the energy requirement is lower in the case of zinc chloride electrowinning.

9:50 AM

The Use of Data Reconciliation to Optimise Metallurgical Plants-Case Study Zn Plant: *Markus Andreas Reuter*[1]; Sabina Grund[2]; Thomas Auping[1]; [1]TU Delft, Raw Matls. Processing, 120 Mijnbouwstraat, Delft 2628RX The Netherlands; [2]Consultant, Alter Postweg 12, Dorsten 46282 Germany

This paper discusses data reconciliation as a tool to assist in the modeling and optimisation of metallurgical plants. After discussing the theory the methodology is illustrated using an industrial hydrometallurgical zinc plant as a case study. Various sections of the plant are mass balanced and various fundamental and practical useful relationships are derived. These are discussed in terms of among others process control, metallurgical control, accounting and environmental monitoring.

10:10 AM

Spouted Bed Electrowinning of Zinc from Zinc Chloride Electrolytes: *A. Roy*[1]; J. W. Evans[1]; C. Allen[2]; [1]University of California, Dept. of Matls. Sci. and Eng., Berkeley, CA 94720 USA; [2]Noranda Inc., Tech. Ctr., 240 Hymus Blvd., Point-Claire, Quebec H9R1G5 Canada

The spouted bed electrode (SBE) is a particulate electrode in which metal particles may be grown from a small “seed” to particles that are a few mm across. In prior work at UC Berkeley the SBE has been studied to determine its suitability for use in electrowinning zinc (from both conventional acid sulfate electrolytes and from alkaline electrolytes) and copper. The paper describes the results of a laboratory investigation into the application of this electrode for electrowinning from zinc chloride electrolytes. The anodic reaction was the evolution of chlorine and, when steps were taken to minimize attack of the zinc particles by this chlorine, high current efficiencies (as high as 93%) were obtained. The dependence of cell performance on some operating/design parameters such as current density (up to 4381A/m2) was examined. Reasonable electrical energy consumptions (as low as 3kWh/

kg Zn) were found under many circumstances. Zinc deposits appeared metallic to the naked eye and dense under the SEM.

General Non-Ferrous Pyrometallurgy: Pyrometallurgical Processing of Minerals and Metals

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee

Program Organizers: Robert L. Stephens, Cominco Research, Trail, British Columbia V1R 4S4 Canada; Pekka Taskinen, Outokumpu Research Oy, Pori FIN-28101 Finland

Tuesday AM   Room: Bayou B
March 14, 2000   Location: Opryland Convention Center

Session Chair: Adrian C. Deneys, Praxair, Tarrytown, NY 10591-6714 USA

8:30 AM

Effect of Temperature and Oxygen Partial Pressure on Phase Equilibria of Natural Chromite Minerals: *Vilas D. Tathavadkar*[1]; *Clair C. Calvert*[1]; Animesh Jha[1]; M. P. Antony[1]; Martin Wilkinson[2]; [1]University of Leeds, Dept. of Matls., Leeds, West Yorkshire LS29JT UK; [2]Elementis Chromium, Egalescliff, Stockton-on-Tees, UK

Chromite ore is of immense importance to the extraction of chromium metal and chemicals. The mineralogy and crystal structure of chromite phase are therefore important part of our investigation on the extraction chemistry of chromium species. In this paper, we have investigated the effect of heat treatment on mineral composition and structure as a function of temperature and gaseous atmosphere. The effect the processing parameters i.e. temperature and oxygen potential on the microstructure and composition of mineral phases formed have been systematically investigated in view of the phase equilibria in the Cr_2O_3-Al_2O_3-Fe_xO_y-MgO system and spinoidal decomposition reactions. Experiments were carried out in order to study the differences in the crystallography of different chromite minerals. Phase equilibria in natural chromite minerals has been investigated by calcining chromite mineral in air, argon and 5% hydrogen + argon atmospheres over a temperature range from 200 to 1200°C. The effect of the oxygen partial pressure and temperature on the phase constituents of the calcined product has been discussed. The microstructural changes were examined using scanning electron microscope (SEM) and the changes in elemental composition in different phases were analysed by electron probe micro analyser (EPMA). The results obtained from EPMA studies were used to calculate lattice parameter of the spinel lattice and these values were compared with the values obtained from X-ray powder diffraction data. It is evident from the experimental results that spinoidal decomposition of complex spinel phases has occurred under the influence of different oxygen partial pressures and temperatures.

8:55 AM

Chlorination Kinetics of Xenotime with Chlorine in Presence of Carbon: Marco Antonio Gimenes[1]; *Herenilton Paulino Oliveira*[1]; [1]Faculdade de Filosofia, Ciências e Letras de Rib. Preto-USP, Chem., Av. Bandeirantes 3900, Ribeirão Preto, São Paulo 14040-901 Brazil

The utilization of chlorination in extractive metallurgy and advanced ceramics areas has been widely investigated as a preparative route in order to obtain intermetallic compounds, which are used as precursors in the development of new materials and process. The major processes of decomposition of rare-earth ores use sulfuric acid and sodium hydroxide at high temperatures; however, there is not much work concerning chlorination. In this work, a systematic study of the reaction between xenotime ($REPO_4$), chlorine, and carbon has been performed. Particular emphasis was on kinetics studies to establish optimized conditions for the reaction. The kinetics of chlorination of xenotime raw material by rare-earth elements/compounds has been studied over a temperature range from 600°C to 1100°C. The influence on the rate of conversion of xenotime to $RECl_3$ of temperature, partial pressure of chlorine, carbon content, and particle size were investigated. A global rate equation that includes these parameters has been developed. The results show that the process follows the unreacted core shrinking model with a formation of a product layer. The powder X-ray diffraction technique corroborated the model showing clearly the patterns related to the formation of yttrium oxychloride (YOCl), indicating that the reaction mechanism involves the presence of an intermediate step before the formation of lanthanide chloride. We thank FAPESP for financial support (proc.:1997/05779-1).

9:20 AM +

Reaction Sequences in Sulphide Particle Oxidation: *Esa J. Peuraniemi*[1]; Ari Jokilaakso[1]; [1]Helsinki University of Technology, Lab. of Matls. Proc. and Powder Metallu., P.O. Box 6200, Espoo 02015 Finland

Oxidation of chalcopyrite concentrate and two low-iron copper mattes were studied using a laboratory scale laminar-flow furnace simulating the phenomena taking place in the reaction shaft of a flash furnace. In the experiments, screened fractions were fed into the furnace and sampled after short reaction time intervals by quenching them into a water film. Experimental conditions included temperatures of 1100 and 1300°C with reaction gas oxygen contents from 21 to 75 vol%. Samples were analysed chemically for Cu, Fe and S to define their respective removal rates. Optical and scanning electron microscopy with EDS-analyser were used to examine the phenomena occurring during reactions. Oxidation kinetics as well as ignition of particles are discussed. Changes in particle morphology, size, and composition are viewed to closely follow the development of the oxidation phenomenon and, consequently, to compose a detailed model of dust formation and reaction mechanisms. Data obtained in the experiments enlarges the knowledge of physical and chemical phenomena in flash reactions and serves as reference for computer simulations.

9:45 AM

Thermodynamics of Deoxidation of Molten Titanium and Zirconium: *Yoshinao Kobayashi*[1]; Fumitaka Tsukihashi[2]; [1]National Research Institute for Metals, Matls. Creation Rsch. Station, 1-2-1, Sengen, Tsukuba-shi, Ibaraki 305-0047 Japan; [2]The University of Tokyo, Dept. of Advanced Matls. Sci., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033 Japan

The removal of oxygen from titanium and zirconium is important for use in application because it significantly affects the mechanical and physical properties such as ductility and toughness. However, thermodynamic properties of oxygen in molten titanium and zirconium have not been clarified and the effective deoxidation process of molten titanium and zirconium has not been established. In this study, thermodynamic properties of oxygen and yttrium in molten titanium and zirconium have been investigated by a chemical equilibrium technique. Molten titanium, zirconium, and their alloys with aluminum were equilibrated with a Y_2O_3, Al_2O_3, or ZrO_2 pellet in a cold crucible. The results are summarized as follows: $1/2O_2(g)=O(X_O$, in Ti), ΔG^0=-566,000+103T[J/mol](1673 to 1873K), $1/2O_2(g)=O(X_O$, in Zr), ΔG^0=-543,000+64.6T[J/mol](1848 to 1973K), Y_2O_3(s)=2Y(mass pct, in Ti)+3O(mass pct, in Ti), ΔG^0=601,000-262T[J/mol](1991 to 2093K), Y_2O_3(s)=2Y(mass pct, in Zr)+3O(mass pct, in Zr) and ΔG^0=736,000-317T[J/mol](2153 to 2173K). The possibilities of deoxidation by using yttrium-based fluxes are discussed based on the observed thermodynamic data.

10:10 Break

10:20 AM

Study on Electroslag Remelting of Cu-Cr-Zr Alloy: *Xiao Yu Shen*[2]; *Ji He Wei*[1]; [1]Shanghai University, Dept. of Metallic Matls., 149 Yan Chang Rd., Shanghai 200072 PRC; [2]Shanghai Electrical Apparatus Research Institute, 505 Wu Ning Rd., Shanghai 200063 PRC

Electroslag remelting (ESR) of Cu-Cr-Zr alloy has been experimentally investigated. The remelting experiments were carried out with different slags in the CaF_2+NaF, CaF_2+ZrO_2 and CaF_2+NaF+ZrO_2 systems in an ES unit of 25 kg capacity. The influence of slag on the losses of alloying elements (Zr and Cr) has been considered and examined. The technologies of the pressure working and heat treatment of the ER ingot have been described and discussed. Some physical properties of the slag in CaF_2+NaF+ZrO_2 system and the remelted Cu-Cr-Zr alloy have been determined. The results indicated that with the slag in a CaF_2+NaF+ZrO_2 system and other operation parameters employed, a high quality ingot of Cu-Cr-Zr alloy may be made with high yields of Zr and Cr by means of ESR. For the remelted Cu-Cr-Zr alloy with a specified composition of 0.5-mass% Cr and 0.1 mass-% Zr, the softening temperature, hardness and electrical conductivity are 550°C, HRB 75 and larger than 43 MS/m, respectively. The properties of Cu-Cr-Zr alloy products are in accordance with and superior to the requirements and specifications of ISO 5182-1991(E).

10:45 AM

Kinetic Study on the Carbon Thermal Reduction of V_2O_3: *Zhiyu Lu*[1]; Zhitong Sui[1]; Zhenqi Huang[1]; Jing Yang[1]; [1]Northeastern University, School of Matl. and Metallu., Shenyang 110006 PRC

Vanadium is an important alloy element in making high strength low alloy steel in which vanadium can play the role of dispersion strengthening to the steel. In recent years there has been great interest in vanadium carbide (VC), vanadium nitride (VN) and vanadium carbide nitride (V(C,N)), because all of the three compounds can exhibit excellent quality for the addition of vanadium to the molten steel. In making these compounds V_20_3 is an important raw material due to its low oxygen content in the molecule, so the kinetic study on reduction of V_2O_3 can provide valuable information to the production of VC, VN, V(C,N). In this paper the reduction process of V_2O_3 was studied by TG and XRD method, the study results reveal that the reduction rate was controlled by both interface chemical reaction step and mass transport step in the experimental temperature scope (1300K—1700K). The apparent activation energy for the interface chemical reaction was 34kJ/mole; the apparent activation energy for the mass transport step was 32kJ/mole. The study on the reduction mechanism can give the following conclusion. When the temperature is lower than 1300K, the carbon reduction mechanism of V_2O_3 give mainly VC, and there are enough clues indicates that the main reaction is a self-catalyzed reaction at this temperature range.

High-Temperature Superconductors: Coated Conductors

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

Program Organizers: U. Balu Balachandran, Argonne National Laboratory, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, Latham, NY 12110-0461 USA; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA

Tuesday AM — Room: Canal D
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Chandra S Pande, Naval Research Laboratory, Matls. Sci. Div., Washington, DC 20375 USA

8:30 AM Invited

High-Jc, YBCO Conductors Fabricated by Epitaxial Deposition of YBCO on Rolling Assisted Biaxially Textured Substrates (RABiTS): *Amit Goyal*[1]; F. A. List[1]; D. F. Lee[1]; D. M. Kreoeger[1]; M. Paranthaman[1]; X. Cui[1]; R. Feenstra[1]; D. T. Verebelyi[1]; T. Autug[1]; C. Cantoni[1]; D. K. Christen[1]; P. M. Martin[1]; T. Chirayil[1]; C. Park[1]; D. P. Norton[1]; R. K. Williams[1]; E. D. Specht[1]; D. B. Beach[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, P.O. Box 2008; MS 6116, Bldg. 4500S; Rm. B-248, Oak Ridge, TN 37831 USA

Advances in the fabrication of Rolling assisted biaxially textured substrates (RABiTS) and epitaxial deposition or formation of HTS on such substrates is reported. Significant progress has been made in the fabrication of non-magnetic, strengthened, biaxially textured metal templates, deposition of oxide and other buffer layers and in the fabrication of long length substrates and superconductors. High Jc's exceeding 1 MA/cm2 have been demonstrated on epitaxially grown YBCO films on RABiTS using Ni-Cr as the starting template. High Jc's have been demonstrated on a variety of new buffer layer configurations including conducting buffer layers. Efforts are underway to fabricate longer length superconductor samples exceeding 10cm and results obtained will also be summarized. Particular emphasis would be given to microstructural factors affecting Jc in coated conductors. Research sponsored by U. S. Department of Energy under contract DE-AC05-96OR22464 to Lockheed Martin Energy Research Corporation.

9:10 AM

Microstructure of YBCO Films Deposited on Oxide Buffered Rolling Assisted Biaxially Textured Ni Substrates: *Chau-Yun Yang*[1]; S. E. Babcock[1]; A. Goyal[2]; F. A. List[2]; J. E. Mathis[2]; C. Park[2]; M. Paranthaman[2]; D. F. Lee[2]; D. P. Norton[2]; D. M. Kroeger[2]; [1]University of Wisconsin-Madison, Appl. Superconductivity Ctr., 1500 Engineering Dr., Madison, WI 53706 USA; [2]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831 USA

The microstructures of pulsed laser deposited YBCO films on different buffer layer materials including YSZ, CeO_2, and Yb_2O_3 were studied with a view toward identifying current limiting defects and elucidating the roles of the buffer layer material in YBCO coated conductors. The YBCO films deposited on YSZ possess an island microstructure, and the island size increases with increasing film thickness. The observed current limiting mechanisms are unlikely to be low angle grain boundaries between YBCO islands but pores, especially the columnar pores elongated through YBCO [001] direction when the films are over 1 mm thick, second phase particles, and 45° rotated grains. The YBCO film grown on CeO_2 does not show the same island microstructure, rather a single YBCO grain that contains twin boundaries on both sets of {110} planes extend over the entire 10 to 20 microns observable area of TEM specimens. Although some dislocations widely dispersed in the film, no clear domain structure is observed from YBCO on a given Ni grain. Two observed main current limiting factors are open pores and poor YBCO connection across Ni grain boundaries. The results is a films with a low Jc value of just ~ 0.2 MA/cm2. The high Jc (1.7MA/cm2) YBCO film grown on recently developed new buffer Yb_2O_3 has an island microstructure like that of YBCO on YSZ. These results suggest that microstructural details of length scale ranging from sub-micron to many tens of microns depend on the buffer layer. This work is supported by US-DOE through ORNL.

9:50 AM Break

10:00 AM Invited

Direct Measurements of Grain Boundary Transport Currents in YBa2Cu3Ox Coated Conductors: *Dean J. Miller*[1]; Peter Berghuis[1]; Ron Feenstra[2]; Dave K. Christen[2]; [1]Argonne National Laboratory, Matls. Sci. Div., 9500 S. Cass, MSD-223, Argonne, IL 60439 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

The impressive transport properties achieved in YBa2Cu3Ox coated conductors have led to an intense effort to understand the key factors that influence the critical current and current density on this type of conductor. In this work, we aim to understand the current path in these conductors and particularly how grain boundaries influence the critical current density. We use micropatterning techniques to isolate individual grain boundaries on coated conductors prepared using RABiTS substrates. The transport properties measured across these single grain boundaries are then compared to those across grain boundaries from thin film and bulk bicrystals as well as to the global transport properties of coated conductors. In this presentation, details of the patterning and measurement of critical currents will be presented. An emphasis will be placed on the comparison with global transport properties and the influence of the criterion used to establish the critical current. This work was partially supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences and Energy Efficiency and Renewable Energy, under contract #W-31-109-ENG-38.

10:40 AM
IBAD/MOCVD-Based YBCO-Coated Conductor Development: *U. Balachandran*[1]; M. P. Chudzik[1]; R. A. Erck[1]; C. R. Kannewurf[2]; V. Selvamanickam[3]; P. Haldar[3]; [1]Argonne National Laboratory, Energy Tech., 9700 S. Cass Ave., Argonne, IL 60440 USA; [2]Northwestern University, Dept. of Elect. Eng., Evanston, IL 60208 USA; [3]Intermagnetics General Corporation, Latham, NY 12110 USA

Biaxially aligned yttria-stabilized zirconia (YSZ) films were deposited on polished Hastelloy substrates via ion-beam-assisted deposition (IBAD). Atomic plumes were created by electron beam evaporation and an argon-ion gun aided texture development in the YSZ. Effects of ion-to-atom flux ratio and beam optics divergence on in-plane texture of YSZ were investigated. Epitaxial cap layers of cerium oxide were deposited on IBAD/YSZ films by electron beam evaporation. YBCO superconductors of ≈1 µm thickness were deposited on ceria cap layers by metal-organic chemical vapor deposition (MOCVD). The MOCVD processing parameters were optimized and critical currents of >50 A (critical current density >1 MA/cm^2) were obtained at 77K. The deposition conditions required to obtain the textured layers will be discussed. *Work at ANL and part of the work at IGC supported by the U.S. Department of Energy, Energy Efficiency and Renewable Energy, as part of a program to develop electric power technology, under Contract W-31-109-Eng-38. Work at NU supported by the National Science Foundation through the Science and Technology Center for Superconductivity (Grant No. DMR 91-2000).

11:20 AM Invited
A Study on the Grain Texturing Mechanism of YBCO Film on a Silver Alloy Substrate through Peritectic Solidification: *Donglu Shi*[1]; [1]University of Cincinnati, Matls. Sci. & Eng., 498 Rhodes Hall, Mail Location 0012, Cincinnati, OH 45221-0012 USA

Quenching experiments were carried out near the peritectic temperature for the thick films of YBa2Cu3Ox (YBCO) on the silver alloy substrate. The initial YBCO morphology exhibits a column-like grain structure as a result of rapid a-growth when quenched from 1000°C (the sample was pre-melted at 1030°C). A waffle-like structure was observed on the surface of the silver alloy substrate as the quenching temperature was lowered to 950°C providing a much greater driving force. We found that a grain-oriented substrate may not be required to achieve the grain texturing in the peritectic-reaction-controlled process. During solidification, the YBCO grains will nucleate on the surface of the silver alloy in a parallel fashion to minimize its surface energy, and grow along the a-axis rapidly resulting in a textured film.

Honorary Symposium for Professor Oleg D. Sherby: Creep Mechanisms and Behavior A

Sponsored by: Structural Materials Division, Materials Processing and Manufacturing Division, Structural Materials Committee, Shaping and Forming Committee
Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Chol K. Syn, Lawrence Livermore National Laboratory, Manufacturing & Materials Engineering Division, Livermore, CA 94550 USA

Tuesday AM — Room: Bayou E
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Alan Ardell, University of California, Matls. Sci. and Eng. Dept., Los Angeles, CA 90095-1595 USA

8:30 AM Opening Remarks

8:40 AM Keynote
Mechanisms of Time-Dependent Plasticity in Polycrystalline Thin Films on Substrates: *William D. Nix*[1]; [1]Stanford University, Matls. Sci. and Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94087-2205 USA

Mechanisms of time-dependent plasticity in polycrystalline thin metal films (Al, Cu, Au) on silicon substrates are reviewed with particular reference to understanding the softening and hardening effects of dislocations and grain boundaries. Diffusional deformation contributes to plasticity in these fine-grained materials, though it can be inhibited by the presence of thin passivating layers. For unpassivated films it is shown that diffusional deformation involving mass transport between the free surface of the film and the grain boundaries is constrained by kinetic processes at the film/substrate interface and that classical Coble or Herring-Nabarro creep relations do not apply unless the film/substrate is free to slide. For the case of no sliding at the film/substrate interface, diffusional deformation alone cannot relax the stresses completely at high temperatures, with the consequence that dislocation plasticity must be active when full relaxation is observed. For passivated films, plasticity appears to be dominated by dislocation processes. Dislocation plasticity is largely athermal in passivated thin films because the storage of dislocations at the film/substrate and film/passivation interfaces dominates the deformation resistance. The hardening effects of grain boundaries are also explored and compared with the hardening processes that occur in single crystal films. Some time-dependent stress relaxation is observed in Au films at room temperature. This appears to be controlled by thermally activated dislocation cutting processes.

9:10 AM Invited
The Interpretation of Creep Mechanisms in High Temperature Flow: *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA

Several different flow mechanisms may occur in high temperature creep and an identification of the rate-controlling process is generally based on measurements of the stress exponent and the activation energy. This paper considers the different creep processes occurring in crystalline solids and examines procedures for their unambiguous identification.

9:30 AM Invited
Subgrain Strengthening Revisited II: *Michael Ernest Kassner*[1]; [1]Oregon State University, Dept. Mech. Eng., Rogers Hall, Corvallis, OR 97331 USA

This work is a sequel to earlier work with Prof. Oleg D. Sherby and the author on the effects of subgrain boundaries on elevated-temperature plasticity [1]. This work discusses some of the more recent work in this area, including investigations by the author, that attempted to discern the contributions of various substructural features, including subgrain boundaries, on the rate-controlling process for five power-law-creep in single-phase metals. Particular attention is devoted to recent developments regarding internal back-stresses. This will include discussions of recent in-situ TEM, x-ray diffraction and convergent beam electron diffraction experiments on metals to evaluate internal stresses in association with dislocation heterogeneities. [1]O.D. Sherby, A.K. Miller and M.E. Kassner, "Subgrain Strengthening Revisited", Metals Forum, 4, 1981, pp. 53-56.

9:50 AM Invited
Constant Structure Creep of Aluminum: All the Data in the World: *Jeffrey C. Gibeling*[1]; [1]University of California, Dept. of Chem. Eng. and Matls. Sci., One Shields Ave., Davis, CA 95616-5294 USA

The analysis of creep under conditions of constant internal structure is reviewed, with the goal of using these results to develop a better understanding of the mechanisms of deformation. Data from both strain rate change experiments and stress change tests are considered. While both types of experiments give similar results, greater emphasis is given to the latter in the present discussion. Data for aluminum from numerous investigations present a consistent picture over a wide range of temperatures and stresses. These results show that constant structure creep after stress reductions occurs by parallel processes of dislocation glide within subgrain interiors and dynamic recovery associated with subgrain boundaries. After relatively small changes in stress, thermally activated motion of dislocations within subgrain interiors is the predominant mechanism of deformation. In this regime, a thermally activated rate law can describe the creep transients, thereby enabling various activation parameters to be evaluated from the data. In particular, the true activation areas are consistent with thermally activated cutting of forest dislocations. In contrast, dynamic recovery processes dominate the constant structure deformation following relatively large stress reductions. Selected results for other FCC metals and related materials are shown to follow the trends established for pure aluminum. In particular, it is demonstrated that constant structure creep of pure copper and LiF at high temperatures and after small stress changes is also consistent with a description based on thermally activated glide. The differences in behavior between these materials are attributed to differences in stacking fault energy.

10:10 AM Break

10:20 AM Keynote

Unnatural Power-Law Creep Exponents, Nonlinear Monotonic and Cyclic Stress-Strain Curves and Similitude Breaking: *Johannes Weertman*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Many alloys have a "natural" power law exponent of about 3 at the higher temperatures. Pure metals and some alloys have an "unnatural" power law exponent of about 5 at the higher temperatures and an unnatural power law exponent of about 7 (instead of 5) at moderately high temperatures. It is argued in this talk that the discrepancy of the exponent in high temperature creep, as well as the occurrence of nonlinear elastic-plastic stress strain curves, is a consequence of similitude breaking in the underlining dislocation mechanics. (Similitude breaking is a concept introduced by Kuhlmann-Wisdorf Met. Trans. 16A, 2091 (1985) to dislocation mechanics.)

10:50 AM Invited

New Predictions of the Dislocation Network Theory of Harper-Dorn Creep: *Alan J. Ardell*[1]; Marek A. Przystupa[1]; [1]UCLA, Matls. Sci. and Eng., 6531-G Boelter Hall, Los Angeles, CA 90095-1595 USA

The dislocation network theory of high-temperature deformation explains many features of Harper-Dorn (H-D) creep that other theories do not. One is that the dislocation density in the H-D creep regime is independent of the applied stress. Frustration of dislocation network coarsening, arising because Frank's rule cannot be satisfied at the nodes when the network coarsens and dislocations are eliminated, is responsible. The reduction in dislocation density during primary creep in the H-D regime is also satisfactorily explained. Previous equations of the network theory involved formation of only one kind of node resulting from dislocation collisions and annihilation. In real f.c.c. crystals several kinds of interactions are possible, leading to different configurations at dislocation nodes. In the present work we take these into account. We apply the new equations to describe the experimentally measured distributions of dislocation link lengths in Al. We also attempt to provide a self-consistent prediction of creep curves in Al deformed in the H-D regime.

11:10 AM Invited

Effect of Stress-Ratio on Biaxial Creep and Dislocation Microstructures in Recrystallized $Ti_3Al_2.5V$ Tubing: *K. Linga Murty*[1]; S. Nangalia[2]; A. Paradkar[3]; [1]North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA; [2]MCNC Electronic and Information Technologies, RTP, NC 27709-2889 USA; [3]Defence Metallurgical Research Laboratory, Hyderabad, India

Biaxial creep characteristics were investigated on recrystallized $Ti_3Al_2.5V$ tubing by varying the internal pressurization superimposed with axial load at 673K. The stress ratio () of the hoop to axial was varied from 0 to while the hoop and axial creep strains were monitored using a telemetric laser extensometer and an LVDT respectively. The creep locus defined at a constant energy dissipation rate deviated from isotropy with relatively large hardening towards the axial stress axis. The results were fit to the modified Hill's equation from which the creep anisotropy parameters, R and P, were derived and these parameters deviated from unity with R quite large (~6) and P relatively smaller (~0.5). The stress-state dependence of the strain-rate ratio predicted using these anisotropy parameters was in reasonable agreement with the experimental results albeit relatively large scatter was noted in the data. The crystallographic texture was determined using x-ray diffraction via inverse and direct pole figures from which crystallite orientation distribution functions (CODF) were derived. Predictions based on crystal slip plasticity in conjunction with CODF exhibited large deviations, which were initially thought to arise from contributions from twinning and/or second phase. Dislocation microstructures were investigated as a function of the stress level following creep tests at uniaxial (=) and equi-biaxial (=1) loading. At stresses below about 180 MPa, dislocation arrangement was generally random while at higher stresses (at ~211 MPa) distinct subgrain formation was noted. Whereas, uniaxially loaded samples exhibited random distribution of dislocations even at very high stresses (~351 MPa). In all cases, the dislocations are predominantly <11 0> type lying on {10 0} planes. Some <c+a> type dislocations were observed in few low angle grain boundaries, and these are relatively widely separated. No twins were noted while only small amounts of  phase were observed confined mainly to grain boundaries.

11:30 AM Invited

An Evaluation of Power Law Breakdown in Metals, Alloys and Compounds: *Donald R. Lesuer*[1]; Chol K. Syn[1]; Oleg D. Sherby[2]; [1]Lawrence Livermore National Laboratory, L-342, P.O. Box 808, Livermore, CA USA; [2]Stanford University, Dept. of Matls. Sci. and Eng., Stanford, CA 94305 USA

Creep at high stresses often produces strain rates that do not follow a power law relationship between strain rate and stress. At low stress, a power law relationship is observed while, at high stress, greater strain rates are observed than would be predicted by this relationship. This phenomena is referred to as Power Law Breakdown (PLB). In this paper, we examine the available creep data at high stress in pure metals, solid solution alloys, dispersion strengthened alloys and compounds to identify materials characteristics and experimental conditions (stress, temperature and strain rate) that result in PLB. The results are analyzed by assessing the influence of these higher stresses and strain rates on the diffusion coefficient. For a number of Fe-C alloys, PLB can be explained by the increase in dislocation density with increasing stress and the resulting increase in diffusivity. When creep results in the Fe-C alloys are analyzed in terms of an effective diffusivity, which includes lattice and pipe diffusion, PLB disappears. Power Law Breakdown has also been studied in terms of the production of excess vacancies and the influence of excess vacancies on the diffusion coefficient. A simple model has been constructed to study PLB based on the rates of production and annihilation of vacancies. The results provide insight into the physical basis for PLB in metals alloys and compounds. Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract W-7405-ENG-48.

Hume Rothery Award Symposium; Phase Transformations and Evolution in Materials: Session II

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

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA

Tuesday AM — Room: Johnson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: David E. Laughlin, Carnegie Mellon University, Matls. Sci. and Eng., Pittsburgh, PA 15213-3890 USA; Alan J. Ardell, UCLA, Matls. Sci. and Eng., Los Angeles, CA 90024 USA

8:30 AM Invited

Some Aspects of Ordering Energy: *Robert W. Cahn*[1]; [1]Cambridge University, Matls. Sci. & Metallu., Pembroke St., Cambridge CB2 3QZ England

The ordering energy of an alloy with a superlattice determines a number of its properties, including the temperature (actual or virtual) of the order-disorder transition, the specific energy of antiphase domain boundaries, the change in lattice parameter when order is lost, the width of superdislocations, resistance to creep under stress, and some aspects of recrystallization. In spite of the fact that several distinct methods exist to estimate the ordering energy, both experimental and theoretical, not much attention has as yet been paid to this variable. This discursive survey will look at a number of these aspects.

9:00 AM Invited

Dynamical Evolution of Ordering in Alloys: Phase Field and Monte Carlo Simulations: *Alphonse Finel*[1]; [1]Onera/CNRS, LEM, BP72, Chatillon, Cedex 92322 France

Structural or ordering transformations in alloys lead generally to the coexistence of many different phases or domains which form complex microstructures. At a mesoscopic time and space scale, most of the characteristics of these microstructures are determined by elastic effects, in particular growth laws of particle sizes and scaling properties. We will discuss these aspects in a continuous phase field approach valid for coherent systems. We will also present a general atomistic approach, based on Monte Carlo simulations, which, in simple cases, reproduces well-know properties (particle shape transitions, precipitates interactions) and which is able to deal with arbitrary local deformations, coherent or not, such as semi-coherent or incoherent interfaces, dislocations.

9:30 AM Invited

First Principles Concentration Functional Approach to Alloy Phase Stability: *George Malcolm Stocks*[1]; [1]Oak Ridge National Laboratory, Met. and Cer. Div., P.O. Box 2008-6114, Oak Ridge, TN 37831-6114 USA

In this paper I review the origins, successes, and limitations of the first principles concentration functional theory of ordering and phase stability developed in the early 80's [B. L. Gyorffy and G. M. Stocks, Phys. Rev. Letters {\bf 56}, 374 (1983)]. When evaluated in the mean field approximation and combined with the first principles local density approximation Korringa-Kohn-Rostoker coherent-potential-approximation (LDA-KKR-CPA) the, in principle exact, concentration functional approach provides a direct way of relating clustering and short range order to the underlying electronic structure of the disordered phase. I will review applications of the LDA-KKR-CPA concentration functional method to a number of classical problems in alloy theory and to the magnetic phase transition in ferromagnetic transition metals. In each case stress will be placed on understanding the underlying electronic mechanisms (Fermi surface nesting, band filling, charge transfer, ...) that give rise to the observed ordering behavior. I will place particular emphasis on what as been learned in recent years regarding screening in substitutionally disordered alloys and its affect on alloy phase stability based on large cell (100-1000 atom) simulations. Work supported by Office of Basic Energy Sciences, Division of Materials Sciences, US Department of Energy, under subcontract DEAC05—960R22464 with Lockheed-Martin Energy Research Corporation.

10:00 AM Invited

Master Equation Approach to Configurational Kinetics of Non-Equilibrium Alloys and Its Applications to Studies of Phase Transformations: Kirill Belashchenko[1]; Vladimir Dobretsov[1]; German Samolyuk[1]; *Valentin Vaks*[1]; [1]Russian Research Center, Kurchatov Instit., Moscow 123182 Russia

We review a series of works where we use the fundamental master equation to develop a consistent theoretical description of the evolution of non-equilibrium atomic distributions in an alloy. We derive exact equations for the temporal evolution of local concentrations and correlators of their fluctuations, as well as for the free energy of a non-equilibrium alloy. To solve these equations we employ the approximate methods analogous to those used in the equilibrium statistical physics including the kinetic mean-field method and the kinetic cluster methods. In particular, we develop a kinetic cluster field method being a kinetic analogue of the known cluster variation method which combines high accuracy in the description of thermodynamics with great simplification of the calculations. We also suggest a microscopic model to describe the influence of elastic forces on the phase transformation with a lattice symmetry change, such as the tetragonal distortion under $L1_0$ ordering. The developed methods are used for extensive studies of the microstructural evolution under various phase transitions including the decomposition of disordered alloys and orderings of B2, $D0_3$, $L1_2$ and $L1_0$ type both without and with phase separation as well as without and with elastic effects. These studies reveal a number of new and interesting microstructural effects, many of them agreeing well with experimental observations.

10:30 AM Break

10:45 AM Invited

Computational Investigations on the Microstructure Formation in Real Alloy Systems Based on the Phase Field Method: *Toru Miyazaki*[1]; Toshiyuki Koyama[1]; [1]Nagoya Institute of Technology, Dept. of Matls. Sci. & Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan

The kinetic simulation based on the non-linear diffusion equation become very powerful method in fundamental understanding the dynamics of phase transformation with the recent remarkable development of computer. In the present study, we calculate the dynamics of microstructure changes in real alloy systems, such as Fe-Mo, Al-Zn, Fe-Al-Co and GaAs-InP based on the phase field method. The composition dependencies of atomic interchange energy are taken into account so as to be applicable for the phase diagram of the real alloy systems. The elasticity and mobility of atoms are assumed to depend on the local order parameters such as composition, degree of order, etc. Time dependent morphological changes of the microstructure such as formation of modulated structure by spinodal decomposition, strain induced morphological changes of precipitates, the order-disorder phase transition with phase decomposition, discontinuous precipitation will be demonstrated. The results simulated are quantitatively in good agreement with the experimental results in the real alloy systems.

11:15 AM Invited

Evolution of Microstructure and Defect Structure in Polytwinned Ferromagnets: *William A. Soffa*[1]; [1]University of Pittsburgh, Matls. Sci. and Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The L10 family of ferromagnets are interesting magnetic materials from both a scientific and technical point of view. These intermetallics, which include CoPt, FePt, FePd and MnAl, form as stable or metastable phases in the vicinity of the equiatomic alloy compositions. The tetragonal structures are characterized by a high magnetocrystalline anisotropy with an "easy" c-axis and exhibit anisotropy constants in the range K1 ~ 107-108 ergs/cm3. The formation of the L10 phase from the parent phase in these alloy systems generates a characteristic polytwinned structure and defect structure which have important implications regarding the transformation mechanisms involved and the resultant structure-property relationships. In this paper, the development of the microstructure and defect structure characterizing the L10 polytwinned structures will be discussed and related to the nature of the disorder -> order transformation giving rise to these polytwinned ferromagnets. This work has been supported by NSF and DOE.

11:45 AM Invited

The Chessboard and Saw-Tooth-Like Morphologies in Decomposing Alloys: *Yann M. Le Bouar*[1]; Armen G. Khachaturyan[2]; [1]Commissariat a l 'Energie Atomique, DTA/SRMP, Cea-Saclay, Gif-Sur-Yvette 91191 France; [2]Rutgers University, Cer. and Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08855-0909 USA

The understanding and control of the microstructure evolution of multiphase alloys is of critical importance to synthesize advanced materials with given properties. During the last decades, the use of Transmission Electron Microscopy (TEM) has enabled a detailed description of the microstructure of numerous multiphase alloys. We focused here on puzzling microstructures obtained during ordering in the platinum-rich $L1_0$+$L1_2$ two phase region of the Co-Pt system. First, we present a series of TEM images (Dark Field and High Resolution images) to describe the complex microstructures of our Co_x-Pt_{1-x} alloys. Depending of the average concentration, our TEM images show that the microstructure may evolve either towards a chessboard or a platelet-like microstructure. Then, we present two computational methods, based on the continuum stochastic field kinetic equations or the microscopic Master Equation, able to describe a first order phase transition with a cubic -> tetragonal symmetry reduction. No a priori constraints are made on the possible configurations and the sequence of structural pattern. Finally, 2D computer simulations are performed for an elastically isotropic and homogeneous crystal. When the concentration of the alloy is chosen close to the middle of the $L1_0$+$L1_2$ two phase region, our continuous simulations predict the formation of a chessboard microstructure, whose edges are aligned in the elastically soft directions, and show that the coarsening of such a microstructure is only possible with the disappearance of an entire band of the pattern. When the concentration is close to the $L1_2$ stability region, our microscopic simulation explain the formation of the platelet or saw-tooth-like morphologies. All the simulation results are in excellent agreement with our experimental observation in the Co-Pt system.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Stereolithography and Selective Laser Sintering

Sponsored by: Materials Processing and Manufacturing Division,

Program Organizers: David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; James W. Sears, Lockheed Martin, KAPL Inc., D2, 114, Schenectedy, NY 12301 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Rob Wagoner, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Tuesday AM　　Room: Canal E
March 14, 2000　　Location: Opryland Convention Center

Session Chair: David L. Bourell, University of Texas-Austin, Austin, TX 78745 USA

8:30 AM

Solid Freeform Fabrication: A United States Navy Perspective: *Khershed P. Cooper*[1]; George Spanos[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech., Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375 USA

ONR in cooperation with other federal government agencies such as DARPA, OSD and NSF has been sponsoring research in Solid Freeform Fabrication (SFF) technology since it's early days. These agencies have played a major role in promoting the understanding and development of manufacturing processes such as Fused Deposition Modeling (FDM), 3-D Printing (3DP), Selective Laser Sintering (SLS), Shape Deposition Modeling (SDM), Stereolithography (SLA), Selected Area Laser Deposition (SALD), Direct Photo Shaping and others. SFF projects have been pushed to experiment with various homogeneous materials and combination of materials (functionally graded materials). Technologies are finding their way into industry for customized prototypes and even for functional components. But theoretical and experimental work remains to be done and new opportunities in diverse areas such as electronic packaging and biomedical engineering are emerging. This talk will survey some of the projects supported by ONR and other agencies and discuss some possible Navy applications.

9:00 AM

Effect of Build-Plane Orientation on the Mechanical Properties of Parts Made by Fused Deposition Modeling (FDM) and by Stereolithography (SLA): *Nancy S. Losure*[1]; Ward Jensen[2]; [1]Mississippi State University, Swalm Schl. of Chem. Eng., Mississippi State, MS 39762 USA; [2]Oreck Manufacturing, 21180 Oreck Blvd., Long Beach, MS 39560 USA

Rapid prototyping techniques have traditionally been used to produce parts to validate design dimensions and assembly procedures. Stereolithographic (SLA) techniques produce parts with excellent surface finish and detail, but limited strength and problems with dimensional stability. On the other hand, a relatively new process, Fused Deposition Modeling (FDM), produces parts from an engineering resin, ABS, with good surface finish, much higher strength and excellent dimensional stability. It is important that the mechanical properties of prototype parts be understood, particularly with respect to the direction of build so that users can maximize the benefit from a rapid prototyping program. In this work, we will produce tensile, bending and impact specimens in which the build-plane is perpendicular to each of the dimensional axes, in order to determine how the build-plane orientation affects the strength and stiffness of prototype parts produced by FDM and SLA.

9:20 AM

Mechanical Alloying Polymer Blends for Selective Laser Sintering: *Julie Patricia Martin*[1]; Ronald George Kander[1]; [1]Virginia Tech, Matls. Sci. & Eng. Dept., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

In this work, mechanical alloying (MA) is presented as an effective alternative to traditional coating methods for creating co-continuous phases during selective laser sintering (SLS). Specifically, the morphology of mechanically alloyed polymer blends for use in the SLS process is investigated in this research. By varying the charge ratio, time, and temperature of the MA process, the phase domain size of the resulting composite powder can be manipulated and the physical, mechanical, and electronic properties of the blend altered. The mechanically alloyed powder can then be selectively laser sintered into parts containing co-continuous phases. Although polymer/polymer composite morphologies are studied here, the MA process is also a viable technique for creating SLS powders using ceramics or metals.

9:40 AM

Selective Laser Sintering of Polymer-Polymer, Polymer-Metal, and Polymer-Ceramic Composite Powders Made by Cryogenic Mechanical Alloying: *Jeffrey Patrick Schultz*[1]; Ronald George Kander[1]; Carlos Tres Ayala Suchicital[1]; [1]Virginia Tech, Matls. Sci. and Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

Cryogenic mechanical alloying (CMA) offers a new means of producing composite powders for selective laser sintering (SLS). Unlike composite particles made by a coating process, both materials are continuous throughout the particle. Consolidation of these composite particles via SLS offers the possibility of forming a co-continuous microstructure in parts produced by SLS. The work presented is an initial investigation into the mechanical properties and microstructural characteristics of three SLS material systems formed by CMA: a polymer-polymer, polymer-ceramic, and polymer-metal system. The polymer-polymer composite has applications in fabrication of functional prototypes and direct manufacturing, and the polymer-metal polymer-ceramic systems are both used for fabrication of green structures and direct manufacturing.

10:00 AM

Direct Laser Fabrication of High Performance Metal Components via SLS/HIP: Suman Das[1]; *Martin Wohlert*[1]; Joseph J. Beaman[1]; David L. Bourell[1]; [1]University of Texas at Austin, Mech. Eng., MC 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 processing (HIP) to rapidly produce low volume or "one of a kind" high performance metal components. The advantages of in-situ encapsulation include elimination of a secondary canning step and container material, no container-powder interaction, reduced pre-processing time, and reduction in post-processing steps compared to HIP of canned parts. SLS/HIP is currently being developed for superalloy 625 and Ti-6Al-4V. Microstructure and mechanical properties of material processed by SLS/HIP are comparable to conventionally processed material. Results of SLS/HIP development for superalloy 625 are presented.

10:20 AM Break

10:40 AM

Selective Laser Sintering with Meso-Scale Features: *Nicole Harlan*[1]; Seok-Min Park[1]; Joseph J. Beaman[1]; David L. Bourell[1]; [1]University of Texas at Austin, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

Recent work in Selective Laser Sintering of materials at the University of Texas at Austin demonstrates the capability to produce parts with fine features of the order of 100 μm to 200 μm. Zirconia powder was pre-processed into spherical particles, laser sintered with a sacrificial polymer binder, infiltrated and post-sintered to higher density. Optical micrographs show that hole sizes of 180 μm are possible in fully ceramic components. Several examples illustrating the potential of Selective Laser Sintering of parts with meso-scale features will be presented.

11:00 AM

Zirconia Molds for Titanium Casting: *Nicole Harlan*[1]; Seok-Min Park[1]; David L. Bourell[1]; Joseph J. Beaman[1]; [1]University of Texas at Austin, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

A combination of selective laser sintering and colloidal infiltration has been used to create "partially stabilized" zirconia molds for titanium casting. The mold material system was chosen for its low reactivity with molten titanium and thermal shock resistance. The base material, stabilized zirconia mixed with a copolymer binder, is laser sintered into the desired green shape. The binder is removed and replaced by zirconia. The fired parts show graded porosity from a dense surface to a porous interior. The average density of the fired parts can be increased to twice that of the green density. Surface roughness (Ra) is less than 10 μm and flexural strength is sufficient for high temperature casting. A half-scale casting mold for the head of a human femur bone was produced using laser scanned data.

11:20 AM

Solid Free-Form Fabrication of Refractory Metal Components: *Gary K. Lewis*[1]; Joe C. Fonseca[1]; Ron B. Nemec[1]; Tom N. Taylor[1]; Paul Burghardt[1]; [1]Los Alamos National Laboratory, Mail Stop G770, Los Alamos, NM 87545 USA

Directed Light Fabrication, a solid free-form fabrication process, has been shown feasible to build layered components from almost any metal in a single step. This process is viewed as particularly beneficial to the fabrication of refractory metal components by saving multiple powder consolidation, thermo-mechanical processing, joining, and machining steps used in conventional processing. However, refractory metal powders, in contrast to many lower melting point powders that are typically gas or water atomized, are produced by a chemical reduction, precipitation process. DLF processing of these refractory metal precipitate powders results in porosity in the solidified product that is being traced back to trace elements in the powder. Additional powder processing prior to deposition to remove or reduce undesired elements has proven effective in eliminating porosity. Characterization of powders and deposits and optimization of DLF process parameters is leading to successful fabrication of refractory metal components.

11:40 AM

SFF Using Inverted Projection of Liquid Metal Droplets: *Dawn R. White*[1]; Sankaran Subramaniam[1]; Larry Jepson[2]; [1]Ford Research Laboratory, P.O. Box 2053, MD 3135 SRL Bldg., Dearborn, MI 48121-2053 USA; [2]University of Texas-Austin, SFF Lab., Austin, TX 78712-1063 USA

A number of droplet based free form fabrication techniques have been developed. Without exception, these involve the formation and transfer of a droplet to a substrate beneath the droplet origination site. Under these conditions, gravity has the effect of causing droplet spreading upon impingement. Factors such as surface tension, viscosity of the droplet, droplet liquid fraction, etc. affect the degree of spreading and wetting involved, however, the result is that part geometry is difficult to control precisely. Thus, SFF parts made using droplets often have fairly rough or irregular surfaces, which require finish machining for many applications. If however, the droplet is projected upwards, and allowed to impinge and hang on a "superstrate," gravity and surface tension interact to produce an elongated droplet, with a smooth side. As these hanging droplets accumulate to for an SFF object, our experiments show that it has a much smoother, and dimensionally controllable sidewall, than an object produced in the conventional manner. This paper presents a numerical analysis of the hanging droplet approach to SFF. In addition, experiments were performed using projected aluminum and steel droplets. The results of experiments conducted on single and multiple droplets, and bulk features are also presented.

International Symposium on Iridium: Applications

Sponsored by: Structural Materials Division, Refractory Metals Committee

Program Organizers: Evan K. Ohriner, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; H. Harada, National Research Institute for Metals, Tsukuba, Ibaraki 305 Japan; R. D. Lanam, Engelhard-CLAL, Careret, NJ 07008 USA; Peter Panfilov, Ural State University, Ekatrinburg 62001 Russia

Tuesday AM — Room: Jackson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Richard D. Lanam, Engelhard-CLAL LP, Carteret, NJ 07008 USA; N. I. Timofeev, Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Research Center, Ekaterinburg 620014 Russia

8:30 AM

Characterization of Sputter-Deposited Iridium Oxide Coatings for Medical Implants: *T. Loose*[1]; M. Frericks[1]; T. Giesel[1]; G. Herklotz[1]; [1]W.C. Heraeus GmbH & Company KG, Heraeusstrasse 12-14, Hanau D-63450 Germany

Iridium oxide coatings were produced by d.c. reactive magnetron sputtering of a metallic iridium target in an Ar/O_2 atmosphere. The properties of the deposit depend on the sputter parameters (time, power, oxygen flow, temperature) as well as the substrate material (titanium, stainless steel). Cyclo-voltametry was used to investigate electrochemical properties. Corrosion in 0.9% NaCl-solution was measured by potentiostatic voltametry at the corrosion potential of the uncoated surface. The composition of the iridium oxide was determined by auger electron spectroscopy, the surface morphology by scanning electron microscopy. Adherent, crack free coatings with good mechanical and corrosion stability can be achieved using a metallic interlayer.

8:50 AM

Production of Iridium Crucibles by Electrolysis of Molten Salts: *N. I. Timofeev*[1]; V. E. Baraboshkin[1]; N. Saltykova[2]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]Institute of High Temperature Electrochemistry, Ekaterinburg, Russia

Iridium has the high melting point and unique resistance to aggressive oxidation environments at high temperatures. It is used as container material for growing of high temperature oxide crystals. The mechanical treatment of iridium has essential difficulties because of its considerable hardness. In the Ekaterinburg Non-Ferrous Metals Processing Plant, the electrolysis was performed in the hermetic electrolyzer in argon atmosphere. The electrodeposition of iridium was carried out in the melt of eutectics of NaCl-KCl-CsCl containing iridium chlorides at 500-600°C. The most optimum method can be considered as the combination of the electrolytic iridium refining of metal- and non-metal impurities with the preparation of iridium articles using the method of galvanoplastics. Therefore, the dissoluble anodic material can be used not only in form of pure iridium but also as its scrap. The cathodic matrices were made of graphite having the form of articles. The anodic current density should be as low as to avoid salt passivation of iridium anode. The reversal current and the rotation of the cathode during electrolysis decreased the grain size of iridium compact layer (coating) and smoothed its surface. To avoid the appearance of the structure coating defects (excrescencies, pores) several technological procedures were used; one of which was the preliminary thermal treatment of graphite matrixes in aim to remove the gases from them. After the electrodeposition of iridium coatings onto matrix the latter was destroyed. The roughness of the internal surface of crucible corresponded to the degree of mechanical treatment of the graphite matrix. The electrodeposited iridium has high plasticity and the same density as iridium castings. Microhardness of electrolytic iridium is 320-340 kg/mm^{2}and Vickers hardness is 240-280 kg/mm^2. The purity of iridium is 99.98-99.99% and the main impurities are the metals of platinum group (Pt, Rh). The method of galvanoplastics allows producing various articles (tubes, rings, and disks). It is an ecologically pure and practically waste-free technology.

9:10 AM

Experimental Technology for Production of Disks from High Strength Iridium: *A. V. Ermakov*[1]; A. V. Sedavnykh[1]; L. G. Grokhovskaya[1]; S. G. Tretiakova[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

The sources of radioisotope Ir^{192} are manufactured by radiation treatment of iridium targets in reactor. Iridium discs for this purpose should meet hard conditions, such as (1) no contaminants should be contained in the metal; (2) concavity of discs is about 10^{-2} mm for diameter of 1.5-3.0 mm and thickness of 0.1-0.3 mm; and (3) their edges should not crumble after processing. Technology for manufacture of these discs has been elaborated. Massive iridium crystals, whose purity is 99.9%, are used as workpieces. Mechanical treatment of crystals at high temperatures allows obtaining sheets with grain size of 5 x 10^{-2} mm. Fine grain iridium can be stamped to discs and calibrated during one technological operation. Besides, these discs do not crumble. Another way for suppressing the crumble is the coating of disc edges by aluminum.

9:30 AM

Radioactive ^{192}Ir Gamma-Sources Made from Enriched ^{191}Ir: W. Borneman[1]; M. Frericks[2]; *D. F. Lupton*[2]; H. Rakhorst[3]; [1]Malinckrodt Medical B.V., Petten Netherlands; [2]W.C. Heraeus GmbH & Company KG, Heraeusstrasse 12-14, Hanau D-63450 Germany; [3]Urenco Nederland B.V., Almelo, Netherlands

Radioactive ^{192}Ir sources play an important role in medical applications, especially brachytherapy, and in industrial non-destructive testing (NDT). Due to the nature of nuclear reactors with variable neutron fluxes and sometimes unplanned shutdowns, the supply of iridium sources has not only been under stress, but also the guarantee of a minimum yield (Ci/gram) has been difficult. Current sources are made by irradiating natural iridium, in solid metal form, in a nuclear reactor. Natural iridium, however, contains only 37% ^{191}Ir that is activated to ^{192}Ir, the remainder being ^{193}Ir which is not activated. Urenco has a proprietary gas centrifuge technology that is extensively used for enriching ^{235}U for commercial nuclear power plants. A successful R&D programme has adapted this technology to enrich ^{191}Ir to at least 85% using the process gas IrF_6. By means of a hydrogen reduction treatment, this compound is converted to high purity iridium black. The ultra-fine iridium metal powder is consolidated to compact products by Heraeus. The iridium black is induction melted and then remelted under high vacuum by an electron beam process, to ensure ultra-high purity. The fine powder can also be consolidated by powder metallurgical techniques, but experience has shown this to be a less satisfactory route. As a result of the extremely high purity, forming operations can be carried out more readily than with conventional grades of iridium. The advantage of enriched ^{191}Ir is a much more efficient use of reactor neutrons, resulting in much smaller sources, or sources with much higher specific activity. These efficiency gains outweigh the higher costs of enrichment and pellet manufacturing. Irradiation results have verified a near doubling of specific activity of irradiated 85% ^{191}Ir versus natural iridium giving the following advantages: (1) no minimum yield problem, (2) unplanned shutdowns of reactors do not jeopardize the continuity of supply, and (3) longer shutdown periods of reactors can be overlapped.

9:50 AM

Iridium and Iridium Alloy Utilization in Ignition Devices: *L. F. Toth*[1]; D. A. Toenshoff[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

The high melting and boiling points, corrosion and spark erosion resistances of iridium and iridium alloys make these ideal candidates for electrodes in ignition devices for use in the hostile environment of a typical combustion chamber. The demands for ever increasing environmental and combustion efficiency and longer service life in automobile engines strongly suggest the utilization of iridium and/or its alloys as spark plug electrodes. This paper will examine some of the thermomechanical properties and processing of iridium materials, their incorporation and behavior in various ignition devices, and an insight into some of the obstacles encountered in the use of iridium and potential methods of overcoming them.

10:10 AM Break

10:20 AM

The Use of Iridium in High Temperature Thermometry: *J. Grossi*[1]; [1]Engelhard-CLAL, 4025 Clipper Ct., Fremont, CA 94538 USA

One of the lessor known industrial uses of iridium is the application of it and its alloys in high temperature thermometry. This use is becoming increasingly important with the need to accurately measure higher temperatures especially under oxidizing conditions. This paper will focus on current applications such as gas turbine instrumentation, temperature measurement for crystal growth and industrial furnace control. In addition, practical error considerations; such as utilization of proper extension wires and data acquisition recommendations will be discussed.

10:40 AM

Iridium/Rhenium Combustion Chambers for Chemical Propulsion: *A. J. Fortini*[1]; R. H. Tuffias[1]; R. B. Kaplan[1]; A. J. Duffy[1]; B. E. Williams[1]; J. W. Brockmeyer[1]; [1]Ultramet, 12173 Montague St., Pacoima, CA 91331 USA

Most satellites placed in geosynchronous orbit rely on silicide-coated niobium combustion chambers for apogee topping maneuvers. These chambers typically operate at temperatures between 1300 and 1400°C. At higher temperatures, rapid oxidation of the silicide takes place and catastrophic failure occurs. Current state-of-the-art combustion chambers now comprise a rhenium structural shell protected by a thin film (typically 0.003") of chemical vapor deposited (CVD) iridium on the interior. Iridium is ideal for this application due to its excellent oxidation resistance, high melting point, and thermal expansion match with rhenium, and CVD is the preferred processing technique because the resulting coating is very pure and free of cracks, porosity, pinholes, and other defects. Using this architecture, combustion chambers can now be operated at temperatures well in excess of 1900°C, which yields a dramatic increase in performance.

11:00 AM

The Use of Iridium for Jewelry: *C. Volpe*[1]; K. Vaithinathan[2]; R. Lanam[2]; [1]Tiffany & Company, 143 Sparks Ave., Pelham, NY 10803 USA; [2]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

The principle use of iridium for jewelry has been as an alloying addition to platinum to increase hardness and improve resistance to wear. The compositions and application of the platinum-iridium alloys will be discussed. A recent development is the use of iridium itself in jewelry. Iridium's properties of being very hard and corrosion resistant make it a desirable material for jewelry. Through near-net shape and surface enhancing processes unique designs can be achieved. Examples of the traditional Pt-Ir and new iridium uses will be shown.

11:20 AM

Iridium as Container Material for Oxide Crystal Growing: N. I. Timofeev[1]; B. Dorogovin[2]; *A. V. Yermakov*[1]; S. Yu. Stepanov[2]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]VNIISIMS, Alaxandrov, Russia

Iridium is the sole material for containers, where large single crystals of aluminum-yttrium garnet AYG ($Y_3Al_5O_{12}$), gallium-gadolinium garnet GGG ($Gd_3Ga_5O_{12}$), gallium-lanthanum silicate ($La_3Ga_5SiO_{14}$), and other oxides could be grown. Experience has shown that crucible is the key element of growing technology, which determines purity and quality of crystals. Iridium containers with size up to 200 mm in diameter, 200 mm in height, thickness of walls is 2 mm could be manufactured either traditional technology (rolling of sheets, stamping of cans and welding of crucibles) or galvanoplastic means (production of jointless crucibles). Welding crucibles from single crystal workpieces (wall thick of 2 mm) possess highest working resource for iridium containers: their life-time in GGG is 2000 hours and in AYG is 800 hours, while lifetimes of galvanoplastic ones are 1000 hours for GGG and 120 hours for AYG. Resource of welding containers from electron beam melted iridium is less than for "single crystal" crucibles, but higher than for ones.

11:40 AM

Iridium and Its Application-Present Status in Japan: *T. Maruko*[1]; [1]Furuyametals Company Limited, 1915 Morizoesima, Shimodateshi, Ibaraki 308-0861 Japan

Iridium demand is tremendously increasing, especially since 1995 in Japan, with an increasing variety of applications. One application is the newly developed catalyst to reduce automotive engine emission. Another is in the electrochemical field where about 25% of total iridium demand is consumed for electrodes used in industrial electrolysis, chlor-alkali electrolysis, and electro-galvanizing among others. Demand for the iridium crucibles and parts comprises about 3% of total supply. Those include applications of the compound single crystal growth for advanced electronics devices, YAG laser tips, and SAW devices. Growth in iridium demand in the near future is expected. Due to its high refractory characteristics, iridium finds application in thermocouples, rocket parts, gas-turbine blades, glass manufacturing, and spark plugs. Use of ferroelectric memory so called F-RAM, having superior nonvolatility, speed, and volume and requiring less power consumption, is expected to grow requiring iridium as a best candidate per production of various materials such as PZT and SBZ. We have succeeded in developing various materials and overcoming various difficulties in processing through the special processing techniques including microstructure control, improved purification, and special additives. High efficiency in the recovery iridium is very important to minimize tremendous price changes caused by imbalance in demand and supply associated with increased applications of iridium. New recovering and recycling processes for iridium are discussed.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee

Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Tuesday AM — Room: Lincoln E
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Ray Y. Lin, University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA; Bruce MacDonald, National Science Fundation, Div. of Matls. Rsch., Arlington, VA 22230 USA

8:30 AM

Solubility and Raman Spectra of Niobium (V) Fluoro and Oxo Fluoro Complexes Formed in LiF-NaF-KF-Melts: *Aasmund F. Vik*[1]; Terje Ostvold[1]; Vassilis Drakopoulos[2]; George N. Papatheodorou[2]; [1]Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway; [2]Institute of Chemical Engineering and High Temperature Chemical Processes, Forth, P.O. Box 1414, GR-26500, Patras, Greece

The solubility of Nb (V) as a function of the Na_2O concentration in the ternary eutectic LiF-NaF-KF (FLiNaK) melt at 700°C has been determined. The Raman spectra at different oxide concentrations and temperatures were also recorded. Based on chemical analysis of melt samples and the observed Raman bands, suggestions related to the Nb-O-F complexes formed in the melt are given. In FLiNaK containing 0.22 mole kg^{-1} K_2NbF_7, the following observations were recorded at varying total molar ratios of oxygen to niobium in the melt + the solids formed, n^oO/n^o_{Nb}: 1. $n^oO/n^o_{Nb} < 2$; all Nb (V) and O^{2-} was dissolved. The Raman spectra indicated the presence of monomeric NbF_7^{2-} at low oxide concentrations, which reacted with oxide to form monomeric $NbOF_5^{2-}$ when n^oO/n^o_{Nb} was increasing. $NbO_2F_4^{3-}$ was formed upon further additions of oxide and was the dominating specie at $n^oO/n^o_{Nb} = 2$. 2. $2 < n^oO/n^o_{Nb} < 3$; a solid of the type $AlkNbO_3$ was formed; $n^oO/n^o_{Nb} = 3$; a minimum in the Nb (V) and O^{2-} solubilities were observed. The structure of the species in the melt was not easy to establish due to a very weak Raman signal. 3.3 < r < 4; the $AlkNbO_3$(s) previously formed dissolved, possibly with the formation of both corner and edge sharing distorted NbO_6 octahedra. The solubility of Nb_2O_5 in FLiNaK was measured as a function of temperature (550°C to 800°C), and the dissolution mechanism seems to be given by the reaction: $Alk^+ + Nb_2O_5(s) + 4F^- = NbO_2F_4^{3-} + AlkNbO_3(s)$. $H°_{sol}$ for the reaction (based on concentrations in mole kg^{-1}) was found to be 89 $kJmol^{-1}K^{-1}$.

9:00 AM

High Temperature Electrochemical Study of the Na_2O-MoO_3 System: *Ray Y. Lin*[1]; John F. Elliott[2]; [1]M.L., Dept. of Matls. Sci. and Eng.; [2]University of Cincinnati, Cincinnati, OH 45221 USA

Physical chemistry of the Na_2O-MoO_3 system was studied using a high temperature electrochemical cell with Na, β-alumina as the solid electrolyte. The reference electrode is a solid mixture of tungsten, tungsten sulfide and sodium sulfide. The cell may be expressed as the following. W(s), WS2(s)| Na+| O_2(g), Pt(s)Na_2S(s) β-alumina Na_2O-MoO_3 melt. The activity of Na_2O in the Na_2O-MoO_3 melt in the composition range from 50.87 to 92.18 mol% MoO_3 and temperature from 890 to 1230 K was determined using this cell. Partial molar enthalpy of mixing for Na_2O in the melt was evaluated from the temperature dependence of the activity data using the Gibbs-Helmhotz equation. The value agrees well with those reported in the literature from calorimetric measurements. The activity of MoO_3 in the melt as a function of temperature and melt composition was also evaluated from the Gibbs-Duhem equation with the help from the Na_2O-MoO_3 phase diagram.

9:30 AM

Comments on the Formation Thermodynamics of the Rare Earth Compounds with the 15th and 16th Group Clements: *R. Ferro*[1]; G. Borzone[1]; [1]Universita degli Studi di Genova, Dept. di Chimica e Chimica Industriale, Via Dodecaneso, Genova 31-16146 Italy

In the framework of a description of the general alloying behaviour of the rare earth ® metals, their thermodynamics of reaction with the 15th and 16th group elements of the Periodic Table is reviewed. Also on the basis of our obtained laboratory results, special attention is given to the thermochemistry of formation of the arsenides, antimonides, bismuthides, selenides and tellurides of the R elements. The properties of these substances are also discussed and compared with those of compounds formed by other transition elements similar to the R metals (actinides, etc.). Problems met in the thermochemical characterization and in the experimental investigation of this family of substances are finally highlighted and discussed.

10:00 AM Break

10:10 AM

Thermodynamic Investigations of Pseudobinary Chalcogenide Systems: *M. Shamsuddin*[1]; [1]Banaras Hindu University, Instit. of Tech., Dept. of Metallu. Eng., Varanasi 221-005 India

In recent years thermodynamic measurements on several pseudobinary chalcogenide systems, viz. CdSe-CdTe, ZnTe-CdTe, HgTe-CdTe, ZnSe-ZnTe and PbSe-PbTe and PbSe-PbTe using fused salt galvanic cell technique have been conducted. Thermodynamic data show that these systems do not follow regular solution model and also do not satisfy Darken's formalism. Hence thermodynamic behaviour of these systems have been analyzed in the light of Darken's stability and excess stability functions. The stability and excess stability together with ideal stability, relative stability and relative excess stability parameters have been calculated at different temperatures. The continuous variation of Darken's stability and constancy of excess stability functions with composition confirm that ZnTe-CdTe, HgTe-CdTe, ZnSe-ZnTe and PbSe-PbTe systems consist of single phase field throughout the entire range of composition. On the other hand, in the CdSe-CdTe system, sharp changes in the variation of stability and excess stability functions near $x_{CdTe} = 0.4$ establish that the system consists of two terminal regions (i) $0 < x_{CdTe} \leq 0.35$ and (ii)$0.45 \leq 1.0$, in which the thermodynamic behaviour is relatively simple. The two terminal regions are connected by a narrow transient or central region ($0.35 \leq$ x CdTe ≤ 0.45), in which the behaviour appears to be complicated.

10:40 AM

Thermochemistry and Modelisation in Oxides: *G. Boureau*[1]; [1]Universite Pierre et Marie Curie, Lab. de Chimie Physique, Matiere et Rayonnement, 11 Rue Pierre et Marie Curie, Paris, Cedex 05 75231 France

In the present paper we show that the informations won from experimental thermodynamics are essential in the understanding of oxides: We shall investigate the following fields: energy of formation of defects in bulk oxides (1) and at the interfaces; (2) both, direct experimental investigation and ab initio studies of oxygen vacancies are difficult. The help of thermodynamics is invaluable in providing severe constraints. Ionic and electronic transport. (3, 4). Without any additional parameters, a purely thermodynamic analysis of experimental data provide a good understanding of electronic transport in some particular cases (polaronic models). Statistical thermodynamics (5). Such an approach needs reliable inputs which may have a number of origins. In some cases, thermodynamics allow to make guesses about the nature of defects and of interactions. Comparison with microscopic observation is particularly useful. Then we shall discuss links between thermodynamic data and charge transfer in silicium oxides and in germanium oxides.

11:10 AM

Thermodynamic Considerations on Solid Oxide Fuel Cell Materials: *Harumi Yokokawa*[1]; Katsuhiko Yamaji[1]; Teruhisa Horita[1]; Natsuko Sakai[1]; Hideyuki Negishi[1]; [1]National Institute of Materials and Chemical Research, Energy-Related Matls. Grp., 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

Thermochemical properties of ZrO_2-based ceramics and perovskite-type oxides were analyzed so as to obtain the good correlation with ionic properties and to reproduce the experimentally determined phase relations. For ZrO_2-based ceramics, the interaction parameters for cubic phases were derived from the phase diagram calculation in the absence of the experimental thermochemical properties. Those parameters which were derived for respective valences of dopants show the good correlation with ionic radii of dopants. This allowed us to calculate solubility of transition metal oxides over a wide range of oxygen potential, in which the valence of the transition metal ions changes drastically. The calculated solubility was compared with experimental values determined by Sasaki et al. Good agreement was obtained except for the zirconium vanadium oxygen system.

11:40 AM

Formation Energies of Molybdenum Borides Measured by Voltammetry: *G. Kaptay*[1]; J. Sytchev[1]; [1]University of Miskolc, Miskolc, Egyetemvaros 3515 Hungary

For measurements the molten NaCl-KCl-NaF (5 mol%)-KBF_4 system has been used at 973K, under argon gas. Experiments were performed in the glassy-carbon crucible, which served as counter electrode and reference electrode, as well. Cyclic voltammetric curves were taken at different scan rates from 0.001 V/s till 10 V/s. When silver was used as cathode, a one-step, 3-electron boron deposition was observed as a reversible electrochemical process. However, when molybdenum wire was used as cathode material, several pre-peaks appeared at more positive potentials compared to deposition of B on Ag cathode. The pre-peaks are due to the formation of molybdenum borides of different stoichiometry. The potential difference between the B-peak on the Ag-cathode and between the pre-peaks observed on the Mocathode are related to the formation energies of different molybdenum boride phases. Calculations proved, that our results are very close to the calorimetric results obtained by Meschel and Kleppa [3]. Thermodynamic properties of all molybdenum borides have been estimated by us using our voltammetry data coupled with information given on the Mo-B phase diagram, and taking into account the calorimetric result of [3].

12:10 PM

Equilibrium in the Liquid Fe-Al-N System: *G. Kaptay*[1]; M. S. Yaghmaee[1]; G. Janosfy[1]; [1]University of Miskolc, Miskolc, Egyetemvaros 3515 Hungary

Abstract text not available

Magnesium Technology 2000: Automotive Issues and Recycling

Sponsored by: Light Metals Division, Reactive Metals Committee, International Magnesium Association

Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA

Tuesday AM	Room: Bayou C
March 14, 2000	Location: Opryland Convention Center

Session Chair: Gerald S. Cole, Ford Motor Company, Ford Rsch. Labs., Dearborn, MI 48121-2053 USA

8:30 AM

Conductivity Measurements on Ingots of Magnesium Die-Casting Alloys: *Carlton D. Fuerst*[1]; Cameron J. Dasch[1]; [1]General Motors R&D Center, Matls. & Process. Lab., MC 480-106-224, 30500 Mound Rd., Warren, MI 48090-9055 USA

Magnesium metal suppliers use a variety of methods to establish metal cleanliness, all of which seek to take advantage of differences between the properties of embedded oxide particles and those of the matrix (magnesium metal). Electrical conductivity measurements are a fast and sensitive means of nondestructive evaluation that offers the potential of detecting the electrically insulating oxide particles embedded in the conductive matrix. However, the results presented in this report reveal that other microstructural features also have a large impact on the conductivity of magnesium die-casting alloys. Aluminum concentration and microstructural porosity are two important variables governing conductivity. A 1 wt% increase in the aluminum level produced a 6-8% decrease in the conductivity of samples studied. This dependence on chemistry is large enough to make it easy to distinguish between the low-aluminum AM-type alloys (AM50A and AM60B) and the high-aluminum AZ91D alloy, using conductivity measurements. Increases in porosity also cause a decrease in conductivity; specifically, conductivities dropped about 3% for each 1% increase in porosity. Given its sensitivity to both chemistry and porosity, it is unlikely that conductivity could be used to directly measure oxide inclusions in ingots. Further work would be necessary to determine its usefulness for specially cast samples where chemistry and porosity can be tightly controlled.

8:55 AM

Observations of Intermetallic Particle and Inclusion Distributions in Magnesium Alloys: *John M. Tartaglia*[1]; John C. Grebetz[2]; [1]Climax Research Services, 39205 Country Club Dr., C-40, Farmington Hills, MI 48335-5718 USA; [2]Daimler Chrysler Corporation, Auburn Hills, MI USA

As part of an international program to qualify magnesium alloy ingots as feed stock for die castings, numerous studies have been conducted on the microstructures of AM50, AM60, and AZ91 virgin and recycled ingots. The studies have focused on intermetallic particles and inclusions distributed throughout the ingot bulk and concentrated in surface defect regions such as folds. The studies have included optical macrography and micrography to document the particle distributions; automated optical image analysis for quantifying the particle volume fraction and size; and scanning electron microscopy and energy dispersive spectroscopy to characterize the elemental content of the particles and surrounding matrix. Several exemplary case studies will be presented in this talk.

9:20 AM

Utilization of Centrifugal Casting in Recycling of Magnesium Alloy Scraps: *A. Arslan Kaya*[1]; Serdar Sevik[1]; Havva Zeytin[1]; [1]Tubitak-Mam, Marmara Rsch. Ctr., Matls. and Chem. Tech. Instit.-Matls. Grp., P.O. Box 21, Gebze-Kocaeli 41470 Turkey

The total production and consumption figures of magnesium metal indicate that recycling will soon become an integral part of the magnesium industry. Recycling of magnesium alloys necessitates the remelting of scrap metal and removal of inclusions as well as adjusting the chemistry before it can be reused for casting. This requires the development of an effective recycling technology for magnesium scrap. This study has been undertaken to assess the feasibility of recycling magnesium alloy AZ91 scrap by utilizing centrifugal casting process as part of the recycling technology. The goal was to concentrate the inclusions in the outer skins of billets during centrifugal casting for subsequent removal. Centrifugal casting parameters were varied to understand their effects on the distribution of inclusions throughout the cross section of billets. Light and scanning electron microscopy aided with microanalysis were used to determine the size, distribution and the types of inclusions. A critical assessment has been made regarding the applicability of the method as a recycling process.

9:45 AM

Magnesium Melting/Casting and Remelting in Foundries: *Horst Dörsam*[1]; [1]StrikoWestofen GmbH, Lorenz-Schott-Strasse 5, D-55252 Mainz-Kastel, Postfach 5909, Weisbaden D-65049 Germany

New furnaces and casting plants are described, having a capacity of 100 to 5400 kg at melting capacities of 100 to 1500 kg magnesium per hour. Tiltable, gas-heated furnace designs are also available which are mostly used as melting furnaces for clean recycling material for subsequent casting in ingot moulds. The application of modern ingot heating and charging machines is shown which prevent that humid material is charged into a liquid melt. Units for charging of ingots with the most different dimensions are available. The capacity is, for example, 36 ingots which can be applied by hand in a ferry‘s wheel. A preheating takes place and an automatic charging into the crucible furnace in accordance to the casting capacity. Throughputs of up to 1000 kg magnesium per hour at an average ingot size of 8 kg are possible. A mixed gas control cabinet enables the mixture with one another of up to three different mediums in a previously exactly defined relation. Normally, dry air is mixed with SF6 or SO2. CO2 as third gas can also be added. Thus, it is possible to constantly mix the desired protective gas into the furnace to cover the magnesium melt. A survey of the magnesium pumps available on the market with experimental values from the practical casting is given, especially those from Norsk Hydro and Dynarad. Casting quantities of 0.5 to 30 kg per shot are possible. The desired casting quantities can be preset and are casted directly into the filling chamber of the diecasting machine by means of the casting tube or in another casting form. Existing remelting plants are described and a preview is given about the developments which are to be expected, as for example the “inhouse recycling” plant with ingot casting machines and for liquid charging towards the pouring furnaces which is getting more and more important. New developments of furnace series with optimized handling systems and geometric sizes to minimize the respective space required for the furnaces are shown as well as economical and environmental aspects will be described in brief, especially for the trendy large casting parts in high pressure diecasting.

10:10 AM Break

10:20 AM
Filling and Solidification Modeling of Noranda's Magnesium Wheel Casting Process: *Randy Sheng*[1]; Don Argo[1]; [1]Noranda Technology Centre, 240 Hymus Blvd., Pointe-Claire, Quebec H9R1G5 Canada

Since 1993, Noranda has made significant efforts in developing expertise in the area of aluminum wheel casting simulation and process improvement. Recently, a counter-pressure wheel casting machine at Noranda Inc. Technology Center was successfully modified to cast magnesium wheels. To supply necessary process data such as the cycle time and the mold-cooling intensity, model simulations of the casting process were carried out using different magnesium alloys. The model results revealed that compared to aluminum alloy A356, the casting of magnesium wheel requires more process optimization. The lower volumetric heat content of magnesium alloys is more likely to cause an over-cooled mold and premature solidification in the wheel-rim region. Furthermore, the long solidification path from the wheel rim to the hub requires an optimized mold-temperature distribution to maintain a directional wheel solidification pattern. A carefully determined cycle time and programmed mold cooling process is therefore needed to obtain a stable casting operation and to produce sound magnesium wheels.

TUESDAY AM

10:45 AM
Materials Comparison and Potential Applications of Magnesium in Automobiles: *Alan A. Luo*[1]; [1]General Motors Research and Development Center, Matls. & Process. Lab., 30500 Mound Rd., P.O. Box 9055, Mail Code 480-106-212, Warren, MI 48090-9055 USA

In this paper, the material properties, structural performance, mass saving potentials, design and manufacturing characteristics of magnesium are compared against various competing materials such as cast iron, steel sheet, aluminum alloys and polymers. The current and potential automotive applications of magnesium are reviewed, and the technical challenges for these applications are also discussed. Recent alloy development for powertrain applications and the creep resistance of several experimental magnesium alloys are critically reviewed. Generic R&D needs for expanding the use of magnesium in automobiles are given at the end of the paper.

Materials Processing in the Computer Age III: Process Optimization and Control

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Tuesday AM — Room: Lincoln A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Vaughan R. Voller, University of Minnesota, Civil Engineering, Minneapolis, MN 55455 USA; Hani Henein, University of Alberta, Adv. Matls. and Process. Lab., Edmonton, Alberta T6G2G6 Canada

8:30 AM Opening Remarks

8:40 AM Keynote
Optimization of Manufacturing Processes: *Daniel Anthony Tortorelli*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. and Indust. Eng., Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

Structural optimization programs are often used to design products for minimum weight subject to constraints on stress, displacement, etc. These optimization problems are solved iteratively; and in each iteration 1) the appropriate finite element analyses are performed, 2) the cost and constraint functions are evaluated, 3) the sensitivities of these measures are computed, and 4) a nonlinear programming algorithm is used to perform a convergence check and, if necessary, update the design and initiate another iteration. These algorithms generally converge in a few iterations. However, to attain such desirable convergence characteristics, accurate sensitivity information must be available. The aforementioned optimization is for structures whose response is governed by the theory of linear elasticity. Unfortunately, manufacturing processes cannot be modeled with linear theories. To design such nonlinear systems we may use the same iterative optimization algorithm discussed above. However, the nonlinear analysis and subsequent sensitivity analysis are more complex. To facilitate the treatment of such design problems, the analysis and sensitivity analysis are derived for a general nonlinear system. It is shown that the computational demand that is required to perform the sensitivity analysis is only a small fraction of that which is required to perform the actual analysis. Three specific examples are provided to illustrate these methods. 1) The geometry of a polymer extrusion die is designed to minimize velocity and residence time variations over the outlet. A Hele-Shaw analysis is used to model the flow of the melt through the thin cavity. 2) An element-by-element discontinuous Galerkin method is used to simulate precipitate nucleation and growth during the quench process of an aluminum alloy extrusion. In the subsequent optimization, the process parameters are determined to control distortion and the size distribution of precipitates. 3) The die shape in an aluminum drawing process is designed to minimize distortion. The steady-analysis is modeled using a novel mixed method in which the displacement and internal field variables appear as the primary unknowns.

9:20 AM Keynote
Process Control: From PID to CIM: *J. Fraser Forbes*[1]; [1]University of Alberta, Chem. and Mat. Eng., 536 Chem. Mineral Bldg., Edmonton, Alberta T6G2G6 Canada

The meaning of the term "process control" has evolved significantly since its widespread use began in the manufacturing industries in the 1940's. Further, the scope of process control applications has drastically increased both in terms of breadth of industries using process automation technologies and the vertical integration of these technologies within a single industry. This evolution and integration of process control technologies within the manufacturing industries has proceeded to such an extent that process automation is an integral part of modern business practices. This paper will examine the current status of process control technology from simple process regulation through process performance optimization to business planning. Each stage of the operations automation hierarchy will be discussed both in terms of the current state-of-the-art and the directions in which these technologies are evolving. The key tools and tasks necessary for success at every level of the automation hierarchy will be introduced. Finally, the critical challenges that must be faced in accomplishing the goal of complete Computer Integrated Manufacturing (CIM) will be described. Finally, although this paper addresses issues of practical importance to any manufacturing company, the discussions will be framed in terms of the particular challenges facing and the opportunities available within the materials manufacturing industries.

10:00 AM Break

10:10 AM
Optimizing Heat Treatment Techniques for Large Castings and Forgings: *Koushik Ray*[1]; [1]Concurrent Technologies Corporation, Matls. Eng. Grp., 100 CTC Dr., Johnstown, PA 15904 USA

Military Specification testing requirements dictate that mechanical properties of large castings and forgings are to be examined at a particular depth (for e.g. at a depth of 3" for a 12" thick casting). The experimental method for determining the heat-treatment parameters to produce optimum mechanical properties, by uniquely heat-treating several identical blocks, is impractical for economic and material handling reasons. This paper describes a method in which Finite Element Analysis (FEA) is used to determine the heat treatment required to obtain the optimum mechanical properties at a given depth. First, thermal response for a heat treatment at a given depth in the block is generated using FEA. This thermal response is then experimentally matched for a thin slice of the block material, so that it receives the same thermal response as if it were within the material, at that depth. By generating thermal response profiles for different heat treatments on the large block using FEA, and experimentally matching each of the thermal responses for different slices, the consequences of a large number of possible heat treatments can be studied by evaluating the mechanical properties of each of the slices. Experimental validation for a predicted heat treatment is also included.

10:30 AM
Optimization and Modeling of Thermomechanical Deformation and Microstructure Development during Metal Forming: Liming Zhang[1]; Ben Q. Li[1]; Reza A. Mirshams[2]; [1]Washington State University, School of Mech. Eng., Pullman, WA 99164 USA; [2]Southern University, Mech. Eng., Baton Rouge, LA 83808 USA

This paper presents a finite element-based numerical optimization approach to the control of thermomechanical deformation and microstructure formation during metals forming processes. A finite element model is developed based on the modification of our in-house code for the solution of non-Newtonian fluid flow and heat transfer to solve the rigid plastic deformation problems with penalty method applied to treat the pressure field. The finite element model is strongly coupled with a microstructure materials model that is capable of representing the microstructure development during metals forging processes. This coupled finite element model then forms as a core module that is used by a Newton based optimization methodology. With this approach, the thermomechanical field variables such as temperature distribution, strain rate and deformation and microstructural properties such as grain size distribution and recrystallization volume fraction can selectively controlled so as to achieve an optimized condition for metals forming. Numerical results will be presented to illustrate the application of this optimized approach to control the forging processes for isothermal and nonisothermal compression of carbon steel and TiAl alloys.

10:50 AM
Optimization of Continuous Slag/Matte Separation in Mitsubishi Process by Using Numerical Heat and Fluid Flow Analysis: Nozomu Hasegawa[1]; *Akira Kaneda*[1]; [1]Mitsubishi Materials Corporation, Cent. Rsch. Instit., 1-297 Kitabukuro-Cho, Omiya, Saitama 330-8508 Japan

In the Mitsubishi Continuous Copper Smelting and Converting Process, slag/matte mixture overflowed from a smelting furnace is separated continuously in an electric furnace (slag cleaning furnace, CL-furnace). In order to improve the separation of slag and matte and to minimize power consumption, the design and the operating conditions of the CL-furnace should be optimized. In the previous study, we developed a numerical model for simulating phenomena in the CL-furnace. The model consisted of 4 modules, electric field analysis, heat and fluid flow analysis, tracer simulation and the particle tracking method. In this study, the model was partially modified, k-ε model was adopted, and the effect of various conditions, such as furnace shape (including accretion) and scale, electrodes configuration, immerse depth of electrodes, thickness of slag layer and so on, was evaluated. And the optimum design and operating conditions are discussed.

11:10 AM
Development of a Model for Online Simulation and Control of a Continuous Steel Slab Caster: Richard A. Hardin[1]; Kai Liu[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mech. Eng., 2412 SC, Iowa City, IA 52242 USA

A computational model for online simulation and control of a continuous steel slab caster is described. First, a highly accurate, though slower than real-time, two-dimensional transient heat transfer model of the caster was developed. The model computes slab thermal and solidification conditions as a function of time-varying casting speed, secondary spray cooling water flow rates and temperature, slab thickness, steel chemistry, and pouring and ambient temperatures. This model was then streamlined into a real-time model that can be used for online simulation and control. Issues involved with the sacrifice of detail in the real-time model are addressed. This model was implemented on a Windows NT-based computer with an online data interface through the caster's Level 1 system. An overview of the computer-caster hardware interface and the multi-threaded graphical user interface is provided. Good agreement between measured and predicted temperatures for actual caster operating conditions is achieved by using a calibration procedure for the spray cooling correlations. Results of parametric studies are presented that provide insight into the effects of transient changes in casting variables on the slab thermal profile and the solidification endpoint. Finally, the development of a real-time, simulation-based algorithm for secondary spray cooling control is described. The use and effectiveness of dynamic spray cooling control for preventing surface temperature excursions from the desired profile is demonstrated.

11:30 AM Invited
An Interdisciplinary Approach Towards Optimal Continuous Casting of Steel: *Bozidar Sarler*[1]; Bogdan Filipic[2]; Miroslav Raudensky[3]; [1]University of Ljubljana, Fluid Mech. and Thermo., Askerceva 6, Ljubljana SI-1000 Slovenia; [2]Jozef Stefan Institute, Dept. Intelligent Sys., Jamova 39, Ljubljana SI-1000 Slovenia; [3]Technical University of Brno, Heat Trans. Lab., Technicka 2, Brno CZ-61669 Czech Republic

This paper describes main elements and practical use of the computational system for automated setting of optimized casting conditions in the continuous casting of steel. The system consists of the heat transfer model of the process, empirical metallurgical criteria, and optimization algorithm. The physical model of the strand temperature distribution relies on the classical mixture continuum formulation for the convective-diffusive heat transport. Fluid flow effects are considered through enhanced thermal conductivity of the liquid phase. The model is solved by the operator-splitting Crank-Nicolson finite volume enthalpy scheme. The solution procedure is coupled with the IDS solidification analysis package. Boundary conditions for the strand cooling rely on combination of the generic and plant specific mold, spray, and roll heat transfer coefficients by coping the effects of the casting speed, casting temperature, casting powder, mold oscillation mode, steel grade, water or air-mist spray type, running water, trapped water on the rolls, and radiation. The quality of the slab is related to seven empirical metallurgical criteria. The criteria include the maximum allowed length of liquid pool, recommended length of liquid pool, recommended surface cooling/reheating rate, surface temperature deviation at given axial location, and minimum surface temperature at unbending region. The criteria ensure the caster safety and limit the internal, longitudinal and transversal surface cracking possibility. The problem of finding optimum casting parameter values is stated as a minimization problem based on the analytical form of the cooling criteria evaluated from the steady-state temperature distribution of the strand. To solve the optimization problem, a genetic algorithm is employed. The algorithm explores process parameters settings heuristically by applying the principles of biological evolution. It starts with a random set of solutions and iteratively improves them through selection and variation. Solutions are evaluated by running the heat transfer model of the process and extracting the values of the empirical metallurgical criteria. The case studies for the ACRONI, Slovenia (stainless steel) and Nova Hut, Czech Republic (microalloyed steel) slab casters are presented.

Packaging & Soldering Technologies for Electronic Interconnects: Kinetics of Interfacial Reaction in Solder Joints

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

Program Organizers: Hareesh Mavoori, Bell Laboratories, Murray Hill, NJ 07974 USA; Srini Chada, Motorola, Plantation, FL 33322 USA; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA USA

Tuesday AM — Room: Lincoln D
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Martin Wieser, AlliedSignal, Plated and Discrete Products, Spokane, WA USA; K. N. Subramanian, Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA

8:30 AM Invited

Modeling 3-D Intermetallic Compound Layer Growth between Sn-Pb Solder and Porous Substrate Coatings: *Polly Hopkins*[2]; Paul Thomas Vianco[1]; Kenneth Erickson[1]; [1]Sandia National Laboratories, Matls. Joining Dept., P.O. Box 5800, MS1411, Albuquerque, NM 87185-1411 USA; [2]Sandia National Laboratories, P.O. Box 5800, MS 0834, Albuquerque, NM 87185-0834 USA

Solder joints in hybrid microelectronic circuit (HMC) electronics are formed between the solder alloy and the noble metal thick film conductor that has been printed and fired onto the ceramic. The noble metal conductors is susceptible to solid-state reactions with Sn or other constituents of the solder, forming one or more intermetallic compounds (IMC). Unfortunately, because of the inherent porosity of thick film conductors, IMC growth in conductors cannot be well predicted by simply applying growth kinetics to a quasi-one-dimensional layer geometry. A previous paper summarized initial 2-D modeling results from coupled experimental and computational work. In the present paper, 3-D intermetallic growth in a porous substrate-solder system has been modeled using a 3-D pore structure determined experimentally by digitizing successive 2-D metallographic cross sections. The effects of the two-phase solder field in the 63Sn-37Pb/76Au-21Pt-3Pd solder-substrate system were addressed. Calculations were based on the reaction couple formed between 63Sn-37Pb solder and 76Au-21Pt-3Pd substrates. Physical constants in the model were evaluated from experimental data. Consumption of the thick film was predicted as a function of time and compared with data from independent experiments. 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.

8:50 AM Invited

Long-Term Aging Study on the Solid-State Reactions between 37Pb63Sn, 14Bi43Pb43Sn, 58Bi42Sn Solders and Ni Substrate: B. L. Shiau[1]; C. Chen[1]; C. E. Ho[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Chem. Eng., Chungli, Taiwan

The nickel layer with a thin Au coating is a very common surface finish in electronic components. During reflow, the Au coating reacts with the solder very rapidly and then disappears into the solder, exposing the Ni layer below. The Ni layer will then react with the solder to form intermetallic compound at the interface. In this study, the solid-state reactions between the 37Pb63Sn, 14Bi43Pb43Sn, 58Bi42Sn solders and the Ni substrate were investigated at several temperatures. Aging time as long as five months was used. It was found that in all cases the reaction product was Ni_3Sn_4 with the layered structure. The growth kinetics for the reactions with the three different solders will be presented. Compared to similar reactions with the Cu substrate, the reactions with the Ni substrate are about an order of magnitude slower. The microstructural evolutions in these three systems will be compared and discussed. The activation energies will also be compared.

9:10 AM Invited

Interfacial Phenomena of the Sn-3.5wt%Ag Solder Alloy on the Cu and the Ni Substrates: *Hyuck Mo Lee*[1]; *Won Kyoung Choi*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., Kusung-Dong Yusung-Gu 373-1, Taejon 305-701 Korea

The interfacial phenomena of the Sn-3.5wt%Ag solder alloy on the Cu and the Ni substrates were investigated during soldering and aging. The soldering was performed at 250°C from 30s up to 10hrs followed by aging at 130°C for 100, 400 and 800hrs. The initial formation of IMC after soldering has been found to exert an effect on the growth and morphology of IMC during aging. The morphology of IMC formed in the soldering reaction was closely related with the change of the enthalpy (ΔH_m) which was generated when the IMC formed in the liquid phase of solder.

9:30 AM

Kinetics of Interfacial Microstructure in Diffusion Couples between Solder and Cu/Ni/Pd Metallization: *G. Ghosh*[1]; [1]Northwestern University, Dept. of Mat. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The intermetallic formation at the solder/substrate interface, both during processing and in service, play a key role in determining the mechanical property and reliability of solder joints. Even though Pb-Sn eutectic solder is widely used in electronic industry, due to various reasons there is a growing interest in the use of Pb-free solders. Furthermore, electronic packaging paradigm requires solders having a hierarchy of melting points. As the solder joint size decreases and the service temperature of the advanced products increases, the issue of interfacial reaction kinetics becomes very critical. Metallization schemes using Ni and Pd are becoming increasingly popular in electronic packaging. We will present a comparative study of interfacial reaction kinetics in diffusion couples between Pb-Sn, Ag-Sn, Bi-Sn eutectic solders and Cu/Ni/Pd metallization scheme. The evolution of microstructures due to both liquid- and solid-state reactions will be presented. The products of interfacial reaction are characterized by SEM, TEM, and AEM. The interfacial microstructures will be discussed in terms of diffusion paths using the calculated isothermal sections of the relevant ternary system.

9:50 AM

Solder-Metallization Interdiffusion in Microelectronic Interconnects: *Eric J. Cotts*[1]; A. Zribi[1]; R. R. Chromik[1]; R. Presthus[1]; J. Clum[1]; K. Teed[1]; L. Zavalij[1]; J. Tova[1]; [1]Binghamton University, Physics Dept., P.O. Box 6016, Vestal Parkway East, Binghamton, NY 13902-6016 USA

The reliability of solder joints is in large part determined by the nature of the intermetallic compounds which form at the solder/metal interface, their stoichiometry and geometry. While dissolution rates of eutectic Pb-Sn solder on Cu substrates are fairly well known, the solderability, and integrity, of different solder/substrate combinations has become an issue. For instance, the increased use of Pd lead finishes, or the use of electroless Ni metallizations, or the increased interest in Pb free solders, creates the need for an increase in the scope of characterization of solder/metal systems. We report on our study of the rates of intermetallic compound formation in solder-metallization systems, and the mechanical integrity of specific solder-substrate combinations such as (Pd; [Ni-P]; Pd/[Ni-P], Au/[Ni-P], Au/[Ni-P]/Cu) substrates in combination with Sn or [Sn-Pb]. We characterize the reaction mechanisms and constants for new phases (such as intermetallics) during short and long term aging, with a view to understanding failure mechanisms. We carried out tests of reactivity (dissolution rate) between various substrate materials and solder alloys using differential scanning calorimetry (DSC). Reaction products were characterized by x-ray diffraction analysis, optical and electron metallography, and cross-sectional transmission electron microscopy. We focused on the reactions which produce alloys shown in mechanical testing to compromise the integrity of the solder joints. We quantify reactivities and joint integrities as a function of initial substrate metallurgy and microstructure.

10:10 AM Break

10:25 AM Invited

Copper Substrate Dissolution in Eutectic Sn-Ag Solder and Its Effect on Microstructure: S. Chada[2]; *R. A. Fournelle*[1]; W. Laub[3]; D. Shangguan[4]; [1]Marquette University, Matls. Sci. and Eng. Pgm., P.O. Box 1881, Milwaukee, WI 53201 USA; [2]Srini Chada, Motorola, Plantation, FL 33322 USA; [3]Solectron GmbH, Solectronstr. 2, Herrenberg 71083 Germany; [4]Ford Motor Company, Visteon Auto. Sys., 17000 Rotunda Dr., Dearborn, MI 48121 USA

The dissolution of Cu into molten Sn-3.5Ag solder and its effect on microstructure were studied by light microscopy, scanning microscopy and x-ray microanalysis. X-ray microanalysis of the average Cu content of samples soldered under various conditions showed that the amount of Cu dissolved during soldering increased with increasing soldering temperature and time and that the rate of dissolution could be described by a Nernst-Brunner equation. Microstructurally it was found that the volume fractions of primary Sn dencrites and eta phase dendrites increase with increasing soldering temperature and time. The microstructural changes can be explained using Sn-Ag-Cu phase equilibria data. A numerical method was developed for calculating the amount of Cu dissolved under non-isothermal conditions, which describes dissolution reasonably well.

10:45 AM

The Growth of Cu-Sn Inter-Metallics in Soldered Cu and the Influence of Ag Content: *Ker-Chang Hsieh*[1]; Hen-So Chang[1]; [1]National Sun Yat-sen University, Instit. of Matls. Sci. and Eng., Kaohsiung, Taiwan

Pure copper soldered with Pb-Sn alloys and Pb-Sn-Ag alloys stored at 150°C for long period of time. The growth of Cu_3Sn and Cu_6Sn_5 inter-metallic phases are examined under SEM and EPMA after holding at 4, 9, 16 and 25 days respectively. The solder alloys include six Pb-Sn binary alloys and nine Pb-Sn-Ag ternary alloys. The intermetallic growth rate constant are expressed as function of alloy compositions, D1/2 = f(Pb wt.%). The influence of Ag are examined a similar way in comparison the inter-metallic growth rate. Ag_3Sn intermetallic phase formed and growth rate decreased. The contents of this research include, 1. sample preparation method; 2. solder/Cu reaction layer microstructure; 3. growth rate evaluation.

11:05 AM

A Kinetic Study of the IMC Growth at the Interface of Ni-Containing UBM's and Eutectic Pb/Sn Solder: *Peng Su*[1]; Tia M. Korhonen[1]; Matt A. Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, Dept. of Matls. Sci. and Eng., 356 Bard Hall, Ithaca, NY 14853 USA

Ni-containing underbump metallizations (UBM's) have been proved to be able to effectively reduce the growth rate of intermetallic compounds (IMC) growth rate at the solder/UBM interface. This effect correspondly reduces the consumption of the wetting metals in the UBM during the reflow. The introduction of Ni into the conventional Cu/Sn system, however, brings extra complexity to the kinetics of the original intermetallic formation reaction. In this research, the UBM/solder reaction was studied with alloy foils of various Ni/Cu ratio and molten solder bath. EDX results of the element distribution at the interface revealed that different Ni/Cu ratios demonstrated different diffusion behaviors, which in turn affected the morphology of the intermetallic as well as the growth rate. A kinetic model of the consumption of the Cu/Ni during the soldering reaction is proposed, and the theoretical calculation is compared with the experimental data.

11:25 AM Invited

Interfacial Reactions in In-Sn/Ni Couples and Phase Equilibria of In-Sn-Ni System: *Sinn-Wen Chen*[1]; Ching-Yu Huang[1]; [1]National Tsing-Hua University, Dept. Chem. Eng., Hsin-Chu 30043 Taiwan

In-Sn alloys are promising lead-free solders. Their reactions with Ni substrate and the phase equilibria of the In-Sn-Ni ternary systems are investigated in this study. In-Sn-Ni alloys of various compositions are prepared and annealed at 160° and 240°C. The phases formed in the samples are analyzed by using metallography and powder XRD. The stable binary compounds are β-In_3Sn, η, Ni_3In, Ni_2In, Ni_3In_9, NiIn, Ni_2In_3, $Ni_{28}In_{72}$, Ni_3Sn, Ni_3Sn_2 and Ni_3Sn_4. In-49.1 wt.%Sn/Ni, In-60 wt.%Sn/Ni, and In-80 wt.%Sn/Ni couples reacted at 160° and 240°C are examined. Although there are many intermetallic compounds in the Sn-In-Ni systems and the phase equilibria relationships are complicated, there is only one compound formed in all the (Sn-In) alloys/Ni couples reacted at both 160° and 240°C up to 144 hours.

11:45 AM

Interfacial Reactions in Zn/Ni and Bi/Ni Couples: *Chih-Ming Chen*[1]; Sinn-Wen Chen[1]; [1]National Tsing-Hua University, Dept. Chem. Eng., Hsin-Chu 30043 Taiwan

β_1-NiZn, η-$Ni_{15}Zn_{21}$ and δ-$NiZn_8$ phases, formed at the interfaces of Zn/Ni couples reacted at both 150° and 200°C. The thickness of β_1 and η phases increased with longer reaction time, while that of the δ phase reached a maximum of about 6 μm. Only one intermetallic, $NiBi_3$ phase, was formed in the Bi/Ni couples reacted at 150° and 200°C. The $NiBi_3$ phase grew with time as well. The NiBi phase which is also a stable binary Ni-Bi intermetallic at both 150° and 200°C showed no sign of existence in the Bi/Ni couples reacted up to 144 hours. In addition to these reaction experiments mentioned above, an electric current of 300 A/cm^2 density was passed through the reaction couples to investigate the electromigration effect upon interfacial reactions of the two systems. The results are identical to those without the passage of electric current, which indicate that electromigration effects of 300 A/cm^2 electric density upon interfacial reactions are insignificant in the Zn/Ni and Bi/Ni systems at 150° and 200°C.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Pressure Technology Applications in the Hydrometallurgy of Nickel

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

Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Tuesday AM — Room: Lincoln C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Nic Hazen, Hazen Research Inc., Golden, CO 80403 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

8:30 AM

Pressure Leaching of Nickel Sulphide Concentrates: *Roman M. Berezowsky*[1]; [1]Dynatec Corporation, Metallu. Tech. Div., 8301-113 St., Fort Saskatchewan, Alberta T8L4K7 Canada

Potential advantages usually cited for the direct pressure leaching of nickel sulphide concentrates, over the traditional pyrometallurgically based processes, include the ability to treat lower grade and/or higher impurity (arsenic, magnesium) feeds, higher cobalt recoveries, and production of elemental sulphur, rather than sulphur dioxide or acid. To date, however, the commercial application of direct hydrometallurgical treatment has been limited to two applications of Sherritt's ammoniacal oxidative pressure leach based process, and to two rather specific applications based on sulphuric acid oxidative pressure leaching. The past decade has witnessed a strong resurgence in interest in the direct pressure leaching of nickel concentrates spurred, in part, by advances and the wider acceptance of solvent extraction for metals separation and recovery, and, the use of ultrafine milling and/or the use of chloride in an acid sulphate based system to promote the leach kinetic of the metals and the yields of elemental sulphur. This paper provides, a discussion on leach chemistry, options on metals recovery from solution, and a review of the existing commercial operations, as well as some of the most promising recently promoted processes.

9:00 AM

Nickel Pressure Leach Process at Outokumpu Harjavalta Metals Oy: *Esa Lindell*[1]; Kari Knuutila[1]; Stig-Erik Hultholm[2]; [1]Outokumpu Harjavalta Metals Oy, Harjavalta Finland; [2]Outokumpu Research Oy, Pori, Finland

This paper deals with the pressure leach of the nickel mattes at Outokumpu Harjavalta Metals Oy. The overall nickel process is also shortly discussed. The nickel plant was modernized and expanded in 1995 which led to a production increase from 17 000 t/a to 40 000 t/a in 1999. Before the expansion only cathode nickel was produced. The additional capacity is nickel powder and briquettes produced in the new hydrogen reduction plant. Ammonium-sulphate is obtained as a by-product. There is also an optional capacity to produce cobalt powder via hydrogen reduction. In the nickel smelter where the Outokumpu DON process is utilized two separate granulated products are produced: flash smelting furnace (FSF) matte and electric furnace (EF) matte. Consequently the nickel refinery has two separate leach circuits one for each type of matte. The FSF matte leach circuit consists of two counter current atmospheric leach stages and two pressure leach stages both in counter current arrangement. The EF matte leach circuit consists of an atmospheric and a pressure leaching stage. All reaction vessels and autoclaves are designed by Outokumpu. Copper in the mattes is separated as a sulphide precipitate rich PGMs which is directed to the copper smelter whereas iron is removed as a hematite residue from the EF pressure leach stage. From the first atmospheric leach stage of the FSF circuit neutral, iron and copper free nickel sulphate solution is obtained. The nickel sulphate solution is further purified in a solvent extraction process where cobalt and other minor impurities are removed. The purified nickel stream is then fed to the electrowinning and to the hydrogen reduction plant. The pressure leach process has been in stable operation since the start-up. Furthermore, the nickel process has reached its design production capacity according to the schedule.

9:30 AM

The Viscous Behavior of Aqueous Slurries of Goethite and Limonitic Laterite: Characterization for Design and Analysis of Tubular Transport and Heating Systems: *Brian C. Blakey*[1]; D. F. James[2]; M. Kawaji[3]; E. Krause[1]; [1]Inco-ITSL, 2060 Flavelle Blvd., Mississauga, Ontario L5K1Z9 Canada; [2]University of Toronto, Dept. of Mech. and Indust. Eng., Toronto Canada; [3]University of Toronto, Dept. of Chem. Eng. and Appl. Chem., Toronto, Canada

Unlike most hydrometallurgical slurries, those containing significant concentrations of goethite are typically highly viscous and shear-thinning, even at solid volume fractions below 5%. The viscosity may also depend upon time. These characteristics must be taken into consideration in slurry transport and heating. The technical literature is replete with apparent contradictions regarding the viscous behavior of these slurries, which may be due, in part, to variations in viscosity-measuring techniques. We present techniques which yield consistent results suitable for use in process design. A striking degree of time-dependent behavior is demonstrated for a laterite slurry. Reliable methods to design and analyze tubular transport and heating systems for time-independent slurries are summarized. Rules of thumb are then offered to account for the effects of time-dependency.

10:00 AM Break

10:15 AM

Application of Hpal at the Syerston Laterite Project: *Geoff Motteram*[1]; [1]Black Range Minerals NL, Syerston Nickel Cobalt Project, Level 10, 190 St. Georges Tce, Perth, Western Australia 6000

The Syerston Nickel Cobalt Project in Central New South Wales is unique amongst the laterite projects being developed in Australia and internationally. The unique nature ensues from the laterite enrichment occurring over an Alaskan style ultramafic intrusion with dunite core. Characteristics that result include a goethite host mineralisation with low magnesium content and rapid extraction rates in High Pressure Acid Leach, along with low clay content and minimal viscosity problems. The base dunite mineralisation exhibits high cobalt to nickel ratios with associated high manganese content. It also results in significant metallic platinum content with associated chrome, plus relatively high scandium content within the goethite ore. The desire to maximise cobalt recovery in the presence of high manganese and chrome influence selection the metal recovery circuit. Whereas the potential to recover refractory platinum from the leach residues and scandium from circuit water introduces further circuit considerations.

10:45 AM

Nickel and Cobalt Recovery from Madagascar Laterite: *Eddie Chou*[1]; Phil Rooke[1]; Alan Williams[1]; Jerry Hanks[2]; G. Houlachi[3]; [1]Hazen Research Inc., 4601 Indiana St., Golden, CO 80403 USA; [2]Phelps Dodge Exploration; [3]Noranda Technology Center, Pointe Claire, Canada

Phelps Dodge, Inc. (PD) is currently developing a nickel/cobalt deposit in Madagascar. Hazen Research, Inc. provides technical assistance to PD to investigate recovery processes fro nickel and cobalt. Various process options were evaluated in laboratory experiments and pilot plant operations. The process selected for the feasibility study consists of feed preparation, high-pressure acid leach, countercurrent decantation wash, sulfide precipitation, sulfide dissolution, solvent extraction, ion exchange, and electrowinning. Among these unit processes, high-pressure acid leach, sulfide precipitation, and sulfide dissolution are applications of pressure technologies. Process development work and design criteria for commercial operations of these unit processes are presented in this paper. Concerns related to scaling in the high-pressure acid leach and the sulfide precipitation processes are discussed. Corrosion rates of various metal coupons under these environments were analyzed.

11:15 AM

Bulong Nickel Operations: A Review of the Commissioning and Early Operations Phases in the High Pressure Acid Leach Plant: *G. L. Frampton*[1]; G. J. McCunn[1]; [1]Bulong Nickel Operation, Tech. Svcs., P.O. Box 10391, Kalgoorlie, WA 6430

The Bulong Nickel Operation is a mining and metallurgical complex located approximately 30km east of Kalgoorlie in Western Australia. The process uses high-pressure acid leaching, solvent extraction and electrowinning to produce nickel and cobalt cathode. The projected ore-processing rate is up to 600,000 tonnes per year, with target production levels of approximately 9000 tonnes per year of nickel metal and 640 tonnes per year of cobalt metal in Phase 1. As expected from a complex, integrated facility consisting of mining, leaching and refining operations, the commissioning and early operating period during the first ten months of 1999 has provided numerous challenges to the process and engineering disciplines. The majority of these challenges have either been successfully addressed already, or are close to being resolved. This paper has been written with the intention of reviewing the performance of the High Pressure Acid Leach Plant during this period. Major issues influencing plant throughput rates, plant availabilities and plant recoveries have been highlighted and discussed. An attempt has been made to discuss these factors using a multidisciplinary approach that examines a number of significant chemistry, process and engineering aspects.

Process Synthesis and Modeling for the Production & Processing of Titanium & Its Alloys: Session I

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, Titanium Committee, Shaping and Forming Committee

Program Organizers: James A. Hall, Oremet-Wah Chang, Albany, OR 97321 USA; F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

Tuesday AM — Room: Knoxville B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: James Hall, Oremet-Wah Chang, Albany, OR 97321 USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

8:30 AM
Modeling of Open Die Forging for Ti-6Al-4V: *D. J. Li*[1]; K. O. Yu[1]; P. A. Russo[1]; J. M. Hjelm[1]; G. W. Kuhlman[2]; [1]RMI Titanium Company, 1000 Warren Ave., Niles, OH 44446 USA; [2]Consultant

Forging is used to refine coarse cast structure, and to develop fine equiaxed alpha for Ti-6Al-4V. It is more difficult to break alpha platelets in the center of large section forgings compared to the edge. This was hypothesized to be the result of lower strain at the center. However, 2.5D deformation modeling clearly shows the center already has the highest effective strain. Based on this fact, it is speculated that the above-mentioned problem is a result of the thermal history at the center rather than strain history. To solve this problem, one approach is to impart more strain into center. A few effective ways have been demonstrated by the deformation modeling. Another approach is to lower the critical strain required for recrystallization of alpha platelets by reducing alpha platelet thickness during beta quenching. The effect of billet section size as well as forging temperature on alpha platelet thickness after water quenching has been studied by thermal modeling. This information has been used to predict production processes that will yield fine grain alpha through the section thickness.

8:55 AM
Application of Finite Element Analysis to the Primary Conversion of Titanium Alloys: *Vasisht Venkatesh*[1]; S. P. Fox[1]; [1]TIMET Technical Laboratories, 8000 W. Lake Mead Dr., Henderson, NV 89015 USA

In order to develop accurate finite element analysis (FEA) techniques to model the primary processing of titanium alloys such as TIMETALâ-17 (Ti-5Al-2Sn-2Zr-4Cr-4Mo), a forging alloy for gas turbine engines, appropriate experiments to generate material properties and validation trials need to be conducted. Utilization of material flow behavior corresponding to specific microstructures that are relevant to the conversion process being modeled is discussed. Appropriate small scale tests for the purposes of FEA model validation and mechanical property database generation are also discussed. Forge validation results of TIMETALâ-17 (Ti-17) using DEFORM, a finite-element analysis software code, showed good agreement with the experimentally observed behavior.

9:20 AM
Redesign of Furnace Heating Practice for 34" Diameter Ti-6-4 Ingots: *Ramesh S. Minisandram*[1]; Robert Mark Davis[1]; Robin M. Forbes Jones[1]; Robert J. McHugh[1]; John W. Pridgeon[1]; [1]Allvac, Allegheny Teledyne Co., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28110 USA

In response to a need for increased furnace capacity and reduced process cycle times, a new heating practice for 34" diameter Ti-6-4 ingots is now being implemented. The new practice was designed based on a wide array of numerical simulations of the ingot heating practice coupled with production-scale experimental trials using nominal 17,000 lb. ingots instrumented with sub-surface thermocouples. The new practice is expected to reduce furnace heating time for ingot product by up to 20%. The practical hurdles that were overcome in the design process will be discussed along with the experimental details.

9:45 AM Break

10:00 AM
Steady State Model for the Radial Forging Process: *Ramesh S. Minisandram*[1]; Robin M. Forbes Jones[1]; Erik G. Thompson[2]; [1]Allvac, Rsch. & Dev., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28110 USA; [2]Colorado State University, Civil Eng., Fort Collins, CO 80523 USA

The radial forging process is a complex process that involves a number of repetitive steps. Radial forging machines, depending upon the type, can operate at a few hundred strokes per minute. The large number of strokes involved make it prohibitive to simulate the process as a transient (stroke-by-stroke) analysis. Allvac's radial forging model makes use of a unique steady state approach that allows for rapid turnaround of simulation results, making it a handy tool that is used on a routine basis in addressing production issues. Details of the model are presented along with a few examples that illustrate model verification and its application in addressing manufacturing problems.

10:25 AM
Modeling Texture Evolution in Titanium Rolling: *Paul Richard Dawson*[1]; Nathan Barton[1]; [1]Cornell University, 196 Rhodes Hall, Ithaca, NY 14853 USA

Mechanical properties of titanium and its alloys are strongly influenced by the crystallographic texture imparted by processing. Anisotropy is readily apparent in the strength of rolled products as well in the formability. This presentation will focus on the simulation of texture evolution during rolling, both for commercial purity titanium and for a two-phase alloy (Ti-6Al-4V). The modeling of plastic flow of titanium is made difficult by the strong yield anisotropy of the hexagonal close packed phase, which arises from the relative difficulty in activating slip modes associated with c-axis extension or compression. We will compare results obtained using several modeling assumptions (upper bound, lower bound, and constrained hybrid) within a finite element formulation for flat rolling which accounts for through-thickness variations in the straining and for texture evolution. We will discuss additional modeling results performed on aggregates of grains in which every grain is individually resolved with finite elements and subjected to an idealized rolling history (plane strain compression). These results quantify the degree to which the strong single crystal anisotropy influences the heterogeneity of deformation over the volume of the aggregate, and provide a means to evaluate simpler assumptions that link the micro and macro scales (again, upper bound, lower bound and constrained hybrid models). We will present results showing the influence of texture on the formability as determined by r-values, and conclude with presentation of detailed analyses of the strain partitioning between the hexagonal close packed and body centered cubic phases.

10:50 AM
Processing of Titanium Cylinders by Roll Forming Techniques: *David U. Furrer*[1]; Robert Niemi[1]; [1]Ladish Company Inc., P.O. Box 8902, Cudahy, WI 53110-8902 USA

Titanium is an important material utilized in numerous aerospace and non-aerospace applications. Components produced from titanium are often in cylindrical geometries for casings, tubes and other uses. Seamless processing of cylindrical structures have shown many advantages, and as such, methods to produce these types of components have been developed and optimized for titanium material. Roll forming is a unique, incremental extrusion process by which net or near-net components can be produced. Often conducted at room temperature, the dimensional tolerance capability can approach machining processes. Commercially pure titanium can be readily processed by roll forming techniques, while alloyed titanium materials require special processing consideration. This process is capable of producing a wide range of cylinder diameters, heights and wall thicknesses. A combination of manufacturing process design and material application can result in high performance and economical titanium components.

Surface Engineering in Materials Science I: Coating/Films Characterization (C)-I

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; John Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA

Tuesday AM Room: Canal B
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, AMPAC & MMAE, Orlando, FL 32816 USA; Yip Wah Chung, Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

8:30 AM
Microstructure of TiO2-x Thin Films Reactively Sputtered by Dc Magnetron: *K. Zakrzewska*[1]; *A. Brudnik*[1]; A. Kusior[1]; M. Radecka[1]; A. Kowal[2]; [1]University of Mining and Metallurgy, Al. Mickiewicza 30, Kraków 30-059 Poland; [2]Polish Academy of Sciences, Instit. of Catalysis and Surf. Chem., Ul. Niezapominajek 1, Kraków 30-239 Poland

GID (Grazing Incidence Diffraction) and AFM (Atomic Force Microscopy) were used to study the progressive structure evolution in thin films of TiO2-x. Thin films were deposited by the reactive magnetron sputtering with the controlled intensity of Ti emission line (l=500 nm). Variation in this parameter reflected a change in the oxidation state of the sputtered metallic target and as a consequence affected the growth of polymorphic forms of titanium dioxide: anatase and rutile. The anatase was found to dominate the structure of films obtained from the oxidized target. The ratio of rutile/anatase phases was found to increase with the increasing intensity of Ti emission line, i.e. decreasing target coverage by oxides. AFM images gave evidence of two types of grains: those of smaller size constituting the base of the sample and large ones formed at the film surface. Good correlation was established between the relative distribution of these species and the structure evolution observed in X-ray diffraction experiments performed at different angles of grazing incidence.

8:50 AM
Determination of the Structure and Chemistry of the Thermally Grown Oxide Scale in Thermal Barrier Coatings Containing a Platinum-Nickel-Aluminide Bondcoat: *Michael R. Brickey*[1]; J. L. Lee[1]; [1]Purdue University, Sch. of Matls. Eng., 1289 MSEE Bldg., Rm. 150, West Lafayette, IN 47907-1289 USA

A chief influence governing the reliability of modern gas turbine engine thermal barrier coatings (TBC's) is the adhesion of a thermally grown oxide (TGO) scale to both the bondcoat and the insulative ceramic topcoat layers. Factors affecting scale adhesion include TGO and bondcoat chemistry in addition to growth and thermal stresses developed during engine service. Several advanced TBCs incorporate an electron beam-physical vapor deposited (EB-PVD) yttria-partially-stabilized zirconia (YPSZ) ceramic topcoat and a platinum-nickel-aluminide (Pt-Ni-Al) bondcoat on nickel based superalloy engine components. It is known that Pt-Ni-Al bondcoats display excellent TGO scale adhesion relative to traditional bondcoat chemistries, but limited microstructural and chemical data exist in the literature for scales formed by this TBC system. Since the TGO is typically 1 to 2 microns thick, analysis of the scale is well suited to transmission electron microscopy (TEM). Conventional TEM, scanning TEM, windowless energy dispersive X-ray spectroscopy, and electron energy loss spectroscopy were among the techniques used to reveal the microstructural and chemical characteristics of the scale. It was found that the scale consists of hexagonal aluminum oxide (-Al2O3) in both as-deposited and in thermally cycled specimens; no metastable Al2O3 allotropes were observed. In the as-deposited specimen, the scale's grains were predominantly columnar. Zr-rich particles were observed to occur both intra- and intergranularly throughout the scale thickness, and their concentration increased upon approaching the YPSZ. Thermally cycled specimens had a morphologically banded scale structure: one band of grains adjacent to the YPSZ had an equiaxed morphology and contained Zr-rich particles; a second band of grains adjacent to the bondcoat had a columnar morphology with axes oriented normal to the TGO/bondcoat interface. It is believed that this is the first experimental observation of a morphologically banded scale containing Zr-rich dispersoids in a TBC system having a Pt-Ni-Al bondcoat. For specimens undergoing up to 10 thermal cycles, both the equiaxed band and the columnar band increased in thickness with increased thermal exposure. However, the proportion of the scale comprised by the equiaxed band tended to decrease with greater cycling. The columnar grains of the as-coated and thermally cycled specimens differed in that the latter contained no Zr-rich particles and were comparatively coarse in size. Porosity and cracks were associated with the irregular interface between the bands, but no porosity was observed at the YPSZ/TGO or TGO/bondcoat interfaces. Since these defects can act as a weak link promoting YPSZ spallation, it is expected that elimination of the banded structure is key to improving TBC reliability. It is proposed that formation of the equiaxed grains and the banded morphology is associated with the Zr-rich dispersoids.

9:10 AM Invited
Synthesis and Characterization of Ultrathin Overcoats: *Yip-Wah Chung*[1]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

The magnetic spacing budget at 100 Gb/in^2 storage density provides only 2nm for the protective overcoat. Such an ultrathin overcoat must have the necessary wear resistance, be smooth enough to allow low flying heights and be sufficiently defect-free to provide corrosion protection for the underlying magnetic media layer. One also requires the overcoat to be compatible with the lubricant and materials in contact. It is desirable that the technique to synthesize such a coating be compatible with existing manufacturing practice. In this paper, we present preliminary results on the synthesis and characterization of three promising candidate coatings: (i) amorphous CNx; (ii) crystalline TiB2 and (iii) hydrogen-free amorphous carbon. Advantages and disadvantages of each coating material and the associated synthesis technique will be discussed.

9:35 AM
Characterization of Oxide-Metal and Grain Boundary Interfaces in Aluminide Bond Coats: *Stacie LeSure*[1]; J. Allen Haynes[1]; Ian G. Wright[1]; Bruce A. Pint[1]; [1]Oak Ridge National Laboratory, Met. & Cer. Div./Cer. Surf. Sys. Grp., P.O. Box 2008, Oak Ridge, TN 37831-6063 USA

In an effort to increase gas turbine efficiency, thermal barrier coating systems (TBC's) are used to protect superalloy components in gas turbine engines. Most TBC systems consist of a superalloy substrate, an oxidation-resistant bond coat and a ceramic top coat. This study will focus on the formation and adhesion of a protective alumina scale at the bond coat- top coat interface. Adhesion of the scale, which is the weak link in TBC durability, is influenced by a number of factors, including bond coat platinum content. Thus, the purpose of this investigation is to examine the role of platinum in improving scale adherence. Analytical Transmission Electron Microscopy (ATEM) will be used to examine the alumina/bond coat interface in bond coats with and without platinum additions. Grain boundary segregation in the bond coat will also be investigated by ATEM. Field emission gun scanning electron microscopy (FEG-SEM) will be used to characterize the microstructures of the bond coats and scales. Additionally, segregation phenomena occurring at the bond coat surface will be studied using hot-stage Auger Electron Spectroscopy (AES).

9:55 AM

Microstructures of TiSi2 Coatings Obtained by Plasma Spray in Both Air and Low Pressure Conditions: *G. Li*[1]; S. Uematsu[1]; [1]Ship Research Institute, Matls. and Process. Div., Shinkawa 6-38-1, Mitaka, Tokyo 181-0004 Japan

Effect of plasma spray processing parameter on the microstructure of coatings was reported in this article. TiSi2 particles (<44Êm) were sprayed, in both air and low pressure conditions, respectively. All experiments were carried out with a conventional DC arc plasma spray device. The phases present in coatings were analyzed by X-ray diffraction (XRD). Microstructures were observed by scanning electron microscopy (SEM). Chemical compositions were examined by electron probe microanalysis (EPMA). The XRD patterns showed that, sprayed in air condition, some starting TiSi2 particles were decomposed into Ti5Si3, which was predominant in coating. In addition, oxide products of TiSi2 were also found. In contrast, TiSi2 phase was a major constitute in coating obtained in low pressure chamber. SEM observation revealed that the features of coatings were characterized by splashing in both cases. It implied that most of the TiSi2 particles were very well melted. The phenomena were discussed. This investigation provided a step further toward the understanding and optimizing of the TiSi2 particles spraying process.

10:15 AM Break

10:30 AM

Grain Structure Development in Plasma Sprayed Coatings: Experiment and Modeling: *Guo-Xiang Wang*[1]; Sanjay Sampath[2]; [1]The University of Akron, Dept. of Mech. Eng., Auburn Sci. & Eng. Ctr. 106B, Akron, OH 44325-3903 USA; [2]State University of New York, Dept. of Matls. Sci. & Eng., Ctr. for the Thermal Spray Rsch., Stony Brook, NY 11794-2275 USA

Plasma-sprayed coatings are made of small splats formed when molten particles impact at a high speed on a substrate. The quality and the properties of a coating therefore strongly depend on the phases and microstructures of each splat. A variety of grain structures have been found in plasma-sprayed coatings, depending on the coating material, substrate material, and process condition. This paper presents some recent experimental observations of grain structures in single splats plasma-sprayed on different substrates. The coating materials used include molybdenum, alumina, and partially yttria-stabilized zirconia, and the substrate materials are steel and glass. It is found that, in most of cases, the ceramic coatings show a columnar grain structure with very high grain density, although occasionally some equiaxed grains were observed. Fine columnar grains were formed in the molybdenum splats sprayed on a steel substrate, but grains with significant large sizes were seen in the Mo splats sprayed on a smooth glass surface. A heat transfer model is developed to analyze the thermal characteristics of splat solidification. The classical nucleation theory is incorporated in the heat transfer model to predict the formation of crystalline nuclei and the melt undercooling at nucleation. Detailed heat transfer analyses are performed using the model to study the effect of coating material, substrate material, and the substrate surface condition on the formation of columnar or equiaxed grains. Solidification mechanisms in a single splat under different conditions are also investigated.

10:50 AM

Relation between Deposition and Recrystallization Textures of Copper and Chromium Electrodeposits: *Dong Nyung Lee*[1]; Joon Hwan Choi[1]; Soo Young Kang[1]; [1]Seoul National University, Matls. Sci. and Eng., Shinrim-dong Kwanak-gu, Seoul 151-742 Korea

The texture of electrodeposits varies with electrolysis conditions and may change after recrystallization. The <100>, <111>, and <110> textures of copper electrodeposits obtained from copper sulfate baths changed to the <100>, <100>, and <SQR(3)10> textures, respectively, after recrystallization. The textures of chromium electrodeposits obtained from the standard Sargent bath remained unchanged on recrystallization. The results are in agreement with the prediction of the strain energy release maximization model. In the model the recrystallized grains orient themselves so that their minimum elastic modulus direction can be parallel to the absolute maximum internal stress direction due to dislocations in the non-recrystallized grains.

11:10 AM

Effects of Pre-Treatments and Interlayers on the Nucleation and Growth of Diamond Coatings on Titanium Substrate: *Yongqing Fu*[1]; Nee Lam Loh[1]; Bibo Yan[1]; Chang Q. Sun[2]; Peter Hing[2]; [1]Nanyang Technological University, Matls. Lab., Schl. of Mech. & Prod. Eng., 639798 Singapore; [2]Nanyang Technological University, Schl. of Appl. Sci., 639798 Singapore

During diamond deposition on titanium substrate, there exist two processes: (1) Diffusion of hydrogen into titanium substrate and the formation of hydride thus degrading the mechanical properties of substrate. (2) Competition among the rapid diffusion of carbon atoms into substrate, the formation of carbide and the nucleation of diamond crystals (thus decreasing the nucleation and growth rate of diamond coating). To increase the diamond nucleation rate and prevent the rapid diffusion of hydrogen and carbon into the substrate, different surface treatments and interlayers were studied in this paper. Results showed that after polishing with diamond pastes and ultrasonically pre-treatment in diamond suspensions, the nuclei density of diamond crystals increased significantly. However, it could not prevent the diffusion of hydrogen into the substrate. Pre-etching of titanium substrate using hydrogen plasma for a short time significantly increased the nuclei density of diamond crystals. Results showed that on TiN interlayer, there was no significant improvement in diamond nucleation and growth, and the deposited diamond coatings showed a poor adherence. There were two mechanisms for diamond nucleation on diamond-like-carbon (DLC) interlayer. DLC film was etched by hydrogen plasma and changed to diamond crystals, and at the same time, new diamond crystals were formed on DLC interlayer in which DLC acted as the precursor for diamond nucleation. However, the so-formed diamond coating had poor adhesion strength. Plasma nitrided layer could prevent the rapid diffusion of hydrogen and carbon into titanium substrate, but results showed a relatively low nucleation density of diamond crystals and poor adhesion. A graded interlayer combining plasma nitriding followed by plasma carbonitriding was effective in preventing the rapid diffusion of hydrogen and carbon into the substrate and improving the nucleation rate and adhesion of diamond coating.

Ultrafine Grained Materials: Severe Plastic Deformation Processing: I

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; S. L. Semiatin, Wright Laboratory, Materials Directorate, Dayton, OH 45440 USA; C. Suryanarayana, Colorado School of Mines, Department of Metal and Materials Engineering, Golden, CO 80401 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM	Room: Polk A/B
March 14, 2000	Location: Opryland Convention Center

Session Chair: Naresh N. Thadhani, Georgia Institute of Technology, Atlanta, GA 30332-0245 USA

8:30 AM Invited

Factors Influencing the Development of Ultrafine Grained Materials through Severe Plastic Deformation: Minoru Furukawa[2]; Zenji Horita[3]; Minoru Nemoto[3]; *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Faculty of Eng., Fukuoka 812-8581 Japan

Equal-Channel Angular (ECA) pressing is a processing method which may be used to achieve substantial grain refinement. Typically, metals subjected to ECA pressing have grain sizes in the submicrometer range. This paper considers the factors influencing the development of a homogeneous microstructure of equiaxed grains separated by high angle grain boundaries, including the pressing speed, the strain introduced on each passage through the die and the effect of specimen rotation between consecutive pressings.

8:55 AM Invited

Formation of Equilibrium Ultrafine Grain Structure in a 6061 Aluminum Alloy: R. Kaibyshev[2]; F. Musin[2]; D. Gromov[2]; *D. R. Lesuer*[1]; [1]Lawrence Livermore National Laboratory, L-342 P.O. Box 808, Livermore, CA 94551 USA; [2]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Ultrafine grain structures have been produced in a 6061 aluminum alloy using ECAE. The average size was 0.4-0.5 μm. A highly non-equilibrium structure was produced with a high density of extrinsic dislocations at grain boundaries which are sources of long-range stress fields. As a result, the alloy demonstrates poor workability and insufficient service properties. Two processing approaches for reducing the internal stress were evaluated. The first approach involved recrystallization annealing which was effective in relaxing internal stresses. However, the relaxation of internal stresses was accompanied by dramatic grain coarsening. Using a recrystallization anneal, microstructures with average grain size less than 1.5 μm could not be produced which were free of internal stresses. In addition, this structure is non-uniform. Secondary recrystallization was observed to occur with the formation of grains which were greater than 10 μm. An attractive alternative for the conversation of a non-equilibrium ultrafine grain structure into an equilibrium one is via plastic deformation. The microstructural evolution during plastic deformation of the 6061 aluminum alloy with an initial submicron structure will be discussed in detail. It was shown by X-ray analysis, TEM examinations and microhardness tests that plastic deformation led to a decrease of internal elastic stresses due to recovery of grain boundary dislocations. Adsorption of grain boundary defects and the formation of a recrystallized structure occurs after high strains and the microhardness decreases. This process was not accompanied by significant grain growth and the crystallite size was essentially stable during plastic deformation. As a result, the formation of an equilibrium submicrocrystalline structure took place. A mechanism of transformation of the non-equilibrium submicrocrystalline structure into a recrystallized one will be considered.

9:20 AM

Microstructural Evolution in an Al-1.7at.%Cu Alloy Deformed by Equal-Channel Angular Pressing: *M. Murayama*[1]; K. Hono[1]; Z. Horita[2]; [1]National Research Institute for Metals, Tsukuba 305-0047 Japan; [2]Kyushu University, Dept. of Matls. Sci. and Eng., Fukuoka 812-8581 Japan

The microstructural evolution during the equal channel angular (ECA) pressing process of an α+θ' two-phase Al-1.7at.%Cu alloy has been studied by transmission electron microscopy (TEM) and energy-filtered TEM. The θ' precipitates are severely deformed after the first pass of ECA pressing that with a strain of e~1. After 8 passes of ECA pressing, most of the θ' precipitates are dissolved and a fine grained (~500nm) supersaturated solid solution is obtained. When the ultrafine grain single phase alloy prepared by ECA pressing is aged for 24 h at 100°C, precipitation of the equilibrium θ phase is observed along the grain boundaries, whereas only GP zones are formed in the undeformed specimen. This suggests that the grain boundaries in the ultrafine grained microstructure provide heterogeneous nucleation sites for the θ precipitates. The supersaturation required for precipitation of the GP zones is lost quickly by diffusion of Cu to the grain boundaries.

9:40 AM

Ultrafine Grain Structure in the Friction-Stir Welding of Aluminum Alloy 2024 at Low Temperatures: *Samuel Benavides*[1]; Ying Li[1]; Lawrence E. Murr[1]; [1]The University of Texas at El Paso, Metallu. and Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

Friction-stir welding involves a solid-state intercalation process facilitated by dynamic recrystallization which accommodates superplastic flow. Adiabatic and frictional heating, a result of high strain and high strain-rate deformation, drive the recrystallization process. In this study we examined the residual grain structures for aluminum alloy 2024 friction-stir welded at starting temperatures which ranged from roughly 30°C to -100°C. The ability to weld any metals or alloys at -100°C is in itself rather extraordinary, especially since the steady-state (recrystallized) grain size appears to be somewhere between 0.5 μm and 0.8 μm. At higher welding temperatures the residual grain size in the weld zone increases to near 10 μm as a result of grain growth. At -100°C the welding produced a very narrow temperature spike of around 240°C, but the residual grain sizes remained in the sub-micron range. In this case the welding was actually performed with the workpiece submerged in liquid nitrogen. Microstructures have been examined in detail using transmission electron microscopy. The weld zone properties were monitored by measuring and comparing residual microhardness profiles through the stir-welded zone and extending into the base metal (workpieces). Research supported in part by a NASA Cooperative Agreement NCC8-137 and a Mr. and Mrs. MacIntosh Murchison Endowment.

10:00 AM

Amorphization of Al-Sm-Based Alloys by Cold-Rolling: *Rainer Johannes Hebert*[1]; [1]University of Wisconsin-Madison, 1500 Engineering Dr. 1217 ERB, Madison, WI 53706 USA

Melt-spinning and splat-quenching as traditional production techniques for amorphous materials often result in the formation of quenched in nuclei and subsequent nanocrystal formation during processing. Intense straining and cold-rolling offer not only the opportunity for the formation of bulk amorphous material but also for a new approach to study the nanostructural evolution during processing. Cold-rolling of Al92Sm8 yielded a partially amorphous sample that revealed a clear glass transition during linear heating in DSC. Modulated DSC reflected a similar Tg for melt-quenched Al92Sm8 thus indicating that amorphous Al92Sm8 is a true glass in contrast to amorphous, short-range ordered structures. A systematic study of Al-Sm based alloys following cold-rolling has been conducted in terms of the dependence of the glass-transition behavior as well as the grain size and microstructure on both the number of rolling cycles and solute concentration. The effect of multicomponent-alloying on the amorphization is accounted for by the influence of selected Ni-additions on the cold-rolling response of Al-Sm alloys. This experience has demonstrated that intense cold-rolling can yield glass/nanocrystal-structures and has the potential for synthesis of bulk nanocrystal or amorphous structures. The support of the ARO (DAAG 55-97-1-0261) is gratefully acknowledged.

10:20 AM Break

10:30 AM Invited

Severe Deformation Based Process for Grain Subdivision and Ultrafine Microstructures: *A. K. Ghosh*[1]; W. Huang[1]; [1]The University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109 USA

Significant interest exists in the materials science community in creating nanocrystalline microstructures in metallic alloys in an effort to obtain improved strength and processability in these alloys. Vapor deposition processes have been used for producing small scale nanostructured samples, but repeated deformation methods, such as 3-axis forging, ECAE (Equal Channel Angular Extrusion), pressurized torsion etc. are found more convenient to create bulk materials with 50 ñ 500 nm size microstructures. Stability of these microstructures to further deformation and to thermal exposure is however a major problem which needs to be addressed in the future years. If the desired product is a sheet, further rolling is required after the severe deformation steps, and this has been found to alter their microstructure. Another issue for process scale-up is die-wall sticking with the moving workpiece, which make scale-up of billet size a nontrivial problem. To minimize these structure-altering aspects of rolling deformation, a surface-shear based deformation technique has been developed and applied to plates and sheets of aluminum alloys to create desired fine-scale microstructures. Initial studies on several Al alloys containing dispersoid particles have been analyzed by optical, and electron microscopy. The role of dispersoids in the structural stability has been studied as a function of annealing temperature and time. This talk will summarize our results to date on this investigation.

10:55 AM Invited

The Influence of Processing Route on the Evolution of Microtexture and Grain Boundary Character during ECA Pressing of Pure Aluminum: *Terry R. McNelley*[1]; Douglas L. Swisher[1]; Zenji Horita[2]; Terry G. Langdon[3]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]Kyushu University, Dept. of Matls. Sci. and Eng., Fukuoka 812-8581 Japan; [3]University of Southern California, Dept. of Matls. Sci. and Mech. Eng., Los Angles, CA 90081-1453 USA

Severe plastic deformation by equal-channel angular (ECA) pressing is a promising approach to achieving extreme grain refinement in aluminum and its alloys. Grain sizes of about 1.0 µm have been reported for pure aluminum processed by repetitive ECA pressing and submicron grain sizes have been attained in aluminum-magnesium alloys. From investigations by transmission electron microscopy the evolution of the deformation microstructure and the roles of recovery and recrystallization during repetitive pressing operations are significantly dependent on the details of the process route. Direct measurement of grain-to-grain misorientation by use of recently developed computer-aided electron backscatter diffraction (EBSD) analysis methods enables the evaluation of microtexture and grain boundary character. These methods have been employed here to examine the evolution of the grain-to-grain misorientation as well as microtexture during repetitive ECA pressing of pure aluminum. Implications to selection of processing conditions will be discussed.

11:20 AM

Characterization of Sub-Micrometer Structures in Heavily Deformed Metals Over the Entire Misorientation Angle Range: *Darcy A. Hughes*[1]; Niels Hansen[2]; [1]Sandia National Laboratories, Ctr. for Matls. and Eng. Sci., P.O. Box 969, M.S. 9403, Livermore, CA 94551-0969 USA; [2]Risø National Laboratory, Matls. Rsch. Dept., P.O.Box 49, Roskilde DK-4000 Denmark

Grain subdivision during deformation results in a distribution of both low and high angle dislocation boundaries surrounding nanometer to micrometer scaled volumes. Key characteristics of these boundaries, including morphology, as well as spacing and misorientation angle distributions, are quantified using high resolution transmission electron microscopy and TEM Kikuchi diffraction as a function of the deformation. It is observed that these boundaries refine the microstructure and that the fraction of high angle boundaries increases with increasing deformation. Different deformation modes and material type can modify the boundary spacing and angle distributions as well as the boundary morphology. A comparison is made between monotonic deformation by rolling or simple shear and more complex paths such as cyclic-extrusion-compression and equal-channel-angle-extrusion. The question is considered of whether these deformation induced high angle boundaries constitute a kind of grain refinement. Part of this work was supported by the Office of Basic Energy Sciences, U.S. DOE under contract No. DE-AC04-94AL85000.

11:40 AM

Ultra-Fine Grained Ferrous and Aluminum Alloys Produced by Accumulative Roll-Bonding: *Nobuhiro Tsuji*[1]; Yoshihiro Saito[1]; Yoshinori Ito[2]; Hiroshi Utsunomiya[1]; Tetsuo Sakai[1]; [1]Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [2]Graduate Student of Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Ultra-grain refining of metallic materials by intense straining has been significantly studied. We have recently developed a novel intense straining process applicable to large structural materials, the Accumulative Roll-Bonding (ARB). In ARB, the rolled material was cut, stacked to be the initial thickness, and rolled again. In order to obtain solid material, the rolling in the ARB is not only a deformation process but also a bonding process (roll-bonding). The ARB was applied to various aluminum alloys, interstitial free steel and low-carbon steel. At various temperatures in the range from room temperature to hot-rolling temperatures, the ARB was successfully carried out. Especially at warm temperatures below recrystallization temperature, the ARBed sheets were filled with ultra-fine grains whose diameter was several hundred nano-meters. Above recrystallization temperature, it was impossible to accumulate strain to obtain ultra-fine grains because of enhanced restoration during reheating. The materials ARBed at ambient temperature did not show ultra-fine grains but complicated deformation structure, however, ultra-fine grains formed in them by low temperature annealing. The formation process of the ultra-fine grains will be discussed. Effect of lubrication in roll-bonding on the properties and the microstructure of the material was also clarified. The ultra-fine grained materials showed very large strength two to four times larger than that of the starting materials at ambient temperature. Interestingly discontinuous yielding appeared even in pure aluminum and IF steel if they had ultra-fine grains. Though the ultra-fine grained materials showed limited ductility (especially uniform elongation) at room temperature, some of them showed low temperature superplasticity at elevated temperature.

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 2

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

Tuesday PM — Room: Memphis A
March 14, 2000 — Location: Opryland Convention Center

Session Chair: C. Patuelli, Universita di Bologna, Dept. di Fisica ed Istituto Nazionale di Fisica della Materia, Bologna I-40127 Italy

2:00 PM
Electrostatic Levitation Processing of Zr-Nb-Ni-Cu-Al Bulk Metallic Glass Forming Alloys: *C. C. Hays*[1]; J. Schroers[1]; U. Geyer[1]; S. Bossuyt[1]; N. Stein[1]; W. L. Johnson[1]; [1]California Institute of Technology, Dept. of Matls. Sci., 138-78, Pasadena, CA 91125 USA

The bulk glass forming ability in the Zr-Nb-Cu-Ni-Al alloy system is well established. One of these bulk metallic glasses, $Zr_{57}Nb_5Cu_{15.4}Ni_{12.6}A$-$l_1 0$ (Vit 106), developed at Caltech exhibits an exceptional glass forming ability (GFA) and has an excellent thermal stability with respect to crystallization. For example, the width of the supercooled liquid region ΔT=(Tx-Tg), as determined from calorimetric measurements is ΔT 70K. This alloy has been investigated by our group in recent electrostatic levitation (ESL) experiments conducted at the NASA Marshall Space Flight Center Electrostatic Levitation facility. Independent ESL experiments on Vit 106 specimens show conclusively that the Time-Temperature-Transformation (TTT) diagram for this alloy is a superposition of high and low temperature branches. The nose of the TTT diagram is located at tnose 1-2 seconds, which prevents vitrification of Vit 106 specimens on free cooling in the ESL. As part of our continuing investigations of alloys in the Zr-Nb-Cu-Ni-Al system, we have identified a neighboring composition, Zr58.5Nb2.8-Cu15.6Ni12.8Al1 0.3, that exhibits a substantially improved GFA compared to Vit 106. This alloys is easily vitrified by standard techniques; e.g., arc melting and melting on a water cooled silver boat apparatus. Using these methods, the critical casting thickness for this composition is near 1cm. The calorimetrically determined ΔT values are as large as ΔT 100K. Differential thermal analysis (DTA) measurements show that the onset of melting for this new alloys is 10 K less than that of Vit 106. When examined in the ESL the new alloys are vitrified by purely radiative cooling. This is the first non-Be containing alloy to be vitrified on free cooling in the ESL. This places the microstructural properties of the ESL processed materials were examined by x-ray diffraction, electron- and optical-microscopy, and thermal analysis methods. These results are used to interpret the complex evolution of the glass forming properties observed in this alloy system.

2:20 PM
Solution Growth of Semiconductor in an Axial Static Magnetic Field: *Yuko Inatomi*[1]; Ayako Kato[1]; Kengo Horiuchi[1]; Takao Maki[2]; Kazuhiko Kuribayashi[1]; [1]The Institute of Space and Astronautical Science, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]Olympus Optical Company Limited, 2951 Ishikawa-chou, Hachiouji, Tokyo 192 Japan

In most of crystal growth experiments of semiconductor under microgravity, the materials were analyzed by static research methods on the ground after the completion of the mission. An in situ observation is considered as a promising methodology during microgravity experiment in order to make full used of the limited experimental opportunities and their resources, because simultaneous observation of the growing interface and the environment can more precisely reveal the influence of supersaturation, temperature and concentration gradients, convection, and surface kinetics on the growth behavior. An in situ observation setup for solution growth process of semiconductor was developed for a high static magnetic field experiment based on near-infrared microscopy. Subsequently the axial magnetic field experiments from 0 to 6 were performed to reveal the influence of strongly-damped fluid flow on the growth surface. As a result, the dissolution and growth rates were successfully obtained using the setup with a high accuracy. The morphological instability of the surface will be discussed in detail.

2:40 PM
The Microgravity Diffusion Experiment for Very Reactive Liquid Metals: *Toshio Itami*[1]; Akitoshi Mizuno[1]; Hirokatsu Aoki[1]; Minoru Kaneko[1]; Tomoharu Fukazawa[1]; Akira Tanji[1]; Yoshito Arai[1]; Kazumasa Goto[1]; Yukiko Yamaura[1]; Natsumi Tateiwa[1]; Sinnichi Yoda[1]; Tadahiko Masaki[1]; Tomihisa Nakamura[1]; Naokiyo Kosikawa[1]; Yukihiro Nakamura[1]; [1]National Space Development Agency of Japan, Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaragi, Japan

The space experiment still has many different features from the laboratory experiments, for example, there is only one chance without the repetition, strictly a requirement for safety, the proceed of long time since sample is delivered, etc. Therefore it is not always easy to perform the microgravity experiments for very reactive materials. In this report description is given for the experimental method for diffusion experiments of liquid Li under microgravity and its application to rocket experiment due to the TR-IA-6-rocket.

3:00 PM
Wetting Phenomena in Immiscible Alloys: *L. J. Little*[1]; J. B. Andrews[1]; [1]University of Alabama, Dept. of Matls. and Mech. Eng., Birmingham, AL 35294 USA

Ground-based processing of binary immiscible alloys leads to sedimentation. In an attempt to obtain dispersed microstructures, early experiments were performed in which immiscible alloys were processed in microgravity. However upon evaluation of the samples from these experiments, it was discovered that a separated structure was obtained, where one phase completely surround the other. One possible cause for this segregation in microgravity-processed immiscible alloys was believed to be perfect wetting. The purpose of this research is to investigate the wetting behavior in immiscible alloys and determine the mechanisms behind the segregation process. A transparent cell assembly will be used in order to permit direct observation of the solidification process. The succinonitrile-glycerol system and the succinonitrile-water system will be utilized in the investigation to evaluate the wetting phenomenon. It is anticipated that this research will give insight into perfect wetting behavior and information concerning its influence on the microstructures obtained from microgravity processing.

3:20 PM Break

3:40 PM
Combustion Synthesis of Porous Glass Ceramic Materials: *A. R. Manerbino*[1]; H. C. Yi[2]; J. Y. Guigne[2]; J. J. Moore[1]; F. D. Schowengerdt[1]; [1]Colorado School of Mines, Dept. of Metallu. and Matls. Eng., Golden, CO 80401 USA; [2]Guigne International Limited, Paradise, Newfoundland A1L 1C1 Canada

Combustion synthesis also know as, Self-Propagating High Temperature Synthesis (SHS), is a processing method which utilizes exothermic reactions to produce advanced materials which could prove to have many applications. This technique is used to produce glasses in the present work. Thermodynamic analysis was carried out to determine the adiabatic temperature (T_{ad}), and actual combustion temperature (T_c), and wave velocity of several chemistries. Microstructural analysis was done using X-ray Diffraction (XRD), light microscopy, and Scanning Electron Microscopy (SEM) to determine the crystalline and glass-forming regions within the samples. Samples produced were porous, typically containing 50-70% porosity. Pores are generated by a number of mechanisms including green density and reactant powders before combustion reaction. This advanced material could be used for a wide variety of applications including filters, bone replacement material, or lightweight high-temperature applications.

4:00 PM
Surface Tension Measurements of Molten Silicon with the Techniques of Free-Fall and Electromagnetic Levitation: *Hideki Minagawa*[1]; Yusuke Goto[1]; Masataka Sasamori[1]; Hideaki Nagai[1]; Masaki Orihashi[1]; Yoshiho Ito[1]; Takashi Tsurue[1]; Yoshinori Nakata[1]; Keiji Kamada[2]; Takeshi Okutani[1]; [1]Hokkaido National Industrial Research Institute, Matl. Div., Agcy. of Industry and Sci. Tech., 2-17 Tsukisamu-Higashi Toyohiraku, Sapporo, Hokkaido prefecture Japan; [2]Japan Space Utilization Promotion Center, 3-30-16, Nishiwaseda, Shinjuku-ku, Tokyo, Japan

The results of surface tension of molten silicon measured by electromagnetic levitator have been compared with these by free-fall experiment. The comparable results of surface tension of molten silicon have been measured, which were demonstrated by electromagnetic (E-M) levitation and free-fall. The measurements by E-M levitator have been performed under normal gravity condition, while measurements by free-fall system have been carried out at the 1.2 s HNIRI (Hokkaido National Industrial Research Institute) drop tower. The values of surface tension derived by the frequency of surface oscillation, which had been measured by E-M levitation and free-fall, were different from each other because of the effect of the perturbation of E-M field for the measurement of surface tension.

4:20 PM
Undercooling Comparison of Pure Cobalt Processed in Crucibles and the MSFC Drop Tube: *Mike B. Robinson*[1]; Delin Li[1]; Tom J. Rathz[2]; [1]MSFC/NASA, Science Directorate 47, Huntsville, AL 35812 USA; [2]UAH, SD47, MSFC/NASA, Huntsville, AL 35812 USA

A large number of undercooling cycles was performed on a single Co drop and many separate samples using crucibles and the 105-meter drop tube. The effects of sample size, material purity, crucible, and flux agent on undercooling were investigated. For fluxing experiments, average and maximum undercooling was observed to be insensitive to sample diameter in the 1 to 10 mm range and material purity between 99.5 and 99.999%. Among the total of 1,155 nucleation events recorded for the same 4-millimeter sample, 90% undercooling values are clustered from 300 to 330 K. Comparably large undercoolings have been achieved in the low gravity, containerless environment of the long drop tube. Undercooling data are analyzed within the framework of classical nucleation theory coupled with statistical approach. The average grain size was decreased from 500 to 30 mm when the undercooling was increased from 20 to 320 K, demonstrating the grain refinement in undercooled pure cobalt.

4:40 PM
Solidification Processing of Immiscible Liquids in the Presence of Applied Ultrasonic Energy: *Shinwoo Kim*[1]; *Richard N. Grugel*[2]; [1]Hoseo University, Dept. of Matls. Sci. and Eng., Chung-Nam Korea; [2]Universities Space Research Association, Marshall Space Flight Ctr., MS-MSD47, Huntsville, AL 35812 USA

Uniform microstructural development during solidification of immiscible liquids on Earth is hampered by inherent density differences between the phases. Microgravity processing minimizes settling but segregation still occurs due to gravity independent wetting and coalescence phenomena. Experiments with the transparent organic, metal analogue, succinonitrile-glycerol system were conducted in conjunction with applied ultrasonic energy. The processing parameters associated with this technique have been evaluated in view of optimizing dispersion uniformity. Experimental results to evaluate microstructural phase distributions, based on other liquid-liquid immiscibility systems, will also be presented. Support from NASA grants NAG8-1231 and NCC8-66 is gratefully acknowledged.

Advanced Technologies for Superalloy Affordability: Affordability Technology for Wrought Alloys

Sponsored by: Structural Materials Division, High Temperature Alloys Committee
Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Tuesday PM — Room: Canal C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Kenneth R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; David Mourer, GE Aircraft Engines, Lynn, MA 01910 USA

2:00 PM Invited
New Materials and Processing to Enhance Affordability: *D. L. Klarstrom*[1]; [1]Haynes International Inc., Eng. & Tech. Depts., P.O. Box 9013, Kokomo, IN 46904-9013 USA

Over the last several years, new alloys and processing have been introduced which offer the superalloy user enhanced affordability. This has occurred in the form of a lower cost material such as HR-120® alloy which has properties equivalent to currently used HASTELLO® X alloy. This alloy has also been found to serve as a significant upgrade to stainless steels for use in landbased gas turbine applications in order to increase engine capability. In another case, gas turbine engine manufacturers have been able to increase use temperatures for seal rings without the need for protective coatings through the use of HAYNES® 242™ alloy, a relatively new, high strength alloy with a low coefficient of thermal expansion. Recent studies have also pointed the way for a two-step heat treatment for the alloy that will substantially reduce the aging time required. The strength of this material can also be substantially increased by coldworking and aging to provide an exceptional alloy for bolting applications. Its high nickel content also provides excellent resistance to stress corrosion cracking. Enhanced fatigue resistance for HAYNES 230® alloy obtained via low temperature processing is also presented and discussed. HAYNES®, HASTELLOY®, HR-120 and 230 are registered trademarks of Haynes International, Inc. 242™ is a trademark of Haynes International, Inc.

2:25 PM Invited
Microstructure Modeling of Forged Components of Ingot Metallurgy Nickel Based Superalloys: *Gangshu Shen*[1]; [1]Ladish Company Inc., Adv. Matls. & Process Tech., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

The application of FEM to superalloy forging made it possible to obtain the detailed thermomechanical histories of each individual location of the forged components. Microstructure modeling procedures were developed to make use of this information for grain size prediction. The same microstructure modeling approach was used for two ingot metallurgy superalloys: Waspaloy and 718. The microstructure evolution evolved in forging process is considered in the following way: static structural changes in preheating, dynamic structural changes in deformation, and static structural changes in dwell resting between operations and final cool down. The microstructure is updated for the entire thermal and deformation processes during a forging. This methodology has been successfully used in various processes such as hydraulic press forging, mechanical press forging and hammer forging on numerous Waspaloy and 718 parts.

2:50 PM

Weight Reduction of Pneumatic Ducting Though Alloy Strength Enhancement: *Gaylord Smith*[1]; Dan Yates[1]; Dan Wallem[2]; [1]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705 USA; [2]Boeing Commercial Airplane Group, Propulsion Sys. Grp., P.O. Box 3707, MS 4T-80, Seattle, WA 98124 USA

Weight reduction of the engines for wide-body aircraft is a critical issue. One area where weight saving is possible is through the use of mill processing to dramatically increase the strength and fatigue resistance of the material employed in the pneumatic ducting, thereby making a reduction in gauge and cost from current alloys possible. Since one of the preferred alloys for this ducting is INCONEL alloy 625, a mill practice was developed to produce a 75 kis yield strength sheet product. This product is described in some detail including certain steps pertaining to the method of manufacture aimed at enhancing fatigue resistance.

3:10 PM

Development of Direct-Aged HAYNES® 242™ Alloy for Fastener Applications: *L. M. Pike*[1]; S. K. Srivastava[1]; M. C. Losch[1]; Carl E. Kelly[2]; [1]Haynes International, P.O. Box 9013, Kokomo, IN 46904 USA; [2]Pratt and Whitney, Aircraft Rd., Middletown, CT 06457 USA

One cost-effective solution for the development of materials is the adaptation of an established alloy for new applications. Improvements in certain application-critical properties may be obtained through modifications in chemistry, processing, etc. and can often be explored at a considerably lesser expense than a full-scale alloy development program. An example of this is the adaptation of HAYNES® 242™ alloy for high-temperature fastener applications. This low-expansion, high-strength alloy is used extensively in gas-turbine engines as a seal/containment ring material. However, in the standard annealed and aged condition the strength properties are less than requisite for certain fastener applications. The development of a direct-aging process (cold-work + aging) has resulted in a material with significantly improved strength while retaining respectable ductility. Room-temperature (RT) yield strengths of more than 1750 MPa (250 ksi) are possible; this represents a 100% improvement over the standard annealed and aged condition. This dramatic increase in strength allows 242™ alloy to be considered for applications where it offers a unique combination of properties not found in competing materials such as alloy 718 or Custom 455®. These properties include low-thermal expansion as well as resistance to stress-corrosion cracking in both chloride and hydrogen environments. Potential applications for direct-aged 242™ alloy include fasteners and high-strength components in both aerospace and land-based gas turbines as well as other demanding industrial environments. HAYNES® is a registered trademark of Haynes International, Inc. 242™ is a trademark of Haynes International, Inc. Custom 455®. is a registered trademark of Carpenter Technology Corporation.

3:30 PM Break

3:45 PM Invited

Development and Utilization of Press Converted Powder Metal Superalloy Billet: *Anthony Banik*[1]; Xavier Pierron[1]; David Furrer[2]; Joe Lemsky[2]; Sushil Jain[3]; [1]Special Metals Corporation, 100 Industry Ln., Princeton, KY 42445 USA; [2]Ladish Company Inc., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110 USA; [3]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206 USA

Powder metal superalloys for turbine disk applications have historically been produced by a two step hot compaction and extrusion sequence to consolidate and refine canned loose powder into consolidated billet material. Powder metal superalloys exhibit relatively low elevated temperature tensile ductility, and as such, extrusion processing has provided a means of bulk deformation under predominately compressive stresses. Recent advances in the consolidation of superalloy powders, which incorporate a sub-solidus hot isostatic press (SSHIP) consolidation practice, improves the consolidated billet ductility such that conventional conversion operations can be utilized to produce fine grain billet for subsequent disk forging. This alternate conversion route results in significant reductions in cost and lot size requirements. Results from disk forging evaluations indicate that through a controlled thermomechanical processing sequence, the microstructural response in the final components can be controlled to provide very uniform controlled grain sizes and ultrasonic inspectability.

4:10 PM

Modeling Grain Size Evolution of P/M Rene88DT Forgings: *Canan U. Hardwicke*[1]; Gangshu Shen[2]; David Furrer[2]; [1]General Electric Company, Corp. Rsrch. and Dev., One Research Circle, Niskayuna, NY 12309 USA; [2]Ladish Company, Adv. Matls. and Process Tech. Dept., 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

In a typical processing route for powder metal produced engine disks, forging is a key processing step influencing the component's final microstructure and hence, mechanical properties. The definition of a forge and heat-treat processing window to achieve the desired microstructures has been an iterative process. In this work, a microstructural model was developed for Rene88 to describe the change in grain size of a billet as it is isothermally forged and then heat treated. The first step was to develop a grain size database using isothermal compression tests with varied temperature and strain rate. Then empirical equations were developed to describe the deformation and annealing behavior. These equations were then integrated with an FEM code to predict the local microstructure variations for a given part geometry. Application of this model should reduce the trial-and-error steps in processing R88 and other commercial superalloys into useful component configurations. This work was supported by the AFRL under the contract number F33615-95-C-5229.

4:30 PM

HIP Bonding of Multiple Alloys for Advanced Disk Applications: *Tammy Marie Simpson*[1]; Allen R. Price[1]; Paul F. Browning[2]; Michael Fitzpatrick[2]; [1]Howmet Research Corporation, Adv. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA; [2]Solar Turbines Inc., Adv. Tech. Matls. and Process., 2200 Pacific Hwy., San Diego, CA 92186 USA

Dual property gas turbine disks were investigated to optimize properties in both the rim and hub sections of the disk. For the rim material, it is necessary to have good high temperature creep rupture life. However, for the hub material, both high burst strength and good low cycle fatigue are important. These properties would increase turbine fuel efficiency by allowing an increase in the turbine inlet temperature and a reduction in the disk cooling air requirements. In the past, rims and hubs of different materials have been consolidated into dual alloy wheels. However, deleterious particles, namely hafnium oxide, may have contributed to planar failure and reduced overall ductility. Howmet has demonstrated the ability to HIP bond disks with tailored material properties in specific areas without the formation of a planar oxide layer. The alloys investigated were Mar-M 246, Mar-M 247 LC, IN 792 Mod5A, and U720 in cast-wrought, Spray-Cast®, and powder metallurgy form.

4:50 PM

A New Method to Improve the Hot-Workability of High-Strengthened Superalloys: *Zhengdong Long*[1]; Jingyun Zhuang[1]; Bo Dong[1]; *Di Feng*[1]; Zengyong Zhong[1]; [1]Central Iron and Steel Research Institute, Dept. of Superalloys, No.76, Xue Yuan Nan Rd., Hai dian District, Beijing 100081 PRC

A new method based on the optimization of microstructure to improve the hot-workability of high-strengthened superalloys is proposed. By this method, the coarsened gamma prime phase is obtained by lower cooling rate at specified temperature range. This paper discusses the detailed processing, microstructure change, hot deformation flow stress, ductility and recrystallization microstructure. The results show that the new method is very effective to improve the hot-workability of this kind of superalloys. Conducting this process, the deformation uniformity is enhanced greatly, the flow stress is lowered and the hot deformation ductility is increased obviously. The mechanism to improve the hot-workability can be explained that the coarsened gamma prime phase optimizes the distribution of alloying elements and lessens its solution strengthening effect at high temperature.

Aluminum Reduction Technology: MHD/Modeling

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland, New Zealand; Georges J. Kipouros, Dalhousie University, Department of Mining and Metallurgical Engineering, Halifax, NS B3J2X4 Canada

Tuesday PM — Room: Sewanee
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Jeff Keniry, Alumination Consulting, Director, Mount Macedon, Victoria 3441 Australia

2:00 PM Invited

Metal Pad Instabilities in Aluminium Reduction Cells: D. Shin[1]; *Alfred David Sneyd*[1]; [1]University of Waikato, Dept. of Math., Private Bag 3105, Hamilton 2001 New Zealand

The electric current flowing through an aluminium reduction cell generates an associated magnetic field, and hence a Lorentz force-field which can destabilise the cryolite-aluminium interface. The cell interior can be regarded approximately as two-fluid system (cryolite/aluminium) in a rectangular container. Determining the waves which can be excited in the system is a classical problem in hydrodynamics, but the Lorentz force adds a new twist. Stability can be analysed by expressing the (small amplitude) wave system as a combination of normal modes, which in the absence of a force field or energy dissipation are non-interacting and of constant amplitude. The Lorentz force however couples the normal modes and may lead to amplitude growth, disrupting the efficient operation of the cell, particularly if two normal modes have similar frequencies (resonance). The cell magnetic field and anode-cathode separation are crucial in determining stability, and a simple physical explanation can be give for the driving mechanism.

2:25 PM Invited

Multi-Physics Modeling of Aluminium Reduction Cells: *Mark Cross*[1]; K. Pericleous[1]; L. Leboucher[1]; T. N. Croft[1]; V. Bojarevics[1]; A. Williams[1]; [1]University of Greenwich, Ctr. for Numerical Modeling & Process Analysis, Wellington St., Woolwich, London SE18 6PF UK

Aluminium cells involve a range of complex physical processes which act simultaneously to provide a narrow satisfactory operating range. These processes involve electromagnetic fields, coupled with heat transfer and phase change, two phase fluid flow with a range of complexities plus the development of stress in the cell structure. All of these phenomena are coupled in some significant sense and so to provide a comprehensive model of these processes involves their representation simultaneously. Conventionally, aspects of the process have been modeled separately using uncoupled estimates of the effects of the other phenomena; this has enabled the use of standard commercial CFD and FEA tools. In this paper we will describe an approach to the modeling of aluminium cells which describes all the physics simultaneously. This approach uses a finite volume approximation for each of the phenomena and facilitates their interactions directly in the modeling-the complex geometries involved are addressed by using unstructured meshes. The very challenging issues to be overcome in this venture will be outlined and some preliminary results will be shown.

2:50 PM

Towards a More Stable Aluminium Cell Via Busbar Configuration Optimization: M. F. El-Demerdash[1]; *A. A. Adly*[1]; S. E. Abu-Shady[1]; W. Ismail[1]; F. M. El-Dawi[2]; [1]Cairo University, Faculty of Eng., Cairo, Giza Egypt; [2]Egyptalum-Aluminium Company of Egypt, Cairo, Egypt

A modification is suggested on the present busbar design used in the prebaked end to end 203 kA Aluminum production cells at EGYPTALUM, Nage-Hammady, Egypt. Both the base configuration currently working and the modified configuration are computed using PACEM3 package for magnetic fields, forces, and stability figures. Results showed more symmetric magnetic fields especially for vertical field component, and more stable stability figures for the modified design as compared to the base design. To reach the modified design, several busbar configurations were considered. Details of those configurations, computed fields results, and corresponding stability levels are presented in detail in the paper.

3:15 PM

Thermo-Electric Design of a 400 kA Cell Using Mathematical Models: A Tutorial: *Marc Dupuis*[1]; [1]GeniSim Inc., 3111 Alger St., Jonquiere, Quebec G7S2M9 Canada

This paper presents a typical application of thermo-electric mathematical models to produce a thermally balance aluminum reduction cell lining design. The paper is structured as a tutorial, the selected example is a modern prebaked PBF cell running at 400 kA. The type of models used are the now standard steady state thermo-electric 3D full cell slice model and the "lump parameters" dynamic process model as well as the newly developed dynamic thermo-electric 2D+ full cell slice process model.

3:40 PM Break

3:50 PM

Electrolysis Cells with Symmetric Magnetic Field: *A. Panaitescu*[1]; *G. Dobra*[2]; [1]University Politehnica of Bucharest, Electrical Eng., Spl. Independentei 313, Bucharest 77206 Romania; [2]S.C. ALRO S.A., General Manager, Str. Pitesti 116, Slatina, Romania

In the cells with symmetrical magnetic field the vertical oscillations of the surface of the molten aluminium have been strongly decreased. This fact has been showed experimentally by the oscillography of the currents of the anodic rods. A standard cell in Hall no 9 loses its stability when the reference voltage decreases below 4V. In the case of the cells that work in symmetrical field, the instabilities appear at lower reference voltages. The reference voltage has been decreased to 3.65 V, and oscillations of the currents with long periods have not appeared. This is a proof that on the surface of the molten metal there are no waves. There are only oscillations with a frequency of about a second, due to the accumulations of gases under the anodes, and which determines only the decrease of the currents through rods. Long periods of time have been necessary to demonstrate the advantages of the technology in symmetrical field. Thus, at present, the data base contains the main technical indicators of cells with symmetrization for the last 910 days (two years and a half). At an industrial cell it can not be reduced only the reference voltage, because the thermal balance of the cell would be modified. The decreases of the voltages have been done with special precaution. The functioning characteristics have been averaged on 30-day periods (monthly), on 6-month periods and one year, because they have relatively important variations in time. The variation of the specific consumption of electric energy, of the efficiency of the electrolysis process (Faraday efficiency), and of the voltage drop on cell have been recorded, and the values of these quantities have been graphically represented. (Figures 1 and 2). One can notice that these cells can function at relatively low reference voltages (3.65 V has been the lowest reference voltage that has been tested and at which the cells have functioned in stability). On the x axes of the graphics there is the month the averaged value refers to. The first month is October 1996. The media have been done on groups of 6 symmetrical cells and 122 cells in the electrolysis Hall no 9 of the Aluminium Works. A specific consumption of electric energy below 13 MWh/t is a remarkable parameter, if one takes into account the type of industrial cells working in symmetrical field. (The experimental cells are open cells). In the second half of 1999 the cells with symmetrization will have a central, punctual supply, and each of them will be controlled by a microprocessor. Under these new circumstances the overvoltages due to anodic effects will be lower and it is expected that the Faraday efficiency should increase. A specific consumption of 12,5 MWh/t is achievable, which will probably be a world novelty for industrial electrolysis cells.

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Visualization of the Metal Pad Waves in the Aluminum Reduction Cell with Pre-Baked Anodes: *A. Panaitescu*[1]; A. Moraru[1]; *I. Panaitescu*[1]; [1]University Politehnica of Bucharest, Electrical Eng., Spl. Independentei 313, Bucharest 77206 Romania

In Hall-Heroult aluminum reduction cells the vertical oscillations of the metal pad have a great influence on the current and energy efficiency of the reduction process. In order to establish the least average anode to cathode distance which ensures a quiet functioning of the cell, a on-line method is necessary to survey the metal pad waves in different operation stages. In the cell with pre-backed anodes the electrolysis current is lead through several-usually four-stubs embedded in each anode block, as in Fig. 1. By measuring the beam to aluminum pad voltage drop and the stub currents, and by using an adequate model of the anode block quart resistance, including the electrolyte layer, the average thickness of the electrolyte bath below each anode quart can be established. On this basis a monitoring system was developed, using an 64 channel acquisition system, which is measuring the stub currents, with a sampling interval of 10 ms. After conditioning the acquired signals and by interoplating in space, for each set of data can be shown the profile of the pad surface. When are not known the channel scale factors stub current/voltage drop, a self-calibration of the data is used, accomplished on a "quiet" operation section. Several post-processing and plotting facilities are provided: plotting of the acquired or filtered signals, versus the time moment of acquisition, separated or grouped together on anode, plotting of the self-calibrated signals on anode rows, plotting of animated 3D aluminium pad's surface, determined with 64 stub signals, and plotting of animated 3D aluminium pad's surface, determined with 16 anode signals. The last two processing are based on the assumption that the signals (currents) vary inversely with the electrolyte layer thickness. For the 3D plot an weighted, direct and inverse, interpolation is used between the centre points which correspond to the acquired signals, using the function griddata from MATLAB. Below two 3D plots are shown (Fig.2 and Fig. 3), which illustrate the results obtained with the functions: cellfq.m for 64 stub signals and cellgq.m for 16 anode signals. The last plot is more poor in details as the first one, because also the primary information is more poor. The 3D plots may be animated, according to the duration of the acquisition cycle (0.64 s), what allows to follow the moving aluminium waves in the cell.

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Model of Process of Electrolysis: A. G. Barantsev[1]; *Vladimir V. Yurkov*[1]; V. C. Mann[1]; T. V. Piskazhova[1]; K. F. Nikandrov[1]; [1]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

A model of the process of reduction of aluminum metal has been developed allowing to describe in the first approximation the dynamic process variables such as bath temperature and bath chemistry, ACD, frozen cryolite ledge thickens, operating voltage and others. A mathematical model comprises several sub-models dedicated to different aspects of reduction process. Based on the presented mathematical model a virtual ("soft') cell was developed. A virtual ("soft") cell can be used to run analyses of various situation in a real ("hard") operating cell, for prior testing the process control algorithms and establishing optimum operating conditions for the newly designed cells and personnel training. Some fragments of the model can be incorporated into the process control system for doubly connected temperature/ratio control.

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3D Thermo-Electric Field Modeling Tool and Its Application for Energy Regime Simulations in Aluminum Reduction Cells: S. A. Sherbinin[1]; V. V. Pingin[1]; A. G. Barantsev[1]; *P. V. Polyakov*[1]; [1]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

The experience in software developing and application for various Russian aluminium plants is confirmed that parallel with universal software complexes, relatively small mobile programs for personal computers have the right to exist. The interface of these programs makes it possible for technologists and designer to work with them without any thorough training in the area of computer technique. Effective algorithms and relatively high speed of calculations allow to check a lot of versions. The developed complex was applied for calculation and analysis of 2d and 3d distributions of thermal and electrical fields in various industrial apparatus for aluminum manufacturing. The models were implemented for both cell operation optimization and new designs developing. Computer investigations of the transfer processes in aluminium reduction cells with a gas evolving anode carried in the following direction. A gas bubble does not practically conduct electrical current and it therefore insulates a certain part of the electrode surface. The 3d mathematical model for the analysis of these phenomena comprises a differential equation, describing the growth and changes of the bubble shape versus time and the Laplace equation for the electrical potential. On the basis of this model the relationship between voltage drop in electrolyte and shape, size and number of bubbles is obtained.

Carbon Technology: Green Mill and Anode Baking

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Morten Sorlie, Elkem ASA Research, Vaagsbygd, Kristiansand N-4675 Norway; Christian Dreyer, Aluminium Pechiney, St Jean De Maurienne 73303 France

Tuesday PM — Room: Knoxville A
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Nigel R. Turner, Tarconord Group, Bitmac Limited, Scunthorpe, North Lincs DN15 6UR UK

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Coke Characteristics from the Refiners to the Smelters: *Raymond C. Perruchoud*[1]; Markus W. Meier[1]; Werner K. Fischer[1]; [1]R&D Carbon Limited, Rsrch. and Dev., P.O. Box 362, Le Châblé, Sierre, VS 3960 Switzerland

Modern smelter technologies require consistent anode behaviour ending with long anode cycles for low gross and net carbon consumptions. Therefore high density but thermal shock resistant anodes as well as high performance forming machines have been developed in the last decade to match this goal. From the raw material side the coke properties like: pulverizing factor and grain stability, and bulk density and resiliency are becoming critical for the production of crack free anodes as they influence not only the fines preparation and pitching aspects but also the extent of lamination of the green anodes after forming and the crack propagation resistance. These mechanical and physical properties are influenced by the type of resid oil but also by the coking and calcining conditions. The impacts of the coke macrostructure (isotropy) from the coking to the calcining steps are reviewed. The blending aspects of green or calcined cokes with different macrostructures and sulfur content are also reviewed taking into account the anode burning behaviour. This knowledge allows optimizations of the coke production from the refineries to their usage in the carbon plant.

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Process Adaptations for Finer Dust Formulations: Mixing and Forming: *Kirstine L. Hulse*[1]; Raymond C. Perruchoud[1]; Werner K. Fischer[1]; Barry J. Welch[2]; [1]R & D Carbon Limited, P.O. Box 362, Sierre CH3960 Switzerland; [2]University of Auckland, Chem. and Matls. Eng., Private Bag 92019, Auckland, New Zealand

Traditionally recipes have used 3000 Blaine dust in the carbon paste due to limitations that existed previously in the processing equipment, particularly in the classifying, weighing, preheating and mixing stages. The optimum processing conditions will change for different recipe conditions and different equipment capabilities. For a given paste plant design, the recipe should be chosen requiring optimum processing conditions as dictated by the plant equipment limitations. This investigation demonstrates how the paste consistency of different recipes can be altered through adjusting the parameters in the preheating, mixing, forming and cooling stages to produce differing levels of anode quality.

This has been executed on pilot and production scale, vibrated and pressed anodes.

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A Method to Determine the Optimal Baking Level of Carbon Anodes: *Marilou McClung*[1]; J. Anthony Ross[1]; Gerald Chovanec[2]; [1]Century Aluminum, Primary Products, P.O. Box 98, Ravenswood, WV 26164 USA

The carbon's performance in the reduction cell is influenced by many factors including, but not limited to, raw materials, aggregate preparation, mixing, forming, and anode baking. To improve the anode's behavior in the electrolysis process each of these steps must be optimized to reduce net carbon consumption. Century's Primary Products Division has developed a systematic method to determine the maximum anode efficiency that can be achieved by the Baking Furnace operation. The method includes comparing baked anode core properties to a bake level indicator. Then using the R & D Carbon Net Carbon equation, the best possible combination of baked anode core properties can then be determined and bake levels adjusted to maximize baked anode performance. This paper will detail the procedures used to maximize the efficiency of the baked carbon, the method to review overall performance of a typical pit distribution and the method of determining bake level.

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A Method to Correlate Raw Material Properties to Baked Anode Core Performance: *Marilou McClung*[1]; J. Anthony Ross[1]; [1]Century Aluminum, Primary Products, P.O. Box 98, Ravenswood, WV 26264 USA

With new environmental regulations effecting cokers, changes in the steel industry's production, 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. These changes affect net carbon, anode density, return butt weight, pitch demand, or optimal aggregate sizing among other factors in a smelter. The challenge for Carbon Plants around the world is to produce a consistently high quality product while experiencing changes in the incoming coke and pitch. Smelters have always had difficulty tracking raw materials changes through the Green Mill, Baking, and Rodding to the anode performance in the cell. Century Aluminum has devised a method of tracking the raw materials from delivery to baked anode core. This paper will outline the methods used and give examples of the effects of raw material changes to the baked carbon.

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Improvement of Existing Anode Baking Furnaces by Use of an Advanced Firing and Control System-Benefits and Results: *Detlef Maiwald*[1]; [1]LVE Verfahrenselektronik GmbH, Essen D45138 Germany

In the primary smelters, a lot of open pit baking furnaces are already a long time in operation for the production of anodes. Many of these furnaces are still manually controlled or just have a very basic automation. Due to this situation the fuel efficiency can be poor and the pollution control according to CO, NOx and unburned carbon emissions may not satisfy the environmental authorities. With the introduction of an advanced firing and control system the fuel efficiency, the quality consistency and the productivity can be highly improved. Advanced control algorithms using the Firing Index Module and the Flooding Monitor Module ensure the complete combustion of the volatile components and increase directly the fuel efficiency. A non parametric neural network model reproduces the basic structure of the specific furnace. On-line test sequences adapts this structure to the actual flue situation and baking condition. The on-line prediction of the anode temperature by a dynamical model is used for the supervision of critical temperature gradients in the anode during pitch burn and for fine tuning of the Firing Index Module to the final baking temperature. Special instrumentation like IR-sensors and opacity meters increase the reliability of the system and can cut the running costs for consumables tremendously.

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Improvements in Albras Bake Furnaces Packing and Unpacking System: *Paulo Douglas Vasconcelos*[1]; [1]Albras Aluminio Brasileiro, Carbon Eng. Grp., Rod. PA483, Km 21, Barcarena, Para 68447-000 Brasil

ALBRAS operates four ring-type bake furnaces. Each furnace is composed by sections made up of six cells separated by partitions flue walls through which the furnace is fired. The cell is about four meters deep and accommodates four layers of three anode blocks, around which petroleum coke is packed to avoid air oxidation and facilitate the heat transfer. Each furnace is serviced by three multipurpose overhead cranes of 15 t/h of coke suction capacity. This paper shows how the Carbon Plant Engineering Group has projected and developed an internal solution using a mathematical model to analyze and optimize the pneumatic conveyor phenomenon, breaking paradigms and increasing more than four times the packing coke suction capacity.

Cast Shop Technology: Melt Shop Operations

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Paul Crepeau, General Motors Corporation, GM Powertrain Group, Pontiac, MI 48340-2920 USA; James N. O'Donnell, Commonwealth Aluminum Corporation, Department of Engineering, Louisville, KY 40202-2823 USA

Tuesday PM Room: Mississippi
March 14, 2000 Location: Opryland Convention Center

Session Chair: Julian V. Copenhaver, NSA Division of Southwire Company, Hawesville, KY 42348 USA

2:00 PM Introductory Remarks

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The Development of the Modern Dross Press: *Ophneil Henry Perry*[1]; [1]J. McIntyre Machinery, Sales, Unit G, Acorn Park Ind. Estate, Harrimans Ln., Dunkirk, Nottingham, Nottinghamshire NG36JR England

Many different technologies for recovering aluminium from dross have evolved over the years. This paper describes how the best aspects of the dross cooler, the dross press and the dross stirrer have been combined to maximize metal recovery. First Generation Machines Dross presses have been used since 1895. Dross is raked from the furnace into cast iron or steel pots and squeezed by a hydraulic ram. Early systems failed due to the problems associated with high temperature of dross and associated sticking. Steel castings tended to crack and break, and meltdowns within the system often occurred. Operating Experience with 1st Generation Machine J.McIntyre Aluminium Ltd is the UKs largest recycler of aluminium, producing 60,000 tons (132mbls) ingot per year. During 1995 we at J.McIntyre Aluminium Ltd started to use a dross press. Although we had some success with medium temperature dross, we experienced serious difficulties with hot or thermitting dross. Our conclusion was that the machine we were operating in 1995 was not much different to the original machine of 1895. From our initial trials, therefore, we had identified the following problems with the dross press: 1) Dross sticking to press head; 2) Short life of press head; 3) Press could not process hot (over 900°C) or thermitting dross; 4) Press could not process low temperature dross (below 675°C); 5) Inconsistent on-site metal recovery from press; 6) No top drain of skull; 7) Environmental problems; 8) Safety. Each of these problems will be addressed in the paper.

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Multi-Functional, Articulated Vehicle which can be Easily Adapted to the Specific Needs of a Cast Shop: *Serge Desgagne*[1]; Charles R. Emond[1]; Luc Boivin[2]; [1]Multi-Functional Vallee Inc., 310

Principale, St-Alban, Québec G0A 3B0 Canada; [2]Alcan Primary Metals, Alma Project, P.O. Box 1500, Jonquiere, Quebec G7S4LS Canada

The use of efficient, specialized mobile equipment, adapted for furnace maintenance and charging purposes, can have a major, positive impact on cast shop's operations. Forklift trucks cannot perform all the tasks required in a cast shop. Customized tools can economically ensure the safety of the furnace operators, the quality of the cast metal, improve productivity and refractory life, while reducing the number of vehicles required. This paper describes the advantages of a multi-functional articulated vehicle, adapted to the cast shops. A quick-action attaching mechanism connects semi-automatically onto a variety of modular tools which perform various tasks such as scrap charging, alloy material charging, stirring, skimming and cleaning the furnace. The tools are rigidly clamped to the vehicle and hydraulically activated for tilting, rotating and positioning. The operator can complete his work cycle without ever having to leave the vehicle. He has an unobstructed view at all times while enjoying the comfort of an air-conditioned cabin. Safety and ergonomics have played a major role in the design of the equipment.

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Protecting Refractories Against Corundum Growth in Aluminum Treatment Furnaces: *Claude Allaire*[1]; Mohamed Guermazi[1]; [1]Ecole Polytechnique of Montreal, CRIQ Campus, 8475 Christophe Colomb Rd., Montreal, Quebec H2M2N9 Canada

In aluminum treatment furnaces, corundum growth may take place at the metal line which promote the deterioration of their refractory sidewall. While conventional non-wetting additives may increase the resistance to corrosion of refractories below the metal line in such furnaces, their efficiency is significantly reduced at the high operating temperature conditions prevailing at their metal line. The purpose of this paper is to present a new additive which permit to protect refractories against the action of corundum growth in the above application.

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An Electrical Method to Monitor the State of an Inductor of a Channel Induction Furnace: *Wim Boender*[1]; John van Heeswijk[1]; Dirk Van Nieuwerburgh[2]; Jan Quisthoudt[2]; [1]Hoogovens Research and Development, P.O. Box 10000, 1970 CA IJmuiden The Netherlands; [2]Hoogovens Aluminum NV, A. Stocletlaan 87, Duffel B-2570 Belgium

In the cast house of Hoogovens Aluminium NV in Duffel, Belgium, nearly all melting furnaces are channel induction furnaces. A smooth operation of these furnaces is important. The inductors that are the heating elements of these furnaces must be extremely reliable and predictable. Hence inductors are monitored systematically. An inductor is an a.c. device for heating liquid metals. Electrically, it is similar to a short-circuited transformer. Its resistance and reactance depend on the resistances and reactances of its primary and secondary circuits. The secondary circuit's resistance and reactance are dependent on the shape and the size of the inductor channels. The changes of these channels due to wear or clogging can be assessed measuring the resistance and reactance of an operating inductor. A comparison with the regular mechanical measurements has shown that the electrical method determines an inductor's state with sufficient accuracy to monitor geometry changes. Therefore it has been adopted in daily operations.

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Remelting by Continuous Feeding of Rolled Scrap into a Melt: *Snorre Farner*[1]; Frede Frisvold[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Trondheim N-7491 Norway; [2]Ålesund College, Ålesund N-6025 Norway

Metal losses during remelting is common when recycling aluminium. Reduction of these losses could give a substantial economic gain. Experiments with continuous feeding of aluminium plates into molten aluminium have been performed. A simple steady-state mathematical model has been developed that gives the temperature profile and the penetration depth into the melt as a function of the feeding velocity, superheat, and the heat-transfer coefficients from melt to solid and from a solidified shell to the plate. A criterion for shell formation is also formulated.The results can be applied to understand more complex systems whereshredded scrap is fed into molten aluminium. The model presented could be of direct interest when feeding rolled scrap into molten aluminium.

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A Kinetic Study on the Antimony Removal of Molten Aluminum by CaSi Powder Injection: *Alfredo Flores*[1]; Juan de Dios Castrejón[1]; [1]CINVESTAV, Unidad Saltillo, Carretera Saltillo Monterrey, Km. 13, Ramos Arizpe, Coahuila 25900 Mexico

The deleterious effect of antimony over the behaviour of sodium or strontium as modifiers of the AlSi eutectic phase has been addressed in different technical papers through the last few years. Nevertheless, there are not enough publications related to the development of processess to remove antimony from molten aluminum alloys. In this sense, this paper describes the experimental results obtained after the application of a technique based on the submerged powder injection of CaSi blends, to remove antimony from molten aluminum. Kinetic measurements have been performed as a function of powder flow rate-to-carrier gas flow rate ratio, temperature of the bath, and powder size. Efficiencies in the order of 65% were reached for the best combinbation of operating parameters, so it was possible to sustain that the method studied is adequated for reaching the objective proposed.

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Dissolution Mechanism for High Melting Point Transition Elements in Aluminum Melt: *Young E. Lee*[1]; Stephen L. Houser[1]; [1]Eramet Marietta Inc., State Route 7 South Riverview Dr., P.O. Box 299, Marietta, OH 45750 USA

Alloying transition elements such as Cr, Fe, and Mn in aluminum melt with a consistent performance is a challenge to cast shop operators because of their higher melting point and density. This study is to provide an understanding of the alloying process for Cr, Fe, Mn by examining their alloying performance and dissolution behaior. The alloying performance for Cr, Fe, and Mn in aluminum melt was determined by measuring the recovery of the alloying elements in aluminum melt and the dissolution behavior by examining the microstructures of the interrupted samples during the alloying pocess. It was observed that the time for a full recovery is shorter for Fe and Mn than for Cr in a similar aluminum melt stirring condition and that their recoveries are affected by the design of the alloying additives as well as by the hydrodynamic condition of aluminum melt. The variation of the microstructures with the processing time shows that the alloying additives for Cr, Fe, and Mn go through the dissolution sequence of i ncubation, exothermic reaction, and dispersion in aluminum melt but their dissolution kinetics are different from each other. The dissolution mechanisms are proposed for the alloying process of Cr, Fe, and Mn in aluminum melt.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Crack Initiation, Growth and Fatigue Life (I)

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Zhirui Wang, University of Toronto, Department of Metals and Materials Science, Toronto, Ontario Canada; Charles McMahon, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Pedro D. Peralta, Arizona State University, Department of Mechanical and Aerospace Engineering, Tempe, AZ 85287-6106 USA; J. K. Shang, University of Illinois, Department of Materials Science and Engineering, Urbana, IL 61801 USA

Tuesday PM	Room: Canal A
March 14, 2000	Location: Opryland Convention Center

Session Chairs: L. Kunz, Institute of Physics of Materials, Brno, Czech Republic; K. Sadananda, Naval Research Laboratory, Washington, DC 20375 USA

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Fatigue Crack Initiation in FCC Single Crystals: *Petr Lukas*[1]; Ludvik Kunz[1]; [1]Institute of Physics of Materials As CR, Brno, Czech Republic

Initiation of fatigue cracks both in model fcc single crystals and in fcc single crystals used in engineering practice will be discussed. The emphasis will be placed on the role of the cyclic slip localisation in cyclic plasticity and in crack initiation. It is shown that the formation of persistent slip bands (PSBs) is confined to single crystals of suitable crystallographic orientations and of sufficiently high stacking fault energy cycled at a relatively narrow range of stress and/or strain amplitudes with zero or near-zero mean stresses at not too a high temperature. For single crystals of not suitable orientations and/or low stacking fault energy cycled under stresses and/or strains outside the critical range and/or under high temperatures the cyclic plasticity manifests itself by other forms of slip activity leading to the formation of coarse surface hill-valley topography. Thus the cyclic slip localisation plays always the crucial role in the initiation of fatigue microcracks, but this slip localisation need not be due to the PSBs; the PSBs represents only one of the modes of cyclic slip localisation. The necessary prerequisites for the microcrack initiation are (i) expressive notch-peak topography, (ii) locally higher cyclic plastic strain at the intrusion root, and (iii) irreversible slip processes around the surface intrusions including hardening of material around the root of the surface intrusions. The existing models of crack initiation are accessed in the light of these prerequisites.

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The Influence of Heat Treatment and Solidification Rate on the Behavior of Small Fatigue Cracks in a Cast Aluminum Alloy: *Michael J. Caton*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Dept. of Matls. Sci. and Eng., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; [2]Ford Motor Company, Matls. Sci. Dept., MD 3182/SRL, 20000 Rotunda Dr., Dearborn, MI 48121 USA

For a wide range of stress amplitudes, the fatigue life of cast aluminum specimens is dominated by propagation of small cracks which initiate predominantly from microshrinkage pores. Therefore, an understanding of the small crack behavior and knowledge of the pore size distribution enables the prediction of fatigue properties. A study of fatigue crack growth of small cracks (~20 µm to 2 mm) in cast W319-T7 Al, a commercial Al-Si-Cu alloy used in automotive castings, showed a significant small crack effect as well as an influence of solidification rate on propagation behavior. Faster crack growth occurs in more slowly solidified material, which possesses a lower yield strength. Interestingly, a previous study revealed that the S-N curves are very similar for the over-aged (T7) and peak-aged (T6) conditions of W319. These conditions exhibit considerably different yield strengths and nominally identical pore size distribution. This suggests similar crack growth rates despite the differences in strength. A comparison of small crack growth data for the T6 and T7 conditions will be presented. Crack growth correlating parameters and the role of yield strength will be discussed.

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On Striations and Fatigue Crack Growth in 1018 Steel: *H. Cai*[1]; Arthur McEvily[2]; [1]Motorola Corporation, Austin, TX USA; [2]University of Connecticut, Dept. of Metallu. and Matls. Eng., Storrs, CT USA

A study of the fractographic features developed during fatigue crack growth in a 1018 steel has been carried out. This study included the use of stereographic techniques in a detailed examination of the geometry of striations formed at various ΔK levels. The results are compared with a number of proposed models for striation formation. The relation between the rate of fatigue crack growth in 1018 steel and the spacing of striations is also discussed.

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Initiation and Propagation of Stage-I Cracks in Copper Single Crystals under Load Control: *Ahsan Jameel*[1]; Pedro Peralta[2]; Campbell Laird[1]; [1]University of Pennsylvania, Dept. of MSE, Philadelphia, PA 19104 USA; [2]Arizona State University, Dept. of Mech. and Aerospace Eng., Tempe, AZ 85287-6106 USA

The initiation and propagation of stage-I cracks determines the magnitude of the life in copper single crystals as the transition to stage-II behavior heralds the advent of fracture. The behavior of initiation and propagation of stage-I cracks under strain control is well understood. However, the PSB behavior and consequently the behavior of the stage-I cracks under load control is significantly different and requires an understanding before a unified methodology for predicting fatigue lives of copper single crystals based on the physics of the kinetics of stage-I crack growth can be developed. Stage-I cracks are found to nucleate in PSB's under load control. However, unlike in strain control, these cracks are almost exclusively found in micro PSB's. The cracks then grow along the PSB's with the occasional crack "skipping" on a secondary slip system to an adjacent PSB. These cracks also show a tendency, similar to that in strain control, to "rob" adjacent PSB's of strain and halt the growth of cracks in those PSB's. The extent of this "strain robbing" region under load control is not known. The growth kinetics of the population of the cracks can be modeled as a function of the cumulative strain and leads to a Coffin Manson equation for predicting the fatigue life. It is seen the this Coffin Manson equation provides a lower bound for the fatigue life of the copper single crystals.

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Mixed-Mode Thresholds in High-Cycle Fatigue: *J. P. Campbell*[1]; R. O. Ritchie[1]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., Berkeley, CA 94720 USA

High-cycle fatigue (HCF) is a prime cause of military aircraft turbine engine failures. It results from fatigue-crack growth in blades and disks, initiated at small defects often associated with fretting or foreign object damage. Due to the high frequencies (>1kHz) involved, design must be based on a HCF threshold, such that crack propagation cannot occur within ~10^9 cycles. In this work, we examine the nature of the fatigue threshold, under representative high frequency and high load-ratio conditions, in a Ti-6Al-4V blade alloy with bimodal and lamellar microstructures, with emphasis on behavior under mixed-mode (modes I + II) versus mode I conditions. It is shown that whereas the mode I threshold is decreased with increasing mode-mixity (at $\Delta K_{II}/\Delta K_{1}$ ratios from 0 to 2, at load ratios from 0.1 to 0.8), in the finer-scale bimodal microstructure, provided the driving force is computed in terms of ΔG by summing up the contributions in both mode I and II, the ΔG threshold is actually increased with mode-mixity, such that the mode I value is the worst case. In contrast, preferred crack paths in the coarser lamellar microstructures do lead to reduced ΔG_{TH} thresholds under mixed-mode conditions.

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Unified Approach to Fatigue Crack Nucleation and Growth: *K. Sadananda*[1]; A. K. Vasudevan[2]; [1]Naval Research Laboratory, Code 6323, Washington, DC 20375 USA; [2]Office of Naval Research, Arlington, VA 22217 USA

Unified approach to fatigue crack growth has been developed by the authors using a two parameter approach. It is shown that crack growth involves two thresholds, one in terms of delta K and other in terms of Kmax. Both thresholds need to be satisfied simultaneously although one or the other is the controlling parameter for a given range of load ratio R. It is shown that crack closure is neither necessary or sufficient to account for fatigue crack growth. The anomalous behavior of short cracks and the acceleration and retardation effects under under-loads and overloads have been successfully accounted using this two parameter approach. Using the Kitagawa diagram, it is shown that the concepts of fatigue crack nucleation and growth can be combined to provide a unified frame work that is self-consistent and physically meaningful. Role of internal stressed in crack nucleation, short cracks and overloads are discussed in the frame work of the unified approach.

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Effects of an Oxide Layer on Fatigue Crack Initiation in Iron and a High Strength Low Alloy Steel: *Shrikant P. Bhat*[1]; Morris E. Fine[2]; [1]Ispat Inland Inc., R&D Dept., 3001 E. Columbus Dr., East Chicago, IN 46312 USA; [2]Northwestern University, Dept. of Matls. Sci., 2225 N. Campus Dr., Evanston, IL 60208 USA

While it is well recognized that fatigue cracks initiate at the free surface, most of the mechanistic studies are conducted on carefully prepared, highly polished specimens. In contrast, in most commercial applications that utilize hot rolled steels, the oxide layer formed during the manufacture of the steel remains intact through the in-service life of the component. Through a series of graduate research projects, the Inland-Northwestern University cooperative research program examined the effects of a thermally grown wustite layer of controlled thickness on fatigue crack initiation. In this paper, fatigue crack initiation with and without the wustite layer is compared and modeled. In both commercially pure iron and an 80 ksi HSLA steel, the surface oxide layer drastically changes the crack morphology. A polished surface results in multitude of intrusions and extrusions at low strain amplitudes. In contrast, in the presence of a wustite layer, fewer surface cracks were seen. They appeared earlier in life, mainly occurring along the grain boundaries. Modeling of the crack formation in this paper is based on the accumulation of dislocations at the interface leading to the formation of crack surfaces at a critical dislocation density. The influence of grain size and substrate yield strength on fatigue crack initiation in the presence of an oxide layer will also be discussed.

High Resolution Electron Microscopy in Materials Science: Defect Structures

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

Program Organizers: Diane E. Albert, Los Alamos National Laboratory, MST-6, The Metallurgy Group, Los Alamos, NM 87545 USA; Martin Allen Crimp, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday PM	Room: Canal D
March 14, 2000	Location: Opryland Convention Center

Session Chair: Diane E. Albert, Los Alamos National Laboratory, Matls. Sci. and Tech., Los Alamos, NM 87545 USA

2:00 PM

25 Years of HREM in Materials Research: From Phase Transformations to Interface Reactions: *Robert Sinclair*[1]; [1]Stanford University, Dept. of Matls. Sci. and Eng., Stanford, CA 94305-2205 USA

Twenty-five years or so ago, we were demonstrating the applicability of lattice fringe imaging, at 0.2nm line resolution, to revealing details of phase transformation processes. As microscope resolutions gradually improved and cross-section specimen preparation methods became routine, structure imaging was successfully applied to semiconductor interfaces and eventually to defects in close-packed metals and ceramics. Accordingly high-resolution electron microscopy (HREM) became one of the most important investigative techniques in materials research. Our own work followed the path of studying reactions at interfaces, including of course development of in situ HREM. Following a brief historical overview, our current understanding of interfacial reactions will be presented, drawing on recent examples of direct contemporary technological importance (e.g. graphitization of computer hard disk overcoats, amorphization at Ta-Cu metallization interfaces etc.).

2:30 PM Invited

HREM Characterization of Slip Transmission in Lamellar TiAl: *Hamish Fraser*[1]; Jörg M.K. Wiezorek[2]; Michael Mills[1]; [1]The Ohio State University, Matls. Sci. and Eng., Columbus, OH 43210 USA; [2]University of Pittsburgh, Dept. of Matls. Sci. and Eng., Pittsburgh, PA USA

Alloys based on the intermetallic compound TiAl can be heat-treated to yield various forms of microstructure, among which the lamellar version offers advantages in terms of optimizing strength. This optimization is associated with the scale of the microstructure that can be effected by heat-treatment. While strength may be optimized, room temperature ductility usually remains at quite low values (<2%). Most useful compositions of lamellar TiAl involve (at least) two phase microstructures of TiAl and Ti3Al. Characterization of deformed samples yields the result that in the main the TiAl lamellae deform readily by twinning and slip, there is little evidence for extensive deformation in lamellae of Ti3Al. This study is aimed at developing an understanding of the deformation behavior in lamellar Ti3Al, focusing on the transmission of slip from adjacent lamellae of TiAl and the factors affecting the activation of dislocations whose Burgers vectors with components perpendicular to the basal plane of this compound (i.e.,[c]-component dislocations). It has been found that transmission of slip as well as stress induced activation of dislocations occurs but that generally motion of [c]-component dislocations is a very difficult. HREM studies have shown that the cores of these dislocations are spread fairly significantly out of their slip planes and this presumably accounts for the lack of mobility. This work has been supported by the National Science Foundation, with Dr. Bruce MacDonald as Program Manager.

3:00 PM

Fine Structure of Dislocations and Deformation Behavior of Intermetallic Compounds: *Michael J. Mills*[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43221 USA

Ordered intermetallic compounds are of considerable technological and scientific interest due to their potential as structural materials for high temperature applications. In many cases, the attractive strength at high temperatures and unique flow properties of intermetallics are linked directly to their complex crystal structures (as compared with simple metals) which affect both the core structure and dissociation of dislocations. This presentation will focus on developments in our understanding of plastic flow in several ordered intermetallics, emphasizing the connection between the fine structure of dislocations and macroscopic mechanical properties. The results of experimental investigations using high resolution TEM techniques will be described, as will the direct comparison of these fine structures with atomistic and continuum modeling approaches. The insight that these comparisons provide with respect to the unique flow properties in these alloys will also be presented. Specific examples to be discussed include the anomalous flow strength behavior observed in Ni_3Al, the anisotropic flow properties of NiAl and the deformation of gamma-TiAl at higher temperatures.

3:30 PM Break

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Comparisons of HREM Observations and Theoretical Analysis Based on Ab Initio Calculations of Dislocation Core Structures: *Kevin J. Hemker*[1]; T. John Balk[1]; Oleg N. Mryasov[2]; Yuri N. Gornostyrev[3]; Arthur J. Freeman[2]; [1]Johns Hopkins University, Dept. of Mech. Eng., 122 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Northwestern University, Dept. of Phys. and Astro., Rm. F275, 2145 No. Sheridan Rd., Evanston, IL 60208-3112 USA; [3]Institute of Metal Physics, 18 S. Kovalevskaya St., Ekaterinburg 620219 Russia

Dislocation core structures control the mobility of dislocations in a large number of metals and alloys and play an important role in determining their mechanical behavior. We have implemented a combined experimental and theoretical approach to compare and contrast the dislocation core structures in three different sets of metals and alloys: fcc Au-Ir, L12 Ni3Ge-Fe3Ge and L10 TiAl-CuAu. HREM has been utilized to experimentally observe the arrangement of atomic columns surrounding the dislocation cores, allowing for the characterization of planar and non-planar core dissociations. Theoretical analysis based on the 2D-Peierls-Nabarro model and ab initio calculations have been used to predict dislocation core spreading and splitting and to derive their effects on dislocation mobility. The theoretical predictions also provide atomic level models of the dislocation cores that can be used as inputs for HREM image simulations. Direct comparisons of simulated and experimentally obtained HREM images will be presented and used to characterize the dislocation core structures and to provide benchmarks for the ab initio calculations. This work was supported by the Air Force Office of Scientific Research, Grants No. F49620-98-1-0208 and F49620-98-1-0321.

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Structure and Dynamics of Grain Boundary Defects: *Douglas L. Medlin*[1]; [1]Sandia National Laboratories, Thin Film and Interface Sci. Dept., 7011 East Ave., Livermore, CA 94551 USA

Interfacial defects such as dislocations and steps play important roles in determining the structure and behavior of grain boundaries. Though a long standing issue in materials science, over the last decade the atomic scale information provided by high resolution transmission electron microscopy (HRTEM) has significantly improved our understanding of such defects. After reviewing some of these key advances, I will discuss our work on the structure and behavior of interfacial dislocations present at Σ=3 {112} and {111} interfaces in FCC metals. Two classes of interfacial dislocations play a role in this system: Dislocations with Burgers vector a/6<112> and with Burgers vector a/3<111>. Such dislocations originate as a means of accommodating deviations from ideal lattice coincidence or through lattice dislocation decomposition reactions. Even this relatively simple system yields a rich variety of phenomena that can be directly understood and predicted from the interfacial dislocation structure. In particular, I will concentrate on two examples: (1) glide and climb processes of a/3<111> dislocations leading to interfacial sliding and twin growth and (2) shear processes of a/6<112> dislocations and their impact on interfacial dissociation. Throughout, I emphasize the necessarily close coupling between computation and experiment, with HRTEM observations of defect configurations motivating atomistic and continuum calculations and the resulting calculations providing insight critical to interpreting the observations. This work is supported by the U.S. Department of Energy, in part by the Office of Basic Energy Sciences, Division of Materials Science, under contract DE-AC04-94AL85000.

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HREM of Crystals with Unusual Internal Lattice Bending Formed as a Result of Amorphous-Crystalline Transformations in Thin Films: *Vladimir Kolosov*[1]; A. R. Thölén[2]; [1]Ural State Economic University, Eng. Dept., 8-th March 62, GSP-985, Ekaterinburg 620219 Russia; [2]Chalmers University of Technology, Dept. of Exp. Phys., Göteborg 41296 Sweden

Unusual microstructures formed during amorphous-crystalline transformation were revealed earlier by TEM studies in some thin films [1]. This new phenomenon resides in strong (up to 120°per 1 mm of crystal length) regular, dislocation independent internal bending of the crystal lattice planes (IBCLP) in a growing crystal (realized around an axis lying in the film plane). In this paper we present HREM of some structures, demonstrating the influence of film thickness, composition and lattice orientation on the IBCLP phenomenon. Crystallization was usually initiated by electron beam inside TEM and traced in situ. HREM was performed in a plane view of the samples with a CM200FEG and a slowscan CCD camera and analyzed with the Digital Micrograph software. Bend contour technique was used to evaluate lattice orientation (in combination with selected area diffraction), internal lattice bending and crystal thickness. EDX, EELS and CBED were also used. The most general result: HREM displayed neither any lattice imperfections nor inclusions of amorphous material which could initiate and keep going the IBCLP phenomenon. For the crystallisation front moving to the thinner part of amorphous antimony selenide film evaporated with a strong thickness gradient (thickness range 100¸15 nm) lattice bending is strongly increasing. At the same time HREM reveals a steady mean interplanar spacing while lattice fringes lose the contrast and become less and less discernible as the film gets thinner. The magnitude of internal bending in crystals of hexagonal Se growing (0.5-1 mm/s) in Se-Te films with a gradient in concentration of elements (in the range 5-20% Te) is built up upon the increase of Te content in amorphous film, whereas mean interplanar spacings are the same for the samples with minimal and maximal Te content. It is rather surprising fact since the lattice constants along [001] for Se (0.496 nm) and for Te (0.593 nm) differ significantly. Crystallisation of amorphous iron oxide films is usually realised by formation of alternating circular zones of two kinds which differs in lattice orientation, imperfection and growth rate [2]: the crystal centre (initial nucleus) and the central areas of odd zones with [001] normal to the film plane are highly perfect single crystalline areas growing with the maximal rates, the central areas of even zones (where [001] tends to orient radially in the film plane) are most imperfect and are growing most slowly. At the grain boundaries which are most frequent at the latter areas no misfit or dislocation-like contrast has been observed. In some of the places examined in more detail lattice fringes are passing continuously from one grain to the other and with no very sharp offset at the boundary, corresponding to tilting boundaries. The observed regularities are explained by combination of IBCLP and crystal anisotropy. The interface between amorphous material and crystallized region (analysed in dynamics on the photos made by through-focus series) is rather sharp, with the transition region about 1 nm characterised by intermediate atom ordering and a change of diffraction contrast. HREM of CuSe whiskers with IBCLP grown in amorphous films gives direct evidence of the regular curvature of lattice planes. References: 1. Kolosov V. Yu., Proc. XII ICEM, Seattle, San Francisco Press, v. 1, 574 (1990). 2. Kolosov V. Yu. And Thölén A.R., NanoStructured Materials, 9, 323 (1997). Partial support of RSAS (grant 1557) and RFBR (grant 97-02-17784) is gratefully acknowledged.

Honorary Symposium for Professor Oleg D. Sherby: Creep Mechanisms and Behavior B

Sponsored by: Structural Materials Division, Materials Processing and Manufacturing Division, Structural Materials Committee, Shaping and Forming Committee

Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Chol K. Syn, Lawrence Livermore National Laboratory, Manufacturing & Materials Engineering Division, Livermore, CA 94550 USA

Tuesday PM — Room: Bayou E
March 14, 2000 — Location: Opryland Convention Center

Session Chair: David Bourell, The University of Texas, TX Matls. Instit., Austin, TX 78712-1063 USA

2:00 PM Invited

Creep of Oxide-Dispersion-Strengthened Ferritic Steels: *Brian Wilshire*[1]; [1]University of Wales Swansea, Matls. Eng., Singleton Park, Swansea, Wales SA28PP UK

It has recently been shown that, during creep of oxide-dispersion-strengthened (ODS) alloys, the principal role of the dispersoid particles is to stabilize the retained dislocation substructures present after thermo-mechanical processing. Linking the creep resistance of ODS alloys to their retained dislocation densities then defines three categories of stress/creep rate behaviour, depending on grain size and shape. (i) Category A characteristics are found for alloys with equiaxed grain structures and low retained dislocation densities. (ii) Category B behaviour is observed with polycrystals having high grain-aspect-ratios (GAR), relatively fine grain sizes and high retained dislocation densities. (iii) Category C properties are displayed by ODS single crystals and by coarse grain high-GAR polycrystals, both having high retained dislocation densities. The validity of this classification system is illustrated by reference to the high-temperature creep properties recorded for three types of ODS ferritic steel.

2:20 PM Invited

Critical Assessment of Creep Extrapolation Using OSD and BAS Parameter Methods: *Yoshio Monma*[1]; [1]Kochi University of Technology, Dept. of Environ. Sys. Eng., Tosayamada-cho, Kami-gun, Kochi 782-8502 Japan

Among various TTP (Time-Temperature Parameter) methods proposed for the extrapolation of creep-rupture data, the merit of OSD (Orr-Sherby-Dorn) and BAS (Barrett-Ardell-Sherby) parametric procedures are not recognized widely as compared with the LM (Larson-Miller). Because of the availability of the long-term stress rupture data from the NRIM Creep Data Sheet program, which includes many data points beyond 100,000h for various heat-resisting steels and alloys, we can directly validate the ability of fitting and extrapolation by TTP methods. This paper is to examine the accuracy of extrapolation OSD and BAS methods using computerized data evaluation system. We consider the treatment of the heat-to-heat variation, how to incorporate the microstructural changes during long-time creep in TTP analysis.

2:40 PM

The Effect of Particle Reinforcement on the Creep Behavior of Aluminum Alloys: *Paul E. Krajewski*[1]; [1]General Motors, Rsrch. and Dev. Ctr., MC-480-106-212, 30500 Mound, Warren, MI 48090 USA

The effect of particle reinforcement on the creep behavior of aluminum alloys will be reviewed. The direct and indirect strengthening effects of particle reinforcement are examined in pure, solid solution strengthened, and precipitation strengthened aluminum alloys. In pure aluminum, where strengthening is controlled by dislocation substructure, indirect composite strengthening controls creep behavior. In solid solution strengthened alloys, where a dislocation substructure does not develop, direct strengthening dominates creep behavior and strengthening can be predicted by continuum models. Finally, in precipitation strengthened alloys, indirect strengthening dominates creep behavior. The effect of the particle reinforcements on precipitate distribution and stability can actually lead to composite weakening of aluminum alloys during creep deformation. Ideas for designing a creep resistant aluminum composite will be suggested.

3:00 PM Invited

Tensile Behavior of Open Cellular 7075-T6 Al at Ambient and Intermediate Temperatures: *James C. Earthman*[1]; [1]University of California, Irvine, Dept. of Chem. Eng. and Matls. Sci., Irvine, CA 92697-2575 USA

Abstract text is not available

3:20 PM Break

3:40 PM Invited

Powder Densification Maps and Applications in Selective Laser Sintering Post Processing: *David L. Bourell*[1]; [1]University of Texas at Austin, Texas Matls. Instit., Mech. Eng., MC C22000, Austin, TX 78712 USA

Selective Laser Sintering (SLS) is a Solid Freeform Fabrication process in which a part is built quickly from powders without the use of part-specific tooling. Production of metallic, ceramic and composite parts often requires some form of post-process sintering to achieve full density. Powder Densification Maps represent a potent tool for optimizing the post-processing parameters to achieve full density parts. Such maps are computational representations of part density as affected by time, temperature, pressure and materials properties. Critical to the formulation of densification maps is an understanding of time-dependent plasticity. This presentation will summarize SLS developments at The University of Texas at Austin with emphasis on the utility of powder densification mapping of part post-processing.

4:00 PM Invited

Enhanced Densification of Titanium Powders by Cyclic Transformations under Stress: *David C. Dunand*[1]; Christopher Schuh[1]; Philippe A. Noel[2]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA; [2]Matra Defense, STTN, 20-22 Rue Grange Dame Rose, Velizy 78141 France

The densification of titanium powders is investigated in uniaxial die pressing experiments at 980°C (in the beta-field of titanium), and compared with densification during thermal cycling between 860 and 980°C (about the alpha-beta phase transformation of titanium). In agreement with the 1982 paper by Ruano, Wadsworth and Sherby on white cast iron powders, we observe that thermal cycling enhances densification kinetics of titanium powders through the emergence of transformation-mismatch plasticity (the mechanism responsible for transformation superplasticity) as a densification mechanism. The isothermal hot-pressing data compare favorably with existing models of powder densification, and these models are successfully adapted to the case of transformation-mismatch plasticity during thermal cycling.

4:20 PM Invited

Strain Rate Sensitivity and Creep Behavior of Some Nickel-Based Intermetallic: *Shu-En Hsu*[1]; [1]Hsu-Yang Technologies Company Limited, 80 Erh-Chia Rd., Ying-Keh, Taipei-Hsian, Taiwan

In response to the influence of Prof. Oleg D. Sherby's pioneering contributions to the field of high-temperature mechanical behavior, strain-rate effects and creep behavior of NiAl and TiNi intermetallics have been investigated. NiAl intermetallics were once recognized as ideal aerospace materials for key components in turbo-engines at elevated temperature. However, after many years of study, the outcome was not so successful when strain-rate sensitivity was evaluated. This report reviews the results of the strain-rate study of polycrystalline and single-crystal NiAl-intermetallics. Experimental results show that both the stress exponent and the activation energy for creep increase significantly. However, a decrease of the coefficient of strain-rate sensitivity resulted in reduction of ductility and toughness. In fact, this was a common trend for many other structural intermetallics, such as TiAl or FeAl. As a result, except for some effective toughening mechanisms that can be applied to overcome strain-rate sensitivity, the hot-shortness of these intermetallics prevented their application to crucial components in aero-engines at elevated temperature. On the other hand, unlike most of the structural intermetallics, TiNi has exceptional inherent ductility. Besides the interesting phenomena of shape memory, high damping, and pseudo-elasticity due to martensitic transformation, the co-existence of superplasticity and super-elasticity in the same material at different temperatures will attract more attention among the modern advanced materials. This report will review the strain-rate sensitivity of Cu and V-Co modified TiNi-SMA. It was found that the coefficient of strain-rate sensitivity increased as elastic modulus decreased. Modulus change was due to the stress induced martensitic transformation at low temperature, below Ms. A pseudo-elastic (Super-elastic) material can be obtained by proper control of the martensitic transformation. Alternatively, Cu-modified TiNi-SMA exhibited superplasticity at high temperature, well above Af, where the damping capacity was high since the modulus was very low. The presence of this dual behavior of super-elasticity and superplasticity is technologically significant for manufacturing engineering. Several illustrations of applications for TiNi-based intermetallics used in sporting goods will be presented in this report.

4:40 PM Invited

Singularity of Creep Strength in Ni_3Al at Stoichiometry: Seiji Miura[1]; Yoshinao Mishima[2]; *Tomoo Suzuki*[1]; [1]Hokkaido University, Div. of Matls. Sci. and Eng., Kita-13 Nishi-8, Kita-ku, Sapporo 060-8628 Japan; [1]Kochi University of Technology, Faculty of Tech., Tosayamada-cho, Kochi 782-8520 Japan; [2]Tokyo Institute of Technology, Dept. of Matls. Sci. and Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan

Systematic investigations on both poly- and single-crystalline Ni_3Al have shown the existence of singularity at stoichiometry for creep strength. The singularity had previously been observed for the apparent activation energy for the stress anomaly of the octahedral slip at intermediate temperatures as well as for the athermal stress of the cube slip. We have revealed that the viscous motion of dislocations on (111) plane governs the creep deformation of Ni_3Al at temperatures higher than the peak temperature, and the mechanism controlling the stress anomaly persists in its effect on the dislocation motion even at such high temperatures. Large number of studies have been attempted to explain the stress anomaly with the formation of cross-slipped part of screw dislocation, well-known as the Kear and Wilsdorf (K-W) mechanism, as a basis. This cross-slipped parts which have a double-kink type configuration can be expected to move with a dragging mechanism controlled by diffusivity of vacancy in the high temperature range. The double-kink type locking parts are composed of a pair of jogs, one of which acts as a vacancy source and the other as a vacancy sink for the climb motion. The time for vacancy migration from ones to the others governs the velocity of dislocations. By taking the effect of Al concentration on inter-diffusion coefficient of Ni_3Al, both the singularity at stoichiometry and the effect of off-stoichiometry on creep behavior could be explained.

Hume Rothery Award Symposium; Phase Transformations and Evolution in Materials: Session III

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

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA

Tuesday PM — Room: Johnson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Toru Miyazaki, Nagoya Institute of Technology, Matls. Sci. and Eng., Nagoya 466-8555 Japan; Long-Qing Chen, Penn State University, Matls. Sci. and Eng., University Park, PA 16802 USA

2:00 PM Invited

Multimillion Atom Simulations of Nanostructured Materials on Parallel Computers-Sintering and Consolidation, Fracture, and Oxidation: *Priya Vashishta*[1]; Martina E. Bachlechner[1]; Timothy Campbell[1]; Rajiv K. Kalia[1]; Hideaki Kikuchi[1]; Sanjay Kodiyalam[1]; Aiichiro Nakano[1]; Phillip Walsh[1]; [1]Louisiana State University, Concurrent Comp. Lab. for Matls. Sims., Depts. of Phys. and Comp. Sci., Baton Rouge, LA 70803-4001 USA

Multiresolution molecular-dynamics (MRMD) approach for multimillion atom simulations has been used to investigate mechanical failure in crystalline ceramics, nanophase ceramics, and at interfaces. Structural correlations determined by neutron scattering experiments are used to validate the interatomic potentials used in the simulations. Crack propagation and fracture are studied and the morphology of fracture surfaces is examined. Mechanical failure at semiconductor/ceramic interface is studied by applying tensile strain parallel to the interface. Multimillion atom simulations of oxidation of aluminum nanoclusters and nanoindentation simulation at room temperature and at elevated temperature will be reported along with local stress and temperature distributions. Research supported by the US DOE, NSF, AFOSR, ARO, USC-LSU MURI (DARPA & AFOSR), Austrian FWF, and PRF.

2:30 PM Invited

A Defect Model for Twinning in Ferroelectrics: *David J. Srolovitz*[1]; N. Sridhar[2]; Jeffrey Rickman[3]; [1]Princeton University, Princeton Matls. Instit., Bowen Hall, 70 Prospect Ave., Princeton, NJ 08540 USA; [2]Rockwell Science Center, Thousand Oaks, CA 91360 USA; [3]Lehigh University, Matls. Sci. & Eng., Bethlehem, PA 18015 USA

We examine electrostatic and elastic contributions to the twinning of a thin ferroelectric film. The analysis employed focuses on the multipole character of the imperfections associated with a twin, treating electrostatic and elastic defects within a unified framework. Our analytical results are complemented by simplified descriptions of the twinned system in terms of idealized defects, leading to an intuitive understanding of defect energetics. For completeness, a number of different twin geometries are examined.

3:00 PM Invited

Phase Transitions in Metallic Multilayers: *Hamish L. Fraser*[1]; Rajarshi Banerjee[1]; Suliman A. Dregia[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Dimensionally induced phase transitions in thin films and multilayers is of considerable interest because it lends the ability to tailor material properties by engineering their structure at the nanoscale. Recently, a series of interesting phase transitions have been reported in Ti/Al multilayers wherein Ti transforms from its bulk stable hcp structure to an fcc structure at small layer thicknesses (~ 5nm) and Al transforms from fcc to hcp at ~ 2.5 nm. These reported structural transitions were based on transmission electron microscopy (TEM) studies on sputter deposited Ti/Al multilayered specimens thinned in the cross-section geometry. Subsequently, a classical thermodynamic model has been proposed for explaining phase stability in multilayered materials as a function of the composition and bilayer thickness. Using this model it has been possible to rationalize the effect of cross-section thinning on the phase stability in terms of the anisotropic ingress of hydrogen into the Ti/Al multilayers. The model presents an elegant framework for rationalizing the effect of alloying/impurity additions on the phase stability. Furthermore, the scope of model has been expanded by considering its applicability to other systems including non close-packed ones such as Co/Cr multilayers. Recent experiments have explored in detail the formation of fcc Ti and the coherent to incoherent transition in Ti/Al multilayers as a function of composition and bilayer thickness. In addition, the initial thermodynamic model has been modified to account for the effect of coherency strains on the phase stability. These results will be discussed in detail and a new phase stability diagram will be proposed for Ti/Al multilayers.

3:30 PM Break

3:45 PM Invited

Constrained Phase Transformations: Thermodynamics and Microstructure: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. and Nuc. Eng., College Park, MD 20742 USA

Thermodynamics of constrained phase transformations is discussed. The concept of elastic domains is used for description of polydomain microstructures formed under 3D-, 2D- and 1D- constraint. As examples phase transformations in composites and epitaxial films are discussed, as well as self-constrained coherent phase transformations. Dependence of phase diagrams on constraint conditions is demonstrated.

4:15 PM Invited

Late-Stage Phase Separation in Elastically Stressed Solids: K. Thornton[1]; N. Akaiwa[2]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305-0047 Japan

A major challenge in predicting the late-stage evolution of microstructures in two-phase solids is that the difference in lattice parameters between the particle and matrix phases engenders a long-ranged elastic stress field. Unlike stress-free systems, where the evolution of the microstructure is driven by a decrease in the total interfacial energy of the two-phase mixture, the ripening process in these materials is driven by a decrease in the sum of the elastic and interfacial energies. It is thus not clear if the classical results of interfacial energy driven ripening hold true in elastically stressed solids. Due to the strong shape dependence of the interparticle elastic interactions this issue can only be addressed using large-scale numerical calculations. Through the use of boundary integrals and the fast multipole method, we have examined the morphological evolution of many thousands of elastically and diffusionally interacting particles. We shall discuss the temporal evolution of the statistically averaged properties of these coarsening ensembles, such as the exponent of the temporal power law for the average particle size, the dependence of the rate constant on volume fraction, and the evolution of the spatial correlations between particles.

4:45 PM Invited

Elastic Misfit Interactions at Phase Transitions in Solids: *Akira Onuki*[1]; [1]Kyoto University, Dept. of Phys., Kyoto 606-01 Japan

I will discuss effects of elastic misfit interactions in phase separation and structural phase transitions and present some new computational results in 2D and 3D. I am particularly interested in (i) slowing down of phase separation at high volume fractions with elastic misfit, (ii) critical behavior of binary solids influenced by elastic misfit (where even thermal fluctuations can be elastically affected), and (iii) structural intermediate states realized by a dilation-adjustment mechanism recently proposed by the present author.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Direct Fabrication and Metal Powders

Sponsored by: Materials Processing and Manufacturing Division,

Program Organizers: David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; James W. Sears, Lockheed Martin, KAPL Inc., D2, 114, Schenectedy, NY 12301 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Rob Wagoner, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Tuesday PM	Room: Canal E
March 14, 2000	Location: Opryland Convention Center

Session Chair: Iver E. Anderson, Iowa State University, Ames National Lab., Ames, IA 50011 USA

2:00 PM

Fabrication of Designed Materials: *Debasisch Dutta*[1]; Amit K. Ghosh[2]; Noboru Kikuchi[1]; Jyoti Mazumder[1]; [1]University of Michigan, Dept. of Mech. Eng. and Appl. Mech., Ann Arbor, MI 48109 USA; [2]University of Michigan, Dept. of Matl. Sci. and Eng., Ann Arbor, MI 48109 USA

We describe a new technology for design, representation, and fabrication of graded materials; and components. It involves the computer integration of three core technologies, that of homogenization design method, heterogeneous solid modeling, and direct material deposition. We show the overall system framework and describe each core technique in detail. Examples of design and fabrication of a materials system with desired properties using proposed methodology are presented in the paper.

2:30 PM

The Development of Residual Stress in Laser Engineered Net Shaping (LENS) Fabrication: *Michelle Griffith*[1]; Drew Nelson[2]; Mark Ensz[1]; Daryl Reckaway[1]; Donald Greene[1]; Michael Oliver[1]; [1]Sandia National Laboratory, Albuquerque, NM USA; [2]Stanford University, Stanford, CA USA

In direct laser metal deposition technologies, such as the LENS process, it is important to understand and control the emergence of residual stresses during fabrication. In essence, the thermal behavior and its transients during fabrication must be monitored and, hopefully, controlled to reduce residual stresses to usable levels. This talk will describe techniques to monitor the thermal signature and history during LENS processing. Methods to measure the residual stress in LENS components will be described, and correlation between thermal history, microstructural evolution, and resulting stresses will be discussed. Development of an understanding of residual stress manifestation and ways to reduce residual stress will be discussed.

2:50 PM

Engineering the Microstructure and Properties in 316 SS and Composite Materials: *John E. Smugeresky*[1]; D. M. Keicher[2]; J. A. Philliber[1]; J. Anthony Romero[3]; W. H. Hofmeister[4]; [1]Sandia National Laboratory, Livermore, CA 94551-0969 USA; [2]Optomec Design Company, Albuquerque, NM USA; [3]Sandia National Laboratory, Albuquerque, NM USA; [4]Vanderbilt University, Nashville, TN USA

Using the Laser Engineered Net Shaping (LENS™) process, we have evaluated the process parameter space for making useful fully dense metal shapes with monolithic, composite, and functionally graded microstructures. Samples of 316SS with, and without fine tungsten carbide particles, have been engineered to obtain a range of properties dependent on process parameters and composition. By varying only the process conditions, it is possible to obtain a range of tensile yield strengths between two to three times that of conventionally processed annealed material with little change in ductility. At double the yield strength of annealed material there is no loss in ductility. The results of our microstructure analysis will be discussed to show how both grain refinement and dislocation density affect the mechanical properties. Work supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

3:10 PM

Variable Composition Laser Cladding and Its Application in Laser Direct Casting: *Ken Watkins*[1]; Matt Murphy[1]; [1]University of Liverpool, Dept. of Eng., Laser Eng. Grp., Brownlow St., Liverpool L693BX UK

Variable composition laser cladding has been demonstrated at Liverpool by employing a multiple powder hopper system under computer control. It has been found that compositional control and microstructural variation in tertiary alloy systems can be achieved by this means. At the same time, it has been shown that laser direct build of components from CAD data can be achieved by adapting the laser cladding process to the incremental addition building of components (Laser Direct Casting). This is done using a unique six-axis manipulator that maintains a null point beneath the stationary laser at all times during the build procedure. The objective of this paper is to review these developments and to suggest how the bringing together of these two approaches can be utilized in the production of near net shape components with novel features such as variable composition layers, composite (reinforced) microstructures and SMART component functionality.

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Materials Research in the IRC Relevant to Tooling Produced by Rapid Prototyping and by Direct Laser Fabrication: T. Sercombe[1]; X. Wu[1]; I. T.H. Chang[1]; *M. H. Loretto*[1]; [1]University of Birmingham, Interdis. Rsch. Ctr., Edgbaston B152TT UK

The Interdisciplinary Research Centre (IRC) has a major activity in the area of Net Shape Manufacturing with the recent opening of a new laboratory fitted out with over £3.5M of processing equipment. Among the equipment is a sinterstation with a laser upgraded to 100W and a direct laser fabrication facility with a 1.4kW laser. Programmes are now underway aimed at producing tooling by both of these techniques which aims to produce long-life, fully dense tooling for a number of tool steels for different applications. Preliminary work has suggested that the approach which is being used has promise and progress in these fields and in other closely linked areas of research will be discussed.

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Advanced Mold Design and Construction: *Paul F. Jacobs*[1]; [1]Express Tool, Warwick, RI USA

This presentation will discuss some recent developments in advanced plastic injection mold design and construction. Specifically, information will be presented regarding each of the following important mold characteristics: low thermal inertia inserts, high thermal conductivity mold materials, nickel active surfaces, copper thermal management layers, conformal cooling channels, sequential heating and cooling, quasi-isothermal active mold surfaces, reduced part distortion, and dramatically increased mold productivity. Finite Element Analysis results and actual mold test data will be presented comparing the performance of conventional steel tools with the new, advanced nickel-copper electroformed/steel backed tools.

4:40 PM

Use of Elemental Powder Blends in the Formation of Complex Alloys Using Lens™: *Hamish L. Fraser*[1]; Katrin Schwendner[1]; Sundar Amancherla[1]; Rajarshi Banerjee[1]; [1]The Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

There are some very exciting possibilities afforded by application of LENS™ technology including rapid solidification without the need to post-consolidate particulate and net-shape processing. In contrast, there are some potential barriers to its widespread application, one being the cost involved in procuring pre-alloyed powders, particularly when the alloying elements are reactive, for example Ti. An obvious solution is the use of elemental blends of powders. The present study is aimed at assessing the viability of the use of elemental powders during LENS™ processing. As part of the initial assessment, elemental blends of Ni and Mo powders corresponding to the stoichiometry of Ni_3Mo have been processed and it has been shown that rapidly cooled, single phase material results. The compositional variations over the as-processed samples have been determined and it has been shown that the processed material is rather homogeneous. Among the various factors influencing the use of elemental powders is the heat of mixing. Some control experiments, aimed at assessing the influence of this factor, have been undertaken involving the use of elemental blends of Ti-Cr and Ti-Nb. Finally, the results of the processing of complex alloys taken from the TiAl, Mo-Si-B and Nb-Nb silicide systems will be presented and discussed.

5:00 PM

Microstructural Evolution During LENS Fabrication of H13 Tool Steel: *C. V. Robino*[1]; R. C. Dykhuizen[1]; J. A. Brooks[2]; T. J. Headley[1]; M. L. Griffith[1]; [1]Sandia National Laboratory, Albuquerque, NM 87185 USA; [2]Sandia National Laboratory, Livermore, CA 87185 USA

In the Laser Engineered Net Shape (LENS) process, parts are fabricated by creating a laser melted pool into which particles are injected. Fabrication proceeds by building the structure line by line and layer by layer. The process is thereby similar to multi-pass welding, and complex thermal histories are experienced in different regions of the build. These histories include remelting as well as numerous lower peak temperature thermal cycles. In the current work, models for the evolution of microstructure in H13 tool steel during the various cycles are being developed. These include solidification models, phase transformation models to account for reheating to intercritical peak temperatures, and carbide coarsening models to account for thermal cycles with subcritical peak temperatures. The models are based on classical descriptions of the various processes coupled with experimental measurements for the H13 alloy. The relationships between processing parameters and the resultant microstructures will also be discussed. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

International Symposium on Iridium: Oxidation and Diffusion

Sponsored by: Structural Materials Division, Refractory Metals Committee

Program Organizers: Evan K. Ohriner, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; H. Harada, National Research Institute for Metals, Tsukuba, Ibaraki 305 Japan; R. D. Lanam, Engelhard-CLAL, Careret, NJ 07008 USA; Peter Panfilov, Ural State University, Ekatrinburg 62001 Russia

Tuesday PM	Room: Jackson A/B
March 14, 2000	Location: Opryland Convention Center

Session Chairs: Peter Panfilov, Urals State University, Strength Lab., Ekaterinburg 62001 Russia; Easo P. George, Oak Ridge National Laboratory, Metls. and Ceram. Div., Oak Ridge, TN 37831-6093 USA

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Effect of a Low Partial Pressure of Oxygen on the Grain Growth Characteristics of Iridium Alloys: *C. G. McKamey*[1]; E. H. Lee[1]; J. W. Cohron[2]; A. N. Gubbi[3]; J. L. Wright[1]; L. Heatherly[1]; E. P. George[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Byron Products Inc., 3781 Port Union Rd., Fairfield, OH 45014 USA; [3]Dentsply International, Preventive Care Div., 1301 Smile Way, York, PA 17404 USA

The grain-growth behavior in a low-pressure oxygen-containing atmosphere of an Ir-0.3 wt.% W alloy was studied. This alloy, designated DOP-26, is used for cladding of 238-plutonium oxide fuel in radioisotope thermoelectric generators (RTGs). A small amount of added thorium (~60 wppm) improves the impact properties through two different mechanisms: (1) by segregating to and strengthening the grain boundaries and (2) by forming Ir_5Th precipitates in the matrix which refine grain size. However, earlier studies showed that, in an atmosphere containing a low partial pressure of oxygen (on the order of 1 mPa), the dissolution of the Ir_5Th precipitates and diffusion of thorium to the surface to form ThO_2 is thermodynamically favorable, leading to anomalous growth of near-surface grains and reduced impact strength. In this study, specimens were exposed to oxygen partial pressures of 1.3 and 13.3 mPa at temperatures of 1230, 1280, and 1330°C for times up to 3000 h. The results show the presence of anomalous growth of near-surface grains in this alloy. However, the data also show that there is no significant difference in grain size or grain growth rates for the 1.3 versus 13.3 mPa oxygen levels. This tends to indicate that, at least under these exposure conditions, grain growth in DOP-26 is controlled by the outward diffusion of thorium rather than the inward diffusion of oxygen. This is supported by Auger results which show a depletion of thorium as one moves from the interior towards the surface. Research sponsored by the Office of Space and Defense Power Systems of the U.S. Department of Energy. This work was performed for the Department of Energy at the Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corporation, under contract DE-AC05-96OR22464.

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Oxidation Behaviour of Iridium-Based Intermetallic Compounds Doped with Boron: *I. M. Wolff*[1]; P. J. Hill[1]; [1]Mintek, Phys. Metallu. Div., Private Bag X3015, Randburg 2125 South Africa

Intermetallic compounds based on Ir and Ru are candidate materials for high-temperature applications, notably as spark plug electrode (SPE) materials. The high thermodynamic stability and resistance to deformation at elevated temperature have also profiled these alloys for structural applications. Of particular interest are the systems based on IrAl (B2), Ir_3Nb (L12), and RuAl (B2), which have high melting points, and exceptional mechanical properties. The well-known 'boron ductlising effect' has been found to improve both strength and ductility in the above systems. However, additions of as little as 0.5 at % boron modify the oxidation resistance of the RuAl based compounds, leading to escalated internal oxidation rates at temperatures approaching 1300°C. Current work seeks to elucidate the effect of boron additions on the oxidation resistance of the Ir-Al and Ir-Nb systems. Thermogravimetric analyses and static isothermal oxidation techniques have been used to characterise the operative oxidation mechanisms, which leads to a comparison between the behaviour of the ruthenium- and iridium-based alloys. The companion paper explores the convergence between the singular room temperature toughness in the RuAl system and the superior strength and oxidation resistance in the IrAl system in the RuxIr50-xAl50 series.

2:40 PM

Oxidation Behavior of IrAl Alloys Containing Ni: *H. Hosoda*[1]; S. Watanabe[2]; S. Hanada[2]; [1]University of Tsukuba, Instit. for Matls. Rsch., Tsukuba 305-8577 Japan; [2]Tohoku University, Instit. for Matls. Rsch., Sendai 980-8577 Japan

Oxidation behavior of B2 IrAl alloys containing Ni was investigated. IrAl has large potential to be ultrahigh-temperature oxygen-diffusion-barrier (ODB) due to formation of smart structure composed of Ir and Al_2O_3. Ir plays a role of ODB and formation of iridium oxides (IrO_2 and IrO_3) is suppressed by Al_2O_3 formed above Ir layer. A problem of IrAl fabricated by ingot metallurgy is that oxidation easily occurs at boundaries between IrAl and Ir domains: Ir domains are usually introduced through solidification. This may be solved by a powder metallurgical method and improvement of B2 phase stability, and Ni addition was expected to improve phase stability of B2 IrAl. Alloys containing 51 mol % Al were fabricated by a reactive hot pressing method using high-purity elemental powders. Oxidation tests were carried out under the conditions of (1) continuous heating of 10 K/min up to 1863 K and (2) isotherms between 1273 K and 1863 K in an O_2 atmosphere. Oxidation behavior was studied by simultaneous thermogravimetry (TG)- differential thermal analysis (DTA) and SEM observation. It was found that Ni addition dramatically improves oxidation resistance of IrAl. Effect of Ni addition on mechanical property is also discussed.

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Oxidation of Alloys Based on B2 IrxRu50-xA150: *P. J. Hill*[1]; I. M. Wolff[1]; L. A. Cornish[2]; M. J. Witcomb[3]; [1]Mintek, Phys. Metallu. Div., Private Bag X3015, Randburg 2125 South Africa; [2]University of the Witwatersrand, Schl. of Proc. and Matls. Eng., Private Bag 3, Wits 2050 South Africa; [3]University of the Witwatersrand, Elect. Micro. Unit, Private Bag 3, Wits 2050 South Africa

Alloys for application as spark-plug electrodes, requiring good high-temperature strength and exceptional oxidation resistance at elevated temperatures, are being investigated. Previous work has shown that alloys based on IrAl have exceptionally high hardnesses, and oxidation properties comparable with those of RuAl-based alloys. Additionally, some Ir-based alloys are known for their superior high-temperature mechanical properties. An isostructural B2 phase has been found to form between IrAl and RuAl, allowing substitution between Ir and Ru. Convergence between the room temperature toughness of RuAl, and the superior high-temperature mechanical and oxidation properties of IrAl is being sought. Although a thorough assessment of the oxidation, cyclic oxidation and hot corrosion behaviour of the alloys is required, an initial indication of the alloys' properties can be obtained from isothermal oxidation testing. This paper is concerned with the isothermal oxidation behaviour of IrxRu50-xAl50 alloys in the temperature range 900 to 1400°C. Thermogravimetric testing under air and techniques such as X-ray diffraction and metallography have been used to assess the oxidation behaviour of these alloys, allowing optimal alloy compositions to be selected.

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Mutual Diffusion in Couple of Iridium-Rhenium: *A. Smirnov*[1]; [1]Engelhard-Clal L.P., 700 Blair Rd., Carteret, NJ 07008 USA

Bimetal composition of a rhenium and iridium has provided high-temperature capability for usage in different applications-rocket nozzles or crucibles for crystals growth. The use of different materials at high-temperature requires that allowance be made for possible changes of structure, strength and corrosion properties of composite material as a result of interdiffusion of components. In this work, we have studied mutual diffusion of bimetal compositions of iridium-rhenium, which was obtained by successive electrodeposition to form Ir and Re layers from conforming molten salts. Representative samples of composite material were vacuum annealed at 1950°C for different time. Formation and expansion of diffusion zone will be discussed.

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On Brittle Fracture of Iridium and Iridium-Based Alloys Exposed to Melts of Chemical Elements: V. A. Dmitriev[1]; N. I. Timofeyev[1]; *A. V. Ermakov*[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

Newest area for application of iridium is manufacture of reactors for pyrometallurgical extraction of gold and silver from industrial scraps, inasmuch as it is the sole metal that can be in contact with melted metals during long time. Study of iridium contacted with liquid Ag, Au, Pb, Sn, Zn, and Al in vacuum is the subjects for current work. It was shown that iridium does not chemically interact with melted noble metals and led, while thin films of Au and Ag appear on the samples contacted with melts. Despite this they do not embrittled iridium. On the contrary, liquid zinc, aluminum and tin dissolve iridium, what is the cause for failure of iridium containers.

4:10 PM
Mechanical Properties and Oxidation Resistance of Ir-Added FeAl Alloys: *H. Hosoda*[1]; K. Yoshimi[2]; S. Miyazaki[1]; S. Watanabe[2]; S. Hanada[2]; [1]University of Tsukuba, Instit. of Matls. Sci., Tsukuba 305-8577 Japan; [2]Tohoku University, Instit. of Matls. Sci., Sendai 980-8577 Japan

Effects of micro- and macro-alloying with Ir on mechanical properties and oxidation resistance were investigated for B2 FeAl alloys. Concentrations of Al and Ir were systematically changed to be 30, 35, and 40 mol % AL and 0 (binary FeAl), 1 and 5 mil % Ir, where additional Ir must substitute Fe sites. These Ir-added FeAL alloys were fabricated by a reactive hot pressing (RHP) method using elemental powders of 99.9% purity. RHP was carried out at 1523 K for 10.8 ks under 70 MPa in vacuum. Vacancy-eliminating and vacancy-introducing heating treatments were additionally carried out. ICP-OES chemical analysis, optical microscopy, differential scanning calorimetry up to 1773 K and X-ray diffractometry were carried out for alloy characterization. Mechanical properties were investigated by Vickers hardness tests at ambient temperature and compression tests at elevated temperature. Oxidation behavior was evaluated using simultaneous thermogravimetry-differential thermal analysis at 1273 K in an 0_2 atmosphere. Effect of vacancy on mechanical property and oxidation of Ir-added FeAl alloys will be also discussed.

4:30 PM
Grain-Boundary Diffusion of 57Co and 195Au Atomic Probes in Polycrystalline Iridium: *A. V. Ermakov*[1]; S. M. Klotsman[2]; V. N. Kaigorodov[2]; S. A. Matveev[2]; V. K. Rudenko[1]; G. N. Tatarinova[2]; A. N. Timofeev[2]; N. I. Timofeev[1]; [1]Ekaterinburg Non-Ferrous Metal Processing Plant, Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]Institute of Metal Physics, Russian Acad. of Sci., S. Kovalevskaya 18, Ekaterinburg 620219 Russia

The pioneering study of intercrystallite diffusion of 57Co and 195Au atomic probes in polycrystalline iridium (poly-Ir) produced by deformation and subsequent recrystallization of a single crystal was performed. The sectioning analysis of the diffusion zone was used. It included traditional measurements of "layer activities" and measurements of intensity of single-energy components of the atomic probe radiation from the rest of the sample left after part of the intercrystallite diffusion zone was removed. Functions sensitive to the diffusion profile type showed that a one-dimensional diffusion flow was absent at homologous temperatures, which are extremely low for FCC metals. In poly-Ir two-dimensional diffusion flow was realized at these conditions. Existence of the two-dimensional diffusion flux provides the high occupancy of states which are localized outside the grain boundary core even at such low homologous temperatures as 0.2Tmelt. The intercrystallite diffusion parameters of 57Co in poly-W and poly-Ir at comparable homologous temperatures are nearly equal. This fact is due to the same type of interatomic interactions in d-transition metals.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee
Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Tuesday PM — Room: Lincoln E
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Ramana Reddy, University of Alabama, Dept. of Met. & Matls. Eng., Tuscaloosa, AL 35487 USA; Alexandra Navrotsky, University of California, Dept. of Chem Eng. & Matls. Sci., Davis, CA 95616 USA

2:00 PM
A Thermodynamic Study of the Al-Cr-Nb Ternary System: *Kamal Mahdouk*[1]; Jean Claude Gachon[2]; [1]Universite Ibnou Zohr, Thermo. Metallu. et Rheo. des Mat., B.P. 28/S, Agadir Morroco; [2]Universite Henri Poincare, Lab. de Chimie du Solide Min., UMR 7555 Nancy 1, B.P. 239, Vandoeuvre, Cedex 54506 France

Al-Cr-Nb is one of the ternaries which is of interest in understanding the behavior of many industrial alloys. At the same time, the available results are confusing. We have undertaken an experimental study of both phase equilibria and enthalpies of formation in this system. Alloys were synthesized by reaction between powders of the pure metals and annealing at 1000°C followed by water quenching. Equilibria were characterized by X-ray diffraction and electron microprobe analysis. Enthalpies of formation were measured by direct reaction calorimetry for most of the binary intermetallics as well as for some solid and liquid solutions. The experimental results will be given and compared to existing data available in literature. The ultimate task will be a new assessment of the system.

2:30 PM
Thermodynamic Properties of Ti-Al Alloys: *R. G. Reddy*[1]; Leo Brewer[2]; [1]The University of Alabama, Metallu. and Matls. Eng., P. O. Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]University of California, Chem. Dept., M.C.1460, Berkeley, CA 94720 USA

Activities of Al in Ti-Al alloys were determined using the emf method. The emf of concentration cell was measured in the temperature range 820 to 900K. Experimental results, activities of Al in alloys showed a large negative deviations from the Raoult's law. The activity of Al 2.98x10-3 for the molefraction of Al 0.48 in Ti-Al alloys at 850K was determined.

3:00 PM
A Model for Predicting Thermodynamic Properties of Metallic Solutions from Fundamental Physical Quantities of Constituent Elements: *Peng Fan*[1]; [1]The University of Tokyo, Dept. of Metallu. and Matls. Sci., Hongo 7-3-1, Bunkyo-ku, Tokyo 113-8656 Japan

A new model for predicting thermodynamic properties of metallic solutions from the fundamental physical quantities of constituent elements has been developed, based on the new generation geometrical model proposed by Chou and Miedema model. The equations for interaction parameters in multi-component systems and for activity coefficients and excess Gibbs free energy in ternary systems have been derived. Theoretical discussions on these equations show that it is more reasonable using the new generation geometrical model than using other geometrical models such as Toop model and Kohler model. This model has been applied for predicting interaction parameters in a large number of metallic solutions, and the agreement between prediction and experimental data is reasonable.

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Thermodynamic Properties of Laves Phases: *J. H. Zhu*[1]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Bldg. 4500S, MS 6115, Oak Ridge, TN 35831-6115 USA

Thermodynamic properties of binary and ternary Laves phases have been critically surveyed and reviewed. The enthalpies of formation of many Laves phases have been determined recently by Dr. Kleppa and his coworkers using the high-temperature calorimetry. The vibrational entropies of formation of many Laves phases are now also available. Some regularities in the thermodynamic properties of Laves phases were deduced using the experimental data. 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 of many binary Laves phases were calculated by the semiempirical Miedema model, which showed good agreement with the available experimental data. Finally, the importance of understanding thermodynamic properties of Laves phases in predicting phase stability, point defects, and glass formability in these phases is discussed.

4:10 PM
In Situ Observation of Carburizing Reaction of Solid Iron with Graphite under Different Oxygen Potentials: *Taichi Murakami*[1]; Hiroyuki Fukuyama[1]; Miyuki Hayashi[1]; Masahiro Susa[2]; Kazuhiro Nagata[1]; [1]Tokyo Institute of Technology, Chem. and Matls. Sci., 2-12-1, Ookayama, Meguro-ku, Tokyo 152-8552 Japan; [2]Tokyo Institute of Technology, Metallu. and Ceramic Sci., 2-12-1, Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Exhaustion of fossil fuel and minimization of carbon dioxide emissions have been serious and urgent problems in the current steelmaking industry. From this point of view, further development, aiming at more efficient operations in the ironmaking process, is required for the coming 21st century. Temperature is one of the most effective operational factors in the ironmaking process, which is a key to reduce consumption of energy and emission of carbon dioxide. In order to make production of pig iron at temperatures as low as possible, better understandings of the carburizing reaction of solid iron are essentially required. In the present paper, reactions between solid iron and graphite have been observed in situ using a high-temperature microscope which enables a sample to be heated up to 1473K in one minute. The effect of oxygen potential on the carburizing reaction is also discussed.

4:40 PM
In-SnZn and Al-Sn-Zn New Solder Materials: *A. Mikula*[1]; [1]University of Vienna, Instit. of Inorg. Chem., Waehringerstrasse 42, Vienna A-1090 Austria

Lead-tin solders are commonly used in the electronic packaging due to their unique combination of electrical, chemical, physical, thermal and mechanical properties. Since lead alloys cause great environmental concern and health hazards it is more economical to replace lead in solder materials instead of cleaning up the electronic waste. To get the same or better properties it is therefore necessary to investigate some multicomponent systems. Two candidates are the In-Sn-Zn and Al-Sn-Zn systems. DTA measurements, lattice parameter investigations and micro-probe analyses were performed to determine the phase diagrams. The thermodynamic date was obtained by emf and calorimetric measurements. Based on this data the ternary phase diagrams were optimised by different models. A theoretical investigation of the bulk as well as the surface of liquid solder alloys has been made through the study of the concentration dependence of various properties such as concentration fluctuations in the long wavelength limit, diffusion, surface tension and surface composition. A statistical mechanical theory based on the layered structure has been used to develop expressions for the surface tension and surface composition in the frame-work of self-association and compound formation models.

5:10 PM
Hydrogen Storage Alloys and Their Application to Hydride Batteries: *Kuochih Hong*[1]; [1]Evergreen Energy Technology Corporation, Taipei, Taiwan

Hydrogen can be stored in a suitable metal as a solid metal hydride. There are many potential applications for the metal hydride technology. The most important one today is the development of commercial rechargeable hydride batteries. A metal hydride battery has many advantages over a nickel cadmium battery such as (1) much higher capacity, (2) no memory effect and, (3) no environmental pollution. Not every hydrogen storage material can be used as a hydrogen storage/hydride electrode. The criteria of useful hydrogen storage electrode alloys will be discussed. A semi-empirical formula, based on thermodynamic and electrochemical approach to predict and/or develop a useful hydride electrode alloy, Abx, is presented. There are two major alloy systems: Abx titanium-based and Abx rare earth metal-based alloy, currently used in the nickel hydride batteries. In general, a titanium-based alloy can have a higher electrochemical capacity than that of a rare earth metal-based alloy. However, a sealed nicked hydride battery using a rare earth metal-based alloy as the active material of the anode generally has a better electrochemical performance, including high working potential, high rate charging and discharging and lower internal pressure during overcharging. A brief discussion of making a nickel hydride battery will be described. Finally, several examples of hydrogen storage electrode alloys and their thermodynamic and electrochemical properties will be presented.

Liquid Metal Atomization: Fundamentals and Practice: Overview and Visualization

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: Khershed P. Cooper, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5343 USA; Frank Biancaniello, NIST, Gaithersburg, MD 20899-8556 USA; Stephen D. Ridder, NIST, Gaithersburg, MD 20899-8556 USA

Tuesday PM — Room: Bayou B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Fernand Marquis, South Dakota School of Mines and Technology, Dept. of Matls. & Metal Eng., Rapid City, SD 57701-3995 USA; Khershed P. Cooper, Naval Research Laboratory, Matls. Sci. & Tech., Washington, DC 20375-5343 USA

2:00 PM Opening Remarks and Introductions:
Khershed P. Cooper, Primary Organizer, Powder Materials Committee

2:10 PM Keynote

Metal Powder Production Via Melt Atomization: *Alan Lawley*[1]; [1]Drexel University, Dept. of Matls. Eng., LeBow Matls. Bldg., Philadelphia, PA 19104 USA

The fundamentals and industrial practice of liquid metal atomization are reviewed with particular reference to the interplay of melt properties and processing conditions on powder characteristics (size, size distribution, shape). Empirical relations, models and mechanisms are assessed for commercial and near-commercial water, gas, vacuum and centrifugal atomization and their implications considered with respect to the production of fine powders. The importance of powder yield and particle size distribution on the economics of atomization are illustrated. To conclude, trends and challenges in industrial-scale atomization are delineated.

2:45 PM Invited

Liquid Metal Atomization: Fundamentals and Practice: *Klaus Bauckhage*[1]; [1]Universitat Bremen, Institut fur Werkstofftechnik, Badgasteiner StraBe 3, Bremen D-28359 Deutschland

Atomization of liquid metals can be achieved in a variety of ways, most of them, in principle, show more or less similarity to the atomization techniques of "normal liquids", i.e. of aqueous or oil-based pure liquids, suspensions or emulsions. But since most of the technically interesting metal melts need significantly higher temperatures for atomization compared with normal liquids this in consequence results in a limitation of the variety of those techniques and in modifications of the devices. It is not only the supply of high kinetic energy (with reference to the high surface tension of the majority of melts) but also of heat energy at, in most cases, high temperature levels in order to keep the melt liquid which often in combination with a high reactivity of the liquid metals requires special materials, purpose-built devices and tailor made processes. The fundamentals mainly deal with the heat and mass transfer balances starting at the melt flow in the tundish, including the melt break-up mechanism and ending with the cooling and solidification conditions of the particles in the spray cone. The paper confines the discussion of fundamentals and practice to the most commonly and commercially used atomization techniques as for instance two-fluid atomization (gas/melt and water/melt), centrifugal atomization (by pouring the melt onto a rotating disc), and ultrasonic or ultrasonic assisted atomization.

3:15 PM Invited

Fundamental Fluid Dynamics in Liquid Metal Atomization: *Qingzhou Xu*[1]; Yizhang Zhou[1]; Gerardo Trapaga[2]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. and Eng., Cambridge, MA 02139 USA

Computational fluid dynamic techniques are used to analyze velocity and pressure fields as well as the influences of processing parameters and geometric arrangements during the liquid metal atomization process. The calculated results are summarized as follows. At first, the atomization gas is accelerated to a certain velocity as it expands through the orifice of an atomizer from the high pressure gas reservoir into the low pressure chamber. Under high atomization pressures, the atomization gas at the exit of the atomizer reaches the speed of sound and is typically underexpanded. It is conically focused in front of the liquid metal delivery nozzle, and then, spreads and decelerates into the atmospheric environment. When the atomization gas enters into the chamber at a high speed, recirculating flow fields often form in the vicinity of the delivery nozzle; and meanwhile, negative (or aspiration) pressure zones with respect to the atmospheric pressure may occur there. The aspiration pressure is determined by atomization pressure, delivery nozzle geometry and its protrusion length. In addition, the protrusion length of the delivery nozzle also influences the velocity field since the incoming atomization gas bounces off on its surface in the initial stage.

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Gas Flow Imaging and High Speed Photography of Gas Atomization: *Iver E. Anderson*[1]; R. L. Terpstra[1]; Jason Ting[2]; [1]Iowa State University, Ames Lab., 126 Metals Dev., Ames, IA 50011-3020 USA; [2]Crucible Research, Pittsburgh, PA 15205 USA

High pressure gas atomization (HPGA), a close-coupled, discrete jet atomization method, has proved to be one of the most effective methods of producing rapidly solidified fine metal and alloy powders with high yields less than 20 microns using Ar, N_2, or He gas. Visualization of gas-only flows and high speed photography of the atomization process have provided valuable insight to guide the development of a succession of HPGA nozzles. This presentation will compare the gas flow characteristics of convergent and convergent-divergent single jets and full gas jet ensembles used for HPGA. High speed photography and cinematography characterization of the HPGA process will also be presented, including brief high speed movie selections. An enhanced understanding of the melt disintegration mechanisms that operate during HPGA processing can be derived from this data. Different aspects of this work were supported by USDOE-BES under contract no. W-7405-Eng-82 and the US Naval Research Laboratory.

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Optimization of an Annular Jet Commercial Gas-Metal Atomizer: *Aaron Johnson*[1]; Frank S. Biancaniello[1]; Stephen D. Ridder[1]; P. I. Espina[1]; G. J. DelCorso[1]; [1]NIST, Fluid Flow Grp., 100 Bureau Dr., Gaithersburg, MD 20899 USA

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

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Analysis of Molten Metal Atomization Process Using Modified Phase-Doppler-Anemometry: *Jens Ziesenis*[1]; Jörg Tillwick[1]; Volker Uhlenwinkel[1]; [1]Universität Bremen, FB4/FG01, Badgasteinerstr. 3, Bremen 28359 Germany

Phase-Doppler-Anemometry (PDA) is an appropriate tool to investigate sprays. Since standard phase-Doppler systems can easily be applied to conventional materials, e.g. small water droplets or metal spheres with smooth surfaces, the optical behaviour of the solidifying particles in the liquid atomization process required the modification of the PDA technique. To demonstrate the sensibility of the modified Phase-Doppler-Anemometry results obtained by examining the spray of a moved atomizer are shown. With this modified non-intrusive, optical measurement method not only characterisation of local particle size and velocity distributions but also additional information as for instance the determination of local mass flux distributions of the spray cone can be obtained. For the particle properties the PDA results are compared to results received with different measurement techniques including sieve analysis of particles, that are collected using water filled tubes and with results obtained using a infrared pyrometer for inflight diagnostics on individual particles. Furthermore the comparison between measured PDA results and results of simulations of the metal atomization process is demonstrated.

Magnesium Technology 2000: Alloy Development and Corrosion

Sponsored by: Light Metals Division, Reactive Metals Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA

Tuesday PM	Room: Bayou C
March 14, 2000	Location: Opryland Convention Center

Session Chair: Eli Aghion, Dead Sea Magnesium, Potash House, Beer-Sheva 84100 Israel

2:00 PM

Corrosion and Galvanic Corrosion of Die Cast Magnesium Alloys: *J. Senf*[1]; E. Broszeit[1]; M. Gugau[1]; C. Berger[1]; [1]Darmstadt University of Technology, Instit. of Matls. Tech., Grafenstr. 2, Darmstadt 64283 Germany

One main disadvantage of magnesium alloys is the chemical and electrochemical reactivity of this group of materials. Consequences of this reactivity are problems in functionality, reliability and lifetime of machines and constructions made of these alloys. High purity alloys have reduced corrosion sensitivity. But looking at corrosion behaviour, the corrosion of magnesium alloys for itself is only one part of the corrosion problem. Galvanic corrosion is the other and maybe the main part, especially for machines and vehicles. Typical for the galvanic corrosion of different metallic materials in contact with each other is the enhanced corrosion of the minor noble material. Magnesium alloys are the least noble metallic materials used for machine parts. In this paper corrosion and galvanic corrosion behaviour of four die cast magnesium alloys (AZ 91, AM 60, AS 41, AE 42) are presented and discussed. Looking at the galvanic corrosion the magnesium alloys were connected to different aluminium alloys typically used in the automotive industry.

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Laboratory Evaluation of Galvanic Corrosion Resistance of Anodizing Film on Magnesium: Vladimir Tchervyakov[1]; *Guilian Gao*[1]; John Bomback[1]; Gerry Cole[1]; [1]Ford Research Laboratory, Matls. Sci. Dept., MD 3182 SRL, 20000 Rotunda Dr., Dearborn, MI 48121 USA

Galvanic corrosion of magnesium alloys is the major issue in corrosion reliability of Mg alloys in automotive applications. Anodization is a very effective way of corrosion protection for magnesium alloys. In this paper, the corrosion resistance of AZ91 alloys with different surface treatment (bare, anodized, anodized and sealed) were compared. Electrochemical test results showed that anodization not only improves general corrosion resistance, but also significantly increases its resistance to galvanic corrosion. Sealing the anodized film is also shown to further improve the corrosion resistance.

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Characterisation of Manganese-Containing Intermetallic Particles and Corrosion Behaviour of Die Cast Mg-Al-Based Alloys: *Liu-Ying Wei*[1]; Håkon Westengen[2]; Terje Kr. Aune[2]; Darryl Albright[3]; [1]Luleå University of Technology, Div. of Eng. Matls., Luleå S97187 Sweden; [2]Norsk Hydro ASA, Rsch. Ctr. Porsgrunn, P.O. Box 2560, Porsgrunn N-3901 Norway; [3]Hydro Magnesium, Magnesium Market Dev., 39209 Six Mile Rd., Ste. 200, Livonia, MI 48152 USA

Manganese is an element often used to remove iron from the magnesium melts in order to produce high purity magnesium alloys. A study of manganese-containing particles in the magnesium alloys is therefore of technological significance with regard to the corrosion properties of the Mg-Al-based alloys. TEM/SEM investigations of the as-cast microstructure and the corrosion morphologies revealed that the corrosion resistance of the studied alloys is dependent on (1) the distribution and types of small Mn-containing particles where corrosion pits could form to initiate corrosion fissures, (2) the barrier effects for the fissures propagation in the alloys. Two types of manganese-containing phases were found in the alloys studied. Type I is a flower-shaped phase and type II is of equiaxed or short-bar morphologies. The type II particles have a lower Al/Mn ratio and a much higher cathodic reaction rate than the type I phase. The type II particles could provide sites to form pits and initiate corrosion fissures. The segregated regions of higher Al content adjacent to the grain boundaries and the β-$Mg_{17}Al_{12}$ particles could act as barriers to the propagation of corrosion fissures.

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Mechanochemical Characteristics and Perspectives of New Magnesium-Lithium-Alloys: *H. Haferkamp*[1]; P. Juchmann[1]; V. Kaese[1]; M. Niemeyer[1]; T. Phan-tan[1]; [1]University of Hanover, Instit. of Matls. Sci., Fachgruppe Nichteisen-Metallurgie, Applestrasse 11a, Hannover 30167 Germany

On the base of growing tendencies towards an ecological increase in efficiency of technical products and processes it is the light weight material magnesium that enjoys recurrent attention. However, its basic attraction is presently spoiled by the deficiency of alloys that doesn't allow more complex applications. Therefore the general aspects of the alloy development are focused on improving creep resistance and enhancement corrosion resistance as well as the increase of plasticity. The aim of current research activities at the Institute of Materials Science is the development of Magnesium-Lithium-alloys with further lowered density, high ductility and high corrosion resistance. The presented research work includes a discussion of phase constitutions and their impacts on mechanical and corrosive properties. A variety of Aluminium and Silicium alloyed cph Magnesium-Lithium-materials is tested. Investigations include tensile or impact bending tests, potentiostatic tests, atmospheric corrosion test and water vapour cabinet. The materials are compared to Mg standard materials. A selection of Magnesium-Lithium-materials shows superior strength and ductility properties in comparison to representative standard Magnesium materials. Principal mechanisms of the active corrosion protection of Magnesium alloys are presented and used to achieve a satisfying corrosion resistance.

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Microstructure Property Studies of In Situ Mechanically Worked PVD Mg-Ti Alloys: *Tim Mitchell*[1]; *Panayiotis Tsakiropoulos*[1]; [1]University of Surrey, Schl. of Mech. and Matls. Eng., Guildford, Surrey GU25XH England

Physical vapour deposition is suitable for the production of Mg-TM alloys. Typical microstructures of PVD alloys consist of columnar grains and exhibit inter-columnar porosity and chemical inhomogeneity, the latter being present in bands growing perpendicular to the direction of growth of the columnar grains during deposition. Porosity and chemical inhomogeneity are detrimental to the mechanical properties and the corrosion resistance of the alloys. In situ mechanical working by flailing has been used previously to reduce porosity and eliminate columnar crystals in the microstructures of PVD deposits. In this work flailing was used in the production of PVD Mg-xTi (x=8, 14, 26, 34, 40 and 48wt%) alloys. The microstructures and surfaces of alloys in the as deposited condition and after immersion in 3wt% NaCl were characterised by electron microscopy and surface analysis techniques. The solid solubility of Ti in Mg was extended to 48wt%Ti by PVD. Flailing eliminated the columnar structure and reduced porosity locally in the regions where mechanical working was applied. In these regions there was some evidence of recrystallisation of the microstructure. The corrosion resistance of the flailed alloys decreased with increasing alloying content and was inferior to the corrosion resistance of PVD Mg. This behaviour has been attributed to the localised effects of flailing, with preferential attack occurring along the flailing lines leading to exfoliation of the deposits.

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Studies of Mg-V and Mg-Zr Alloys: *Spyros Diplas*[1]; *Panayiotis Tsakiropoulos*[1]; [1]University of Surrey, Schl. of Mech. and Matls. Eng., Matls. Sci.and Eng., Guildford, Surrey GU25XH England

Physical vapour deposition is a non-equilibrium process which is suitable for the production of Mg-X alloys, where X is alloying element with significantly higher melting point than Mg. Thus, PVD has been used for the development of Mg alloys with improved mechanical properties and corrosion resistance. As part of this effort Mg-TM alloys have been considered, where the choice of TM has been influenced by the need for a beneficial contribution of the alloying addition to the formation of a stable and if possible self-healing surface film, which could improve corrosion resistance in saline environment. Alloy design has selected candidate solute additions which include V and Zr. In this paper we will report on PVD Mg-V and Mg-Zr alloys which have been studied by bulk and surface characterisation techniques in the as-deposited condition and after immersion in 3wt% NaCl. All deposits exhibited compositional inhomogeneity, columnar microstructures and a strong basal texture. The solid solubilities of V and Zr in Mg were extended approximately to 17.5 wt% V (8.9at%) and 10.5 wt% Zr (3.5at%) respectively. The solid solution break up temperature decreased as the V and Zr content in the alloys increased. Pure V precipitated when the extended solid solubility of V in Mg was exceeded. Both the c and a lattice parameters, as well as the c/a ratio decreased with increasing V content in the Mg-V alloys. The increase of the a- lattice parameter and the decrease of the c lattice parameter led to a decrease of the c/a ratio with increasing Zr additions in the Mg-Zr alloys. The two alloy systems behaved differently regarding corrosion resistance. Mg-V alloys exhibited lower corrosion resistant than pure Mg or other Mg alloys, which has been attributed to the absence of V from the surface film formed on the alloys. Mg-Zr exhibited extremely low corrosion rates, which were attributed to the participation of Zr in the surfaces of both the as-deposited and corroded alloys.

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Solubility of Nickel in Molten Magnesium-Aluminium Alloys Above 650°C: *Harsharn Singh Tathgar*[1]; Per Bakke[2]; Eivind Øvrelid[3]; Jo Fenstad[1]; Thorvald Abel Engh[1]; Frede Frisvold[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Alfred Getz Vei 2B, Trondheim N-7491 Norway; [2]Norsk Hydro Research Centre, Porsgrunn N-3901 Norway; [3]SINTEF, Matls. Tech., Trondheim N-7465 Norway

Solubility of nickel in liquid Mg-Al-Ni alloys has been measured in the temperature range 650-900°C for 1-10% aluminium content. Comparison of nickel solubility in pure magnesium with solubility in Mg-Al alloys shows a marked reduction of nickel solubility with a small addition of aluminium. SEM analysis shows that the precipitated phases in equilibrium with the melt are AlNi and $Al3Ni_2$ with the latter becoming more dominant as the aluminium concentration increases. The solubility of Ni in equilibrium with phase AlNi in the melt according to the dissolution reaction AlNi = Al + Ni is given. The thermodynamic parameters evaluated using the least square method are also presented.

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A Perspective View on the Design of Magnesium Alloys: How Computational Thermodynamics Can Help: *Zi-Kui Liu*[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. and Eng., University Park, PA 16802 USA

Alloy development is traditionally conducted by "make-and-see" empirical approach, which is usually costly and time consuming. Alloy theories tend to explain the behavior of materials after their development. The situation is now changing thanks to the accumulated knowledge on alloy theories in the past century and the significant progress in computational techniques in the past decade. One of the emerging fields is the systems materials design. Systems materials design integrates processing, structure and properties through computational thermodynamics, kinetic simulations, and experimental prototype evaluations. The essential feature is to express the design objectives in terms of thermodynamic and kinetic parameters. Thermodynamics has often been viewed applicable to states near equilibrium only. The CALPHAD technique for computational thermodynamics developed since early 1970's has changed this view. This technique couples the phase diagram and thermochemical properties to explicitly characterize all phases in a system, including stable, metastable, and unstable phases over a wide range of temperature, pressure and composition. The modeling of Gibbs energy of individual phases enables the calculation of driving forces between any intermediate non-equilibrium states for simulating dynamic microstructure evolutions. As all commercial alloys, magnesium alloys are multi-component in nature with many intermetallic phases. To develop robust alloys that less sensitive to process variability, phase relations in multi-component alloys under both equilibrium and non-equilibrium conditions are extremely valuable for the design of alloy compositions and processing procedures. In this presentation, the concept of systems materials design applied to magnesium alloys will be introduced, and the CALPHAD technique will be discussed. Particular attention will be paid to the phase relations in the Mg-Al-Zn ternary alloys covering the composition range from AZ series to ZA series along with the future activities of the systems design of magnesium alloys.

Materials Processing in the Computer Age III: Physical and Mathematical Modeling of Materials Processes

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Tuesday PM — Room: Lincoln A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: James W. Evans, University of California, Dept. of Matls. Sci. and Min. Eng., Berkeley, CA 94720 USA; Daniel Paul Cook, Reynolds Metals Company, Corp. Rsch. and Dev., Chester, VA 23831 USA

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Physical Experimentation Coupled with Mathematical Modeling of Aluminum Automotive Wheel Casting Operations: *Daniel P. Cook*[1]; Melissa I. Bloch[2]; Juergen Biermann[3]; Jonathan A. Dantzig[2]; [1]Reynolds Metals Company, Corp. Rsch. and Dev., 13203 N. Enon Church Rd., Chester, VA 23831 USA; [3]SIMTEC Inc., 3663 Broadmoor SE, Grand Rapids, MI 49512 USA

The percentage of automotive parts manufactured from aluminum alloys has been increasing since the price of gasoline started to increase in the 1970's. The car wheel is one of these components which has been most successfully produced in aluminum. Reynolds Metals currently produces roughly 6.5 million wheels per year with production slated to increase to almost 8 million per year in the next two years. In this paper, results will be presented from plant trials conducted in a low-pressure die casting facility. Temperature measurements were conducted at a number of positions in a wheel mold during normal operation. This data yielded information such as the thermal profile in the mold side cores and the mold filling time. This data was used to validate several mathematical models of filling and solidification in wheel casting. Changes in casting practice, e.g. mold coating application, inlet pressure map, die cooling modification, and mold design suggested by further model calculations will be presented.

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A Capacitance Probe for the Measurement of Bubbles in Molten Metals: Qian Fu[1]; *James W. Evans*[1]; [1]University of California, Dept. of Matls. Sci. and Min. Eng., Berkeley, CA 94720 USA

There are many metallurgical processes where bubbles are injected into high temperature melts. Obvious examples are copper converters and the chlorine fluxing units found in the aluminum industry. These melts are opaque and the bubbles within the melt are therefore not readily observed. Electroresistivity probes have been developed (notably by Brimacombe's and by Iguchi's groups) to measure the frequency, size and velocity of bubbles in liquid metals. The probes work by detecting the interruption of electrical contact between a conductor (usually metallic) immersed in the melt and the surrounding melt, as a bubble intercepts the conductor. The paper describes an alternative probe which relies on a measurement of the capacitance between a conductor within the probe and the melt; that capacitance changes as a bubble passes. Unlike the electroresistivity probe, this capacitance probe does not have its conductor in contact with the melt, rather it is shielded by a sheath of refractory material such as alumina. Consequently the probe can withstand hostile environments and one such probe has survived, while detecting bubbles for 30 minutes in molten aluminum. The paper describes the probe construction, illustrates the signals it gives in molten metals and treats the deconvolution algorithm and software necessary to convert the bubble signals to useful information.

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Argon Bubble Behavior in Tundish Nozzles during Continuous Casting of Steel Slabs: *Hua Bai*[1]; Brian G. Thomas[1]; [1]University of Illinois, Mech. Eng. Dept., 1206 W. Green St., Urbana, IL 61801 USA

Argon injection into tundish nozzle is an efficient and widely employed method to reduce nozzle clogging in continuous casting process. It also affects casting operation and product quality by changing the flow pattern in the nozzle and mold. In this paper, a 3-D finite difference model is developed to study the liquid steel-argon bubble multi-phase turbulent flow in continuous casting tundish nozzles. Experiments are performed on a 0.4-scale "water caster" to verify the model by comparing the model prediction with the measurements using PIV (Particle Image Velocimetry) technology. The developed model is then employed to investigate the effects of various variables on flow patterns and jet characteristics for the slide-gate nozzle. Because the input and output between the real-life continuous casting operation and numerical simulation are usually different, HPQF model, based on advanced curve fitting of the multiple variable numerical results, is then developed and applied to convert the numerical modeling results to present trends that correspond with real-life operation conditions.

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Physical Modeling of the Viscosities of Two-Phase Mixtures: *Wei Liu*[1]; Sichen Du[1]; Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Metallu. Dept., Brinellv. 23, Stockholm 10044 Sweden

The viscosities of two-phase mixtures are of great importance in the modelling of the fluid flow in metallurgical processes. For example, in steelmaking and refining, the intimate mixing of metal droplets in the slag phase requires a consideration of the effective viscosities for the mixture. The present work was aimed at an understanding the phenomena underlying the viscous flow in a two-phase mixture with widely differing viscosities. Emulsions of silicone oils of known viscosities (3.45 dPa·s and 10.10 dPa·s at 293 K) with small amounts of water evenly distributed in the same were prepared by subjecting the mixture to uniform stirring using a magnetic stirrer. The uniformity of the emulsions was checked by photography. The viscosities of these emulsions were measured at constant temperature by the rotating cylinder method using Brookfield Digital Rheometer. The uniformity of the method of preparation of the emulsions was confirmed by the reproducibility of the results. The measured viscosities were generally found to be independent of the torque under the experimental conditions so that the two-phase mixture could be considered as a Newtonian liquid. The variations of viscosities with temperature and the effect of addition of a surface-active substance were also studied in this work. The measured viscosities were found to be higher than those of both pure water and silicone oil. The experimental viscosities showed a positive deviation from linearity and the deviation was found to increase with increasing concentration of water. The influence of surface tension on the viscosities was examined by the measurement of contact angle and evaluating the interfacial tension between the silicon oil and water. The results are discussed in the light of the various forces involved in the viscous flow in such systems as well as the chemical affinities.

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Fluid Flow and Particle Removal by Bubble Flotation in a Mechanically Stirred Vessel: *Lifeng Zhang*[1]; Shoji Taniguchi[1]; [1]Tohoku University, Taniguchi Lab., Dept. of Metallu., Aoba-ku, Sendai 980-8579 Japan

In this paper, firstly the 3D single-phase turbulent steady fluid flow in a vessel with four baffles stirred by a single impeller is mathematically simulated. Turbulence is modeled by using the standard model. Sliding-grid method is used to model the impeller. The flow domain is divided into two cylindrical, the inner one rotates at the same speed as the impeller, and the outer one is fixed with the baffles. Results indicate that the high-speed zone is near the paddles, and larger stirring speed generates larger fluid flow velocity. The calculated stirring intensity data are used to analyze the experimental results. Secondly the effects, such as filter pore size, gas flow rate and NaCl concentration, on bubble size are studied by experiment. For the particle removal rate by bubble flotation in this vessel, first order kinetics is adopted. The effects of initial number of the particles, gas flow rate, particle and bubble size, stirring speed and surface condition, on the removal rate constant are discussed, and an empirical equation is derived by experimental data. At last, a simple model is developed to study the particle attachment probability on bubble surface under turbulent flow conditions.

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Study on Mass Transfer Characteristic between Molten Steel and Particles in RH-PTB Refining: *Ji He Wei*[1]; Miao Wang[1]; [1]Shanghai University, Dept. of Metallic Matls., 149, Yan Chang Rd., Shanghai 200072 PRC

The mass transfer characteristic between the powder particles and the liquid steel in the RH-PTB (Powder Top Blowing) refining was investigated in a hydraulic model of 1/5 scale for a 90t RH degasser. The sodium chloride powder with chemical purity was used as the flux for blowing. The mass transfer coefficients of the liquid side were determined under the conditions of PTB. The influences of the main technological parameters on the mass transfer rate were examined. The results indicated that the mass transfer coefficient on the liquid side increases with raising the gas blowing rate, internal diameter of the up-snorkel, circulation rate of liquid and powder size, and decreases with an increase of the internal diameter of the down-snorkel. It was in the range of (1.36-7.30)x10(-4) m/s under the conditions of the present work. The appropriate dimensionless correlations were obtained.

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Mathematical Simulation of Fluid Flow in Gas-Stirred Liquid Systems: *Lifeng Zhang*[1]; Kaike Cai[2]; Shoji Taniguchi[1]; [1]Tohoku University, Taniguchi Lab., Dept. of Metallu., Aoba-ku, Sendai 980-8579 Japan; [2]University of Science and Technology Beijing, Steel. Instit., Schl. of Metallu., Beijing 100083 PRC

In the present paper, based on the two-phase (Eulerian-Eulerian) model, the 3D fluid flows in gas-stirred systems, i.e., air-stirred water vessel and argon-stirred liquid steel ladle, are simulated. In the Eulerian-Eulerian two-phase model, gas and liquid are considered to be two different continuous fields. The phases are assumed to share space in proportion to their volume fractions. The exchange between the phases is represented by source terms in conversation equations. Turbulence simulated by the model is assumed to be the property of the liquid phase. The following effects of mathematical treatments and operational factors on the fluid flow are discussed, such as interphase drag force, turbulent model, the size of bubble, and gas injection mode. Some interesting results are derived. Except the interphase drag force, the interphase lift force should be taken into accounted in order to exactly simulate the fluid flow in gas-stirred systems. Suitable turbulent model and drag coefficient have effect on the mathematical simulation. Injecting small bubbles can realize a well mixing flow condition. The distance between the two gas-injection nozzles has effect on the fluid flow.

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A Kinetic Model of Desulfurization by Powder Injection and Blowing in RH Refining of Molten Steel: *Ji-He Wei*[1]; Shou-Jun Zhu[1]; Neng-Wen Yu[1]; [1]Shanghai University, Dept. of Metallic Matls., 149, Yan Chang Rd., Shanghai 200072 PRC

The desulfurization process by injection and blowing in the RH refining of molten steel and its mechanism were considered and analyzed. Based on the two-resistance mass transfer theory and the mass balance of sulfur in the system, a kinetic model for the process was developed. The related parameters of the model, including the mass transfer coefficients, the effective powder amount in the molten steel being treated for desulfurization, were more reasonably determined. Predicting and modeling for the process by injecting and blowing the lime-based powder flux under the assumed operating modes with the different initial contents of sulfur and amounts of powder injected and blown in a RH degasser of 300t capacity were carried out using the model. The relevant circulation rate of the liquid steel and the powder injection and blowing rate were taken to be 100 t/min and 150 kg/min, respectively. The initial contents of sulfur in the liquid steel to be treated and the amounts of powder injection and blowing were respectively assumed to be 0.007, 0.006, 0.005, 0.004, 0.003, 0.002 mass-% and 10, 8, 6, 5, 4, 3 kg/t-steel. The total treatment time for desulfurization under each mode was set up to be 24 min, that is equivalent to eight circulation cycles of the liquid steel to be treated. The results indicated that the prediction made with this model is in good agreement with some data of the industrial experiments and production practice. Injecting and blowing the lime-based powder flux with the chemical composition of 85 mass-% CaO + 15 mass-% CaF_2 of 3-5 kg/t-steel, it is entirely possible to decrease the sulfur content in the molten steel to the ultra-low level below (5-10)x10-4 mass-% from (60-80)x10-4 mass-%. The total treatment time needed will appropriately be 12-20 min. Intensifying the powder injection and blowing operation and increasing the circulation rate of the liquid steel may effectively raise the rate of the process in the RH refining. The model may be expected to provide some useful information and a reliable basis for determining the reasonable technology parameters and optimizing the technology and process of desulfurization by powder injection and blowing in the RH refining of molten steel.

Packaging & Soldering Technologies for Electronic Interconnects: Interfacial Reaction and Reliability of Solder Joints

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

Program Organizers: Hareesh Mavoori, Bell Laboratories, Murray Hill, NJ 07974 USA; Srini Chada, Motorola, Plantation, FL 33322 USA; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Tuesday PM — Room: Lincoln D
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: D. Frear, Intel; Srini Chada, Motorola, Plantation, FL 33322 USA

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Microstructures and Fatigue Resistance of Electroless-Ni/Solder Interfaces: *Pilin Liu*[1]; Zhengkui Xu[1]; Jian Ku Shang[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

Electroless-Ni is widely used as a diffusion barrier against the rapid metallurgical reaction between solder alloys and copper metallization in both flip-chip and BGA packages. The interfacial microstructures developed between electroless-Ni solder alloys are complicated not only by the reaction of Sn and Ni, but also by the phosphorous dissolved in the nickel. In this work, the changes in the interfacial microstructure following reflow and aging, and the resulting fatigue resistance of the Ni-solder interfaces were studied by transmission electron microscopy and interface-fracture mechanics techniques. The results showed that phosphorous had a strong influence on both the interfacial microstructure and interfacial fatigue resistance. The effects were highly dependent on phosphorous concentration in the nickel and on thermal processing conditions. The potential for improving interfacial fatigue resistance by reducing P-concentration will be discussed.

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Under Bump Metallizations for Lead Free Solders: *Tia M. Korhonen*[1]; Peng Su[1]; Matt A. Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, MSE, 356 Bard Hall, Ithaca, NY 14853 USA

In order to use lead free solders in flip chip bonding, compatible underbump metallizations (UBMs) are needed. To obtain good adhesion, the wettable layer of the underbump metallization, which is usually Cu, must have sufficient resistance to reaction with the solder. Most commonly used lead-free solders, such as eutectic Ag-Sn and Bi-Sn, contain large amounts of tin, so that the Sn-Cu reaction during the reflow is very intense and can deplete the UBM of copper, which causes dewetting and failure of the joint. Since Sn-Ni reaction is slower, Ni is a viable alternative as the wettable layer. Another approach is to use a CuNi alloy. In this study, UBM's with different CuNi alloys were fabricated and reflowed with lead-free solders. The solder/UBM interfaces were analysed with SEM to find out how the Ni-concentration affects the reaction, and how much Ni is needed to obtain a sufficiently slow reaction rate. Shear tests were also performed to assess the reliability of the joints.

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Interfacial Reaction and Solder Joint Reliability of Sn-3.5Ag, Sn-3.3Ag-0.7Cu Solders in Lead Frame Chip Scale Package (LF-CSP): *Seung Wook Yoon*[1]; Sung Hak Hong[1]; Yoon Hwa Choi[1]; Chang Jun Park[1]; *Jong Tae Moon*[1]; [1]Hyundai Electronics Corporation, Memory Rsch. Div., Ichon, Kyoungki-do 467-701 Korea

To evaluate the Pb-free solders in application of CSP packages, Sn-3.5Ag and Sn-3.3Ag-0.7Cu solders were studied in the fields of interfacial reaction and solder joint reliability. Various substrates were prepared by electroplating as Ni, Sn/Ni, Ag/Ni on Cu alloy and with electroless Au/Ni/Cu substrate. Each jointed samples were characterized after environmental tests such as temperature cycles (-65°C~150°C) and high temperature storage test (150°C). Their fractured surface, microstructure of solder joint interface and of bulk solder ball were examined and analyzed by optical microscopy, XRD, SEM and EDX. The different types of reflow profile were investigated to find out the optimum reflow condition and the specimens prepared by higher reflow temperature showed the superior solder joint strength. The solder joint strength and the microstructural change were observed with number of reflow cycle in considering the real board mounting, too. To simulate the real surface mounting condition, Sn-3.5Ag and Sn-3.3Ag-0.7Cu balls were attached on LF-CSP packages and each components were placed on PCB board, then temperature cycle test was performed. To compare the solder joint reliability, Sn-36Pb-2Ag solder ball was also applied. After T/C test (-65°C~150°C) of PCB board level mounting, interfacial reaction and microstructure such as crack initiation site and crack propagation, were investigated. Also the Daisy electrical test PCB board was prepared and evaluated the solder joint reliability with T/C test.

3:05 PM

Effects of Solder Reflow on Wettability, Microstructure and Mechanical Properties: F. Guo[1]; S. Choi[1]; *J. P. Lucas*[1]; K. N. Subramanian[1]; [1]Michigan State University, Matls. Sci. & Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Solder joints used in electronic applications undergo reflow operations. Such operations can affect the solderability, interface intermetallic layer formation and the resultant solder joint microstructure.

These in turn can affect the overall mechanical behavior of such joints. In this study the effects of reflow on solderability and microstructure were studied. These studies were carried out with Sn-Ag solders, with or without Cu or Ag reinforcements, using Cu and Cu-Ni-Au substrates. Mechanical properties were carried out on joints made with the same solders using copper substrates.

3:25 PM

BGA Underfill Reliability for Underhood Automotive Electronics: *Ping Kwong Seto*[1]; John Evans[1]; Wayne Johnson[2]; [1]Daimler Chrysler Corporation, Huntsville Elect. Div., 100 Electronics Blvd., Huntsville, AL 35824 USA; [2]Auburn University, 200 Broun Hall, Auburn, AL 36849 USA

As the use of Ball Grid Array (BGA) packages proliferate, the demand for packaging density and cost savings is driving automotive electronics towards smaller form factor BGA to be used in the underhood environment. In order to ensure the solder joint integrity of the BGA components are not compromised due to this miniaturization, DaimlerChrysler Huntsville Electronics (DCHE) and Auburn University have embarked on a study to use BGA underfill to achieve the reliability goal. Since the use of underfill for flip chip has been demonstrated to be very effective in enhancing solder joint life. In this multiple phases study, DCHE is evaluating the feasibility of using underfill to extend the BGA solder joints while still meeting the aggressive manufacturing cycle time. The reliability impact of various printed circuit board (PCB) finishes and pad geometries in thermal cycling performance are also assessed. BGA packages ranging from 27mm to 15mm body sizes are included in the study.

3:45 PM Break

4:00 PM Invited

Interfacial Microstructure and Mechanical Reliability of Sn-Ag Solder Joint on Electroless Ni-P Film: *Yoshiharu Kariya*[1]; Kumiko Nakamura[1]; Yasunori Tanaka[2]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Matls. Sci. and Eng., Shibaura 3-9-14, Minato-ku, Tokyo 1088548 Japan; [2]NEC Corporation, Mobile Comm. Div., Ikebe-cho 4035, Tsuzuki-ku, Yokohama, Kanagawa 224 Japan

Packages of the area array mounting type which are suitable for high-density mounting are receiving attention, and the production volume of fine pitch BGA (ball grid array)-type packages is consequently increasing. With diminishing solder joint sizes, characteristics of interfacial microstructure which affect the mechanical reliability of the joins are an issue that can not be neglected. Recently, electroless Ni-P/Au plating has been used for surface finishes of substrate or electrode to meet a demand on mounting technology of fine pitch BGA. However, BGA solder joint on electroless Ni-P plating often exhibits poor mechanical reliability because brittle interfacial reaction layers which are formed during soldering process. Therefore, understanding the interfacial microstructure between solder and electroless Ni-P plating and its effect on the mechanical reliability is required in order to develop highly reliable solder joints. In this paper, the interfacial microstructure between Sn-Ag eutectic and electroless Ni-P plating has been characterized using SEM and TEM. The effect of interfacial microstructure on the mechanical reliability of actual CSP (chip size package) will also be presented.

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Mechanical Behaviour of Transfusion Bonded Joints: *Tommi O. Reinikainen*[1]; Roope Nikander[2]; Jorma K. Kivilahti[2]; [1]Nokia Research Center, Nokia Grp., P.O. Box 407 FIN-00045 Finland; [2]Helsinki University of Technology, Lab. of Elect. Prod. Tech., P.O. Box 3000, TKK FIN-02015 Finland

A recently introduced novel joining method-Transfusion Bonding (TFB)-allows fluxless Pb-free microjoining by utilising metallurgically compatible low melting point metals being bicoated chemically or electrochemically (/1/). Essentially, it combines the benefits of intermetallic- and paste-free joining and bonding, and therefore it can be used also for very fine-pitch electronics assembly. This type of joining and bonding technique is becoming ever more important, when thinner diffusion barrier or adhesion layers, coated overlayers and especially smaller solder joint volumes are encountered in very high density electronic assemblies. Moreover, since there are only a limited number of new viable eutectic alloy candidates for soldering applications, the TFB technique being based on the formation of Sn-rich solid solution joints can make use of more new metallurgical options for tailoring low temperature interconnection materials. If the undercoating is pure Sn or Sn-based alloy and the topcoating is pure Bi, the bonding occurs well below 200°C and the resulting microstructure of the joints is essentially dilute Sn[Bi] solid solution, the concentration of Bi being originally somewhat higher in the middle of the joint than near the component lead or substrate interfaces. Dissolved Bi has a very strong hardening effect on Sn (/2/). However, if the amount of Bi exceeds significantly the solubility limit, about 3.5 wt-% at room temperature, pure Bi will precipitate at the grain boundaries; at low supersaturation bismuth tends to precipitate as globular particles while at the higher supersaturations it will precipitate discontinuously along the grain boundaries. In the latter case the reaction product has an embritteling effect on the joints. In this work the mechanical behaviour of several dilute Sn[Bi] solid solutions have been studied, and respective material models and parameters were assessed and implemented in a finite-element program. The effect of the Bi distribution in the solder joint is analysed by the FEM and the joining process parameters are modified accordingly to achieve optimum reliability. The simulation results are verified by experiments which are conducted by shear tests with dimensions similar to real solder joints (/3/). 1. J.Kivilahti and K.Kulojärvi, 'A New Reliability Aspect of High Density Interconnections', The Proc. of Design and Reliability of Solders and Solder Interconnections", TMS Annual Meeting, Orlando, 9-13 February 1997, USA, pp. 377-384. 2. T. Reinikainen and J. Kivilahti, ´Deformation Behaviour of Dilute SnBi(0.56at%) Solid Solutions´, Metallurgical and Materials Transactions A, Vol. 30A, January, 1999, pp. 123-132. 3. T. Reinikainen, M. Poech, M. Krumm and J. Kivilahti, ´A finite-element and experimental analysis of stress distribution in various shear tests for solder joints´, Trans. ASME J. Elec. Pack., Vol. 120, March, 1998, pp. 106-113.

4:45 PM Invited

The Effect of Solder Paste Residues on RF Signal Integrity: *Laura J. Turbini*[1]; James Brokaw[1]; John A. Williams[1]; Juergen Gamalski[2]; [1]Georgia Institute of Technology, Matls. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA; [2]Siemens AG, Dept. ZT ME6, Siemensdamm 50, Berlin D-13623 Germany

Wireless devices such as pagers and cellular phones are becoming common consumer items. These products require low loss RF signal propagation which is affected by material choices and processing conditions. This paper examines the effect of a series of no-clean solder pastes on signal integrity using an RF test circuit which sends a broadband RF signal through a gallium arsenide antenna switch and measures its transmission using a network analyzer. The test circuit also measures signal leakage. This paper reports on two different test vehicles, one that used a 900 MHz antenna switch, and the other that used a 2.0 GHz antenna switch. The transmission and leakage readings were taken daily for 20 days while the test vehicles were under accelerated aging conditions of 85°C and 85% RH. Average values for the readings for each solder paste were plotted to provide comparison among the pastes. The comparison data clearly distinguish solder pastes that provide consistency throughout the test period from those which do not.

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Microstructure and Interfacial Characteristics of Electronic Interconnects: *F. A. Khalid*[1]; S. E. Benjamin[1]; M. Rashid[1]; [1]GIK Institute of Engineering Sciences, Topi, Nwfp Pakistan

The importance of electronic interconnects have been increased with the advancement in technology due to the miniaturization of electrical components. This has also lead to an increase in the number of input/output terminations. Consequently not only the solder joints have increased but also the joint dimensions have decreased in electronic packages with increased speed and greater packaging density. However, reliability has become crucial because of their use to control operational and safety functions in aerospace and automobile applications. The present work focuses on the examination of microstructural features and their influence on the interfacial properties of substrate using optical, scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). A series of solder alloys with and without lead alloying developed were used to investigate interdiffusion characteristics and the formation of the intermetallic phases to elucidate interfacial properties of solder alloys.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Pressure Technology Applications in the Hydrometallurgy of Copper and Zinc

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee
Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Tuesday PM Room: Lincoln C
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Gerry Bolton, Dynatech Corporation, Fort Saskatchewan, Alberta T8L4K7 Canada; James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA

2:30 PM
Chemistry and Mechanisms of High Pressure Acid Leaching (HPAL) Chalcopyrite Flotation Concentrates: *C. J. Ferron*[1]; N. McKay[1]; I. Dymov[1]; D. Butcher[2]; [1]Lakefield Research Limited, 185 Concession St., Postal Bag 4300, Lakefield, Ontario KOL-2110 Canada; [2]General Gold Australia

The feasibility of applying high pressure acid leaching (HPAL) as an alternative to smelting for chalcopyrite concentrates was tested on various concentrate samples from the Guelb Moghrein deposit in Mauritania. Initial bench scale tests on various concentrates confirmed HPAL to be technically feasible. Further bench and pilot plant tests using steady-state recycle streams, revealed the significant impact of gangue materials in the concentrate, in particular magnesium minerals on the leach process. Kinetic sampling during the pressure leach indicated an initial copper precipitation, followed by dissolution. Possible mechanisms of chalcopyrite dissolution during HPAL are briefly discussed.

3:00 PM
The Treatment of Chalcopyrite with Nitrogen Species Catalyzed Oxidative Pressure Leaching: *Corby G. Anderson*[1]; [1]Montana Tech., The Ctr. for Adv. Min. and Metallu. Process., Rm. 221, LLC Bldg., Butte, MT 59701 USA

Today, with a stringent economic and environmental climate prevailing in the copper business, there is increased interest in evaluating new processing alternatives for production. Hydrometallurgical pressure oxidation of copper concentrates is one of the more viable approaches and several technological candidates have emerged. Of these, an overlooked but, ironically the first industrially proven methodology, utilized nitrogen species catalyzation in the oxidizing pressure leach system to produce copper via SX/EW. This may prove to be a feasible process alternative for the future. In this paper, the history of the system and its application to chalcopyrite concentrates will be outlined. In particular, a novel methodology for effective recovery of precious metals from chalcopyrite concentrates will be discussed. Finally, the perceived economics of this unique industrially proven process will be delineated.

3:30 PM
Pressure Acid Leaching of Zinc and Copper Concentrates by Dynatec: K. R. Buban[1]; *M. J. Collins*[1]; I. M. Masters[1]; J. Stiksma[1]; [1]Dynatech Corporation, Metallu. Tech. Div., Fort Saskatchewan, Alberta T8L4K7 Canada

Pressure leaching of zinc sulphide concentrates has been practiced commercially for nearly two decades. The process, which includes direct leaching of concentrate with spent electrolyte in an autoclave, has been commercialized at four separate locations to date. In pilot studies carried out in the Dynatec lab in Fort Saskatchewan, dozens of additional zinc concentrates have been shown to be amenable to pressure leaching. This paper highlights recent improvements in zinc pressure leaching that have been identified through process development studies, and how the experience gained in these studies has been utilized in the development of a new copper pressure leach process.

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Pressure Leaching of Copper Refinery Slimes: *Bradford C. Wesstrom*[1]; [1]Phelps Dodge Refining Corporation, 6999 N. Loop Rd., El Paso, TX 79915 USA

The extraction of metals from Copper Refinery Slimes may involve many different processes. This paper describes the use of pressure leaching for decopperizing of copper refinery slimes at the El Paso Refinery. Process equipment will be described, along with the process chemistry, unit operations and unit processes associated with the decopperizing process. Copper contained in the raw slime ranges from 15-35%. After autoclaving less than 1% copper remains.

4:45 PM
Outokumpu Process for the Precious Metal Refining from Copper Anode Slime: *Olli Jarvinen*[1]; [1]Outokumpu Wenmec Oy, P.O. Box 103, Riihitontuntie, 7 E 02200 Espoo

The capacity of the Outokumpu Pori refinery is today about 125 000 mt in 1998. The anode slime amount is about 700-800 mt. The copper is removed with pressure leaching. Selenium roasting is done with the gas roasting. The doré smelting in the furnace is done with the tilting rotating converter with oxy-fuel burner (TROF). Doré anodes are treated in the silver electrolysis. Gold is recovered from the gold mud with the quick hydrochloric acid leaching process. The important point in the leaching is to get into contact the solid copper with the oxygen and liquid. Outokumpu has developed an efficient agitator for this purpose. The GLS-agitator (GLS gas, liquid and solid) is powerful for mixing oxygen, liquid and solid together.

Process Synthesis and Modeling for the Production & Processing of Titanium & Its Alloys: Session II

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, Titanium Committee, Shaping and Forming Committee
Program Organizers: James A. Hall, Oremet-Wah Chang, Albany, OR 97321 USA; F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey, USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

Tuesday PM Room: Knoxville B
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Sam Froes, University of Idaho, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey, USA

2:00 PM
Effect of Grain Size and Strain Rate on Room Temperature Mechanical Properties of a TiAl Intermetallic Alloy: M. R. Shagiev[1]; A. V. Kuznetsov[1]; *G. A. Salishchev*[1]; R. M. Imayev[1]; O. N. Senkov[2]; F. H. (Sam) Froes[2]; [1]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia; [2]University of

TUESDAY PM

Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA

Gamma TiAl alloy samples with grain sizes varying from 0.4 μm to 17 μm were produced by an isothermal forging in the temperature range 800-1000°C followed by annealing at 770-990°C. Tensile mechanical properties of the specimens were determined at room temperature and microstructures before and after deformation were analyzed using TEM. Peak ductility of 6.9% was achieved at a strain rate around 0.1 s^{-1} and grain size of 8 μm. The increased ductility was accompanied with development of single-system deformation twins. Activation of multiple twinning in specimens with larger grain sizes and suppression of the twin development in specimens with the grain size below 1 μm led however to considerable decrease in ductility. The yield stress versus grain size followed the Hall-Petch relation with the Hall-Petch parameter k_y=0.4 MPa $m^{0.5}$, that is the dependence was about 5 times weaker as compared to the coarse-grained material.

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High Temperature, High Strain Deformation of Coarse-Grained Beta-CEZ Titanium: *Henry J. Rack*[1]; C. R. Robinson[1]; [1]Clemson University, Cer. and Matls. Eng., 110 Olin Hall, Clemson, SC 29670-0907 USA

The high temperature, high strain deformation behavior of coarse grained Widmanstatten Beta CEZ (Ti-4.8Al-2Sn-3.8Zr-3.8Mo-1.9Cr-1Fe-0.045O) has been investigated between strain rates of 1 to 2x10-4 s-1 at temperatures between 1013 and 1138K. Under these conditions the compressive stress-strain response exhibited an initial maximum followed by flow softening with increasing strain. At the lowest temperatures and highest strain rates examined this flow softening behavior was followed at the highest strains examined by strain hardening. Dynamic material modeling indicated that stable flow and therefore optimal deformation conditions can be expected above strain rates of 10-2 s-1 at temperatures between 1025 and 1100K. This presentation will discuss how establishment of this stable flow regime involves a balance between grain boundary associated deformation, resolution of the phase, dynamic spheroidization of the grain boundary and lamellae, dynamic recovery and recrystallization of lamellae and dynamic grain growth. Stable flow was associated with dynamic recovery and recrystallization of the constituent and phases, while unstable flow at the lowest temperature examined, 1013K, was associated with shearing of grain boundary and void formation at/interfaces, the former predominating at high strain rates, the later at the lower strain rates examined. With increasing deformation temperature void formation was progressively replaced by localized flow within the grain boundary region incorporating dynamic spheroidization, dissolution of the phase and dynamic recrystallization of the phase. Finally at the highest temperatures and strain rates, unstable flow was associated with flow localization and kinking of the lamellae within the near-grain boundary region.

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Low-Temperature Superplasticity of TiAl and Ti3Al Based Alloys: *R. M. Imayev*[2]; G. A. Salishchev[2]; V. M. Imayev[2]; M. R. Shagiev[2]; N. K. Gabdullin[2]; O. N. Senkov[1]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia

The data on superplastic behavior of TiAl and Ti3Al intermetallic alloys with a high ordering energy and submicron grain size are summarized. By decreasing grain size from 10 ìm to 0.1 ìm, the superplastic temperatures were decreased from 1000-1200°C to 600-900°C and the strain rate range was substantially extended towards the higher strain rates. The effects of composition, superlattices type, and grain size on low-temperature superplastic properties of titanium aluminides was investigated. An increase in the volume fraction of the Ti3Al phase in TiAl based alloys led to improvements in the superplastic properties such as ductility and strain rate sensitivity and more homogeneous deformation. The activation energies of superplastic flow were determined to be close to the activation energy of grain boundary diffusion. The considerable decrease in superplastic forming temperatures opens possibilities in cost reduction by using less expensive tools and deformation processes.

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The Influence of Solution Treatment Temperature on Martensitic Phase Transformations in IMI 550: *Henry J. Rack*[1]; Krishna Kharia[1]; [1]Clemson University, Cer. and Matls. Eng., 110 Olin Hall, Clemson, SC 29670-0907 USA

The influence of solution treatment temperature on the martensitic phase transformations observed in IMI 550(Ti-4Al-4Mo-2Sn-0.5Si) has been investigated. When solution treated of equilibrated samples is conducted at temperatures of above 960°C followed by rapid cooling a hexagonal martensite, δ', is formed. However at temperatures between 850 and 960°C, rapid cooling results in the formation of orthorombic, δ". Finally below this temperature region the β phase is stable-no martensitic transformation occurring on rapid cooling. This transition from δ' $\delta_{primary} + (\delta' + \beta_{retained})$ $\delta_{primary} + (\delta'' + \beta_{retained})$ $\delta_{primary} + \beta_{metastable}$ with decreasing solution treatment temperature will be discussed by considering the effect of solution treatment temperature on alloy partitioning and the influence of this partitioning on phase stability as revealed by x-ray, transmission electron microscopy and elevated temperature neutron diffraction.

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Characterization of a Gamma-TiAl Sheet Produced from Blended Elemental Powders: *Javaid I. Qazi*[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; Valadimir S. Moxson[2]; [1]University of Idaho, IMAP, Mines Bldg. Rm. # 321, Moscow, ID 83844-3026 USA; [2]ADMA Products Inc., 8180 Boyle Park Way, Twinsburg, OH USA

Gamma titanium aluminide sheet of a nominal composition of Ti-46.5Al-2Cr-3Nb-0.2W was produced from blended elemental powder. A novel loose sintering approach combined with hot rolling at temperatures near alpha-transus temperature was used. The sheet was characterized using X-ray diffraction, optical microscopy and TEM. A fully lamellar structure was produced with a colony size of around 100mm. The material consisted of two phases: gamma-TiAl and about 20% of Ti3Al. The porosity level was about 2%. Mechanical properties of the sheet were also studied.

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Ductility of a Ti3Al Intermetallic: Effect of Grain Size and Partial Disordering: *R. M. Imayev*[2]; N. K. Gabdullin[2]; O. N. Senkov[1]; G. A. Salishchev[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia

The effect of grain size and partial disordering on ductility and flow stress of an intermetallic Ti3Al was studied in the temperature range of 20 to 800°C using tension and compression testing. The ductility of the fully ordered material increased considerably when the grain size decreased from 27 μm to 0.1 μm, and, at the smaller grain size, an elongation of 4.8% was achieved at room temperature. Partial disordering of the crystal lattice led to a decrease in ductility at temperatures below 500°C but had no influence on ductility at higher temperatures. The critical grain size at which a brittle-to-ductile transition occurred in the fully ordered material was determined for each temperature studied. This critical grain size increased as the temperature increased. Dislocation slip changed from a localized planar mode to a more uniform fine slip, the slip line spacing decreased, cross-slip developed, and relaxation capability of grain boundaries enhanced when the grain size decreased below the critical value. The fracture mode changed from a brittle transcrystalline mode to a brittle intercrystalline mode and, finally, to a ductile mode when the grain size decreased and temperature increased. Transcrystalline fracture was observed in specimens with grain sizes above the critical grain size, while intercrystalline and ductile fracture modes occurred in specimens with grain sizes below the critical grain size. Specimens with submicron sized grains exhibited features of superplastic flow at 600°C and above.

Research and Development Efforts on Metal Matrix Composites: New Directions in MMC Research

Sponsored by: Joint ASM-MSCTS/TMS-SMD Composites Committee; Young Leaders Committee
Program Organizers: John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; Warren H. Hunt, Aluminum Consultants Group Inc., Murrysville, PA 15668 USA

Tuesday PM	Room: Bayou A
March 14, 2000	Location: Opryland Convention Center

Session Chairs: John J. Lewandowski, Case Western Reserve University, Matls. Sci. and Eng., Cleveland, OH 44106 USA; Benji Maruyama, US Air Force, Air Force Rsch. Lab., Wright Patterson AFB, OH 45433 USA

2:00 PM Invited
Directions for Research in Discontinuously-Reinforced MMC's: *Daniel B. Miracle*[1]; Benji Maruyama[1]; [1]AF Research Laboratory, Matls. and Manu. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

A great deal of progress has been made in the past three decades in the understanding, development, processing and implementation of discontinuously-reinforced MMC's. Significant markets are now established in the military and commercial aerospace industry, as well as automotive, electronics and recreation product sectors. However, dramatic additional benefits in affordability, performance and supportability are expected from more widespread application of this pervasive metals technology. The results of a recent analysis of potential applications in the aerospace industry will be provided. Suggestions for research that will be required to support the development of MMC's for these applications will be provided and discussed.

2:30 PM
New Directions in the Characterization of Composite Microstructures: *Jonathan Edward Spowart*[1]; [1]Air Force Research Laboratory, Matls. Direct., AFRL/MLLM Bldg. 655, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433 USA

Previous experimental and theoretical work has shown that there is strong correlation between the fracture toughness of discontinuously-reinforced metallic matrix composites and the spatial arrangements of the reinforcing particles. However, until recently this aspect of the composite microstructure has received little attention. Previous attempts to characterize particle distributions in composite microstructures have concentrated on the statistics of the particle-particle spacings. These approaches, based either on Radial Distribution Functions (RDF's) or tessellation schemes, reveal only limited information about the spatial arrangements of individual particles. In this paper, new characterization techniques are demonstrated whereby the inter- particle connectivity is given priority over inter-particle proximity. The importance of the connectivity of potential damage sites is discussed in terms of crack propagation phenomena. The analysis allows multi-scale clustering parameters to be defined, using fractal geometry techniques. Results are given both for experimentally obtained and artificially simulated microstructures. Cellular automata-based crack propagation simulations reveal preferential paths for crack propagation in clustered microstructures, and show strong correlations between the degree of particle clustering and selected crack path metrics. Using crack propagation simulations, fracture toughness predictions are obtained from the microstructures of two different PM-processed SiC- reinforced 7093Al materials with contrasting particle spatial distributions, following the approach of Rice and Johnson. There is good agreement between the predictions and the experimentally-obtained fracture toughness values for these materials.

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Development of a New Discontinuously Reinforced Aluminum MMC: *Aaron C. Hall*[1]; [1]The University of Illinois, Dept. of Matls. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

A new metal matrix composite based on high aspect ratio aluminum diboride flakes (diameter/thickness > 100) has been developed. This new material is a discontinuously reinforced aluminum composite in which AlB2 flakes that are prepared in situ act as the stiffening phase. In situ preparation of the AlB2 reinforcement simplifies processing making this composite a low cost material (~ $5.00/kg). Target applications include automotive brake rotors and turbine components where a combination of excellent wear resistance and high specific properties are required. Mechanical testing and simulation of braking conditions using a laboratory scale dynamometer suggest promise for this material. Current development efforts are focused on elucidating low cost synthesis routes that will allow preparation of high aspect ratio AlB2 in the absence of AlB12 and other borides. As part of this effort the growth of AlB2 in liquid aluminum has been extensively studied. It has been found that the rate at which a liquid aluminum boron alloy is cooled dramatically affects the aspect ratio of the resulting AlB2. In addition, the aluminum rich region of the aluminum boron phase diagram has been reexamined. The peritectic temperature has been found to be significantly lower than that presented in the literature.

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In-Situ Ductile Metal/Bulk Metallic Glass Matrix Composites Formed by Chemical Partitioning: *Choongnyun Paul Kim*[1]; Chuck C. Hays[1]; William L. Johnson[1]; [1]California Institute of Technology, Matl. Sci., W.M. Keck Lab. of Eng., Mail Stop 138-78, Pasadena, CA 91125 USA

A new class of ductile metal reinforced bulk metallic glass matrix composite material have been prepared that demonstrate improved mechanical properties. This newly designed material exhibits both improved toughness and large plastic strain to failure. The remarkable glass forming ability of bulk metallic glasses allows for the preparation of ductile metal reinforced composites with a bulk metallic glass matrix via in-situ processing; i.e. chemical partitioning. The incorporation of a ductile metal phase into a metallic glass matrix yields a constraint that allows for the generation of multiple shear bands in the metallic glass matrix; this stabilizes crack growth in the matrix and extends the composite strain to failure. Specially, by control of chemical composition and processing conditions a stable two-phase composite (ductile metal in a bulk metallic glass matrix) is obtained on cooling from the liquid state. The crystalline beta-phase has a dendritic morphology with a particle size and periodicity that is determined by the chemical composition and processing conditions. These ductile metal particles impose intrinsic geometrical constraints on the bulk metallic glass matrix that leads to the generation of multiple shear bands under mechanical loading. Sub-standard Charpy specimens prepared from this new composite material have demonstrated Charpy impact toughness numbers that are 250% greater than that of the bulk metallic glass matrix alone. Bend test have shown large plastic strain to failure values of ~4%. Specimens tested under compressive loading exhibit large plastic strains to failure on the order of 8%. Another key factor to the improved behavior is the quality of the interface between the ductile metal beta-phase and the bulk metallic glass matrix is chemically homogeneous. This clean interface allows for stable deformation and for the propagation of shear bands through the beta-phase particles. X-ray diffraction, microstructural, and mechanical property results are presented for a ductile metal reinforced bulk metallic glass matrix composite based on bulk glass forming composites in the Zr-Ti-Cu-Ni-Be system.

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Metal Matrix Composites Based on Aluminum and Optical Fibers: *Ferdinando Felli*[1]; Antonio Paolozzi[2]; Michele A. Caponero[3]; [1]University of Rome "La Sapienza", Dept. ICMMPM, Via Eudossiana 18, Roma 00184 Italy; [2]University of Rome"La Sapienza", Dept.

Aerospaziale, Via Eudossiana 18, Roma 00184 Italy; [3]Enea Frascati Research Center, Via Enrico Fermi 46, Frascati, Roma 00044 Italy

Embedding optical fiber into structural components is a very promising technology for real time health monitoring. Recently the interest for producing low cost and reliable sensor is increased in the mechanical, civil and aerospace engineering community. Aluminum and polyimide coated optical fibers have been successfully embedded in different aluminum alloys by using both cast and colamination techniques. The studied samples have been subjected to the following tests: mechanical tests in order to verify the structural integrity; optical transmission tests to highlight the fiber integrity after embedding process; metallographic observation of the fiber-matrix interface; interferometric tests in order to verify that the embedded optical fiber can be usefully used as a global sensor for measuring variation of the physical properties of the hosting material. The possibility of embedding optical fibers in aluminum alloys has been demonstrated. The obtained aluminum composites show interesting performances.

4:00 PM

Creep and Stress Rupture Behavior of Be/Al Composite Materials: *Shihong Gary Song*[1]; J. T. Beals[1]; V. C. Nardone[1]; [1]United Technologies Research Center, 411 Silver Ln., Hartford, CT 06108 USA

Aluminum-beryllium composites are known for their ultra lightweight and exceptionally high specific stiffness and have found broad applications as static and low-stress components in aerospace industry. An increasing interest in the alloys has recently been seen for dynamic, high-temperature, and high-stress applications for aircraft and engine components. However, there is an apparent scarcity in literature of the property data of these alloys, in particular, the secondary mechanical property data such as creep, fatigue, and fracture toughness. The present investigation concerns the time dependent deformation and rupture behavior at elevated temperatures of a commercial aluminum-beryllium alloy "AlBemet 162". The creep and stress rupture properties of the alloy were measured and discussed in comparison with that of ceramic particulate reinforced Al matrix composites. Fractography of the specimens were conducted and analyzed in connection with the measured creep and stress rupture properties.

4:20 PM

Microstructure Control of High Strength High Conductive Cu Based In-Situ Composites: *Hirowo G. Suzuki*[1]; K. Mihara[2]; S. Sakai[2]; K. Adachi[3]; [1]National Institute for Metals Japan; [2]Furukawa Electric Company Limited; [3]Sumitomo Metal Industries Limited

Development of high strength high electrical conductive Cu based in-situ composites is potentially useful in the electric and electronic industries. Microstructure control is useful to obtain high strength and high conductivity. Based on the Cu-15wt%Cr binary alloy, the effects of microalloying elements such as Zr, Fe and Sn were examined. Processing involves ingot making by induction heat melting, hot stage forging, solution treatment, cold rolling and aging. Heavy cold rolling is effective to refine the lamellar spacing of second phase (Cr fiber) and aging treatment gives precipitation strengthening and recovery of electrical conductivity. Dynamic recrystallization occurs in the copper matrix during heavy cold rolling more than h=5.0 (here, h=lnA0/A) in a Cu-15wt%Cr binary alloy, while banded structure is formed and higher strength is attained in the Cu-15wt%Cr-0.2wt%Zr alloys, indicating the difficulty of recovery. Age hardening is remarkable in this alloy due to the acceleration of Cr precipitation. The addition of 0.2 to 0.5wt% of Fe does show work softening. Fe is scavenged to the second phase of Cr during solidification. The effect of Sn is similar to that of Zr although the electrical conductivity drops more than the case of Zr. The tensile strength follows Hall-Petch type equation with lamellar spacing of Cr. Cr phase becomes quite ductile in the Cu matrix. In the conference, the microstructure control is emphasized to optimize the strength as well as electrical conductivity.

4:40 PM

Preparation and Mechanical Properties of Al-MgAl2O4 In-Situ Particle Composites: *P. C. Maity*[1]; S. C. Panigrahi[1]; P. N. Chakraborty[1]; [1]National Institute of Foundry and Forge Technology, P.O. Hatia, Ranchi 834003 India

Metal matrix in-situ particle composites are being developed in recent years with the objective of producing thermodynamically stable fine particulate reinforcements in the matrix having clean interface free from reaction products. In the present work, attempts have been made to produce Al matrix MgAl2O4 particle reinforced in-situ composites by addition of reactive oxide particles in an Al-2 Mg alloy. To prepare Al-MgAl2O4 in-situ particle composites, up to 2 wt% of Fe2O3, Cr2O3 and TiO2 particles were incorporated into Al-2 Mg alloy melt by vortex method. MgAl2O4 and MgO particles formed in all the composites by reaction between the oxide particles and the alloy melt. In addition, pure Fe, Cr and Mg2Ti2O5 also formed in the respective composites. The reaction between the oxide particles and Al-2 Mg alloy melt were nearly complete. The microhardness of the composites improved due to dissolution of the reduced elements such as Fe, Cr etc. to limited extent. The reduction in microhardness in a few composites is associated with the presence of porosity and depletion of Mg from the matrix. Additional factor to increase the hardness of the composites is the dispersion of MgAl2O4 and MgO particles. Porosity resulted in poor tensile strength, except where the microhardness was substantially improved. Elongation % is higher than that of the base alloy for all the composites probably due to depletion of Mg from the matrix and presence of microporosity in the structure.

5:00 PM

Continuous Fiber Metal Matrix Composites at 3M: *Herve Deve*[1]; T. L. Anderson[1]; J. P. Sorensen[1]; S. R. Holloway[1]; [1]3M, Met. Matrix Comp., 3M Center Bldg. 60-1N-01, St. Paul, MN 55144-1000 USA

3M R&D efforts on continuous fiber metal matrix composites will be reviewed. In particular, the development of aluminum/alumina fiber composites wires for high voltage power transmission lines will be discussed.

Surface Engineering in Materials Science I: Coating/Films Characterization (C)-II

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

Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; John Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA

Tuesday PM Room: Canal B
March 14, 2000 Location: Opryland Convention Center

Session Chairs: D. L. Cocke, Lamar University, Gill Chair of Chem. and Chem. Eng., Beaumont, TX 77710 USA; L. J. Maksymowicz, University of Mining and Metallurgy, Instit. of Elect., Kraków 30-059 Poland

2:00 PM Invited

High Energy and Spatial Resolution XPS Analysis of Surface-Modified and Heterogeneous Polymers: *Julia E. Fulghum*[1]; [1]Kent State University, Chem. Dept., Kent, OH 44242 USA

Surface analysis of polymers has historically been viewed as being both of particular interest and of special difficulty. XPS is particularly useful in the analysis of polymers since chemical information can be acquired, frequently with minimal damage to the sample. Recent developments in XPS instrumentation have enhanced the analysts ability to acquire interpretable chemical data from samples of increasing complexity. Along with these developments, however, come increasing demands for analysis of multicomponent or multilayered samples with ever-decreasing feature sizes. This talk will focus on the use of angle-resolved, small area, and imaging photoelectron spectroscopy for the analysis of complex polymer surfaces. The use of derivatization methods to enhance XPS sensitivity will also be discussed. Both core and valence band photoelectron spectra will be used in the evaluation of surface modifications. Lateral and vertical heterogeneities in polymer blend samples will be demonstrated using small area and imaging XPS in correlation with imaging FTIR and phase-contrast AFM. This work has been partially supported by NSF (DMR89-20147), Dow Chemical and 3M.

2:25 PM

Elemental and Chemical Identification of Sub-Micron Metal Precipitates in Silicon Using Synchrotron-Based X-Rays: *Scott McHugo*[1]; [1]Lawrence Berkeley National Laboratory, Adv. Light Source Ctr., Mail Stop 2-400, Berkeley, CA 94720 USA

Metal impurity precipitates in silicon were studied with a focus on the ability to retain metal impurities away from the active device region of integrated circuits by use of oxygen precipitates and their growth-related defects in the bulk of the material. With x-ray absorption spectromicroscopy (m-XAS), we have characterized the chemical state of sub-micron scale Cu and Fe impurity precipitates in silicon. Furthermore, with x-ray fluorescence microscopy (m-XRF), we have studied the dissolution rate of metal precipitates in silicon as a function of thermal treatment. Based on our results, we present theoretical analysis of metal precipitate stability in silicon and discuss the feasibility of metal contamination of device regions.

2:45 PM Invited

Spectroscopic Ellipsometry Measurements of Thin Metal Films: *Harland G. Tompkins*[1]; [1]Motorola Inc., Motorola Labs., Physical Sci. Rsch. Labs., Tempe, AZ 85224 USA

Optical methods are used to determine the thickness of thin metal films, with emphasis on spectroscopic ellipsometry and transmission. We discuss the conditions where this is possible and how to determine the optical constants for the material. The determination of the thickness of each of two metals in a bi-metalic stack is discussed. Finally, by measuring thickness with these methods and measuring weight-gain, we determine the density of platinum deposited by evaporation and deposited by a simple sputter deposition method. The resulting optical constants suggest that the microstructure of films from the two different methods will not be the same and x-ray diffraction and sheet resistance measurements verify that this is the case. Specifically, the significantly lower extinction coefficient of the sputter-deposited films correlates with a higher sheet resistance.

3:10 PM

Gas Adsoption on Highly Diffusing Surfaces of (Sn,Ti)O2 Thin Films: *M. Radecka*[1]; K. Zakrzewska[1]; [1]University of Mining and Metallurgy, Al.Mickiewicza 30, Cracow 30-059 Poland

Recently, it has been demonstrated [1] that (Sn,Ti)O2 thin films prepared by rf reactive sputtering from metallic targets are promising candidates for gas sensing devices. The sensor performance, especially its sensitivity, can be greatly improved by increasing the density of centers active for gas adsorption. Despite a quite well known method such as an appropriate doping or incorporation of catalysts, a simple creation of large surface-to- volume ratio seems to solve this problem. In general, thin oxide films grown in the reactive process, i.e. in the presence of oxygen, have smooth surfaces. The post-deposition annealing enhances the surface roughness only to some extent. However, the post-deposition oxidation of previously grown metal films results in formation of highly diffusing surfaces of (Sn, Ti)O2. This method known as RGTO (rheotaxial growth and thermal oxidation) has proved to be efficient for quite a number of oxide compounds [2]. The key feature of this technique is the substrate temperature higher than the melting point of deposited metal or alloy. In the present work we show that very rough samples could be obtained independently of the substrate temperature as long as the initial state is metallic. The light scattering experiments in the ultraviolet, visible and near-infrared ranges were performed in order to study the surface development. The correlation was found between the film morphology as determined by SEM and the level of the diffused reflection of light. Electrical resistivity measurements in changing gas atmospheres were used to study gas adsorption processes. [1] M. Radecka, K. Zakrzewska, M. Rekas, SnO2-TiO2 solid solutions for gas sensors, Sensors and Actuators B 47(1998) 194. [2] G. Sberveglieri, G. Faglia, S. Groppelli, P. Nelli and A. Camanzi, A new technique for growing large surface area SnO2 thin films (RGTO technique), Semicon. Sci. Technol. 5(1990) 1231.

3:30 PM Break

3:40 PM

Humidity Characteristics of Thin Metal Oxide-Zeolite Films: *Jun-ichi Kodama*[1]; [1]Kinki University, Elect. Dept., 3-4-1 Kowakae, Higashi-Osaka, Osaka 577-8502 Japan

It is earnestly demanded for producing sensitive, stable and reproducible thin film humidity sensor of which resistance can respond quickly in the humidity atmosphere. It was found that the mixture of compound zeolite and metal oxide thin films such as NiO, CuO and Fe2O3 fabricated by evaporation, exhibit a good humidity characteristics. Compound zeolite itself exhibits humidity characteristics. But, it is not desirable to use it as a hygrometer by itself because of its high resistivity and its hygroscopic swelling. Metal oxides above mentioned not only prevent those troubles but also have a function to produce a conduction carrier. I will report in the paper about (1) fabrication method including how to decrease a film resistance by using surface processing, (2) humidity sensitivity by means of resistance measurement which changes from the order of 105 Ohm in 30%RH to the order of 102 Ohm in 80%RH and (3) stability and reproducibility.

4:00 PM

Construction of a New C-H-O Ternary Diagram for Diamond Deposition from the Vapor Phase: *Mahendra K. Sunkara*[1]; Sally C. Eaton[1]; [1]University of Louisville, Dept. of Chem. Eng., Louisville, KY 40292 USA

A new C-H-O ternary diagram based on radical species composition is constructed that distinguishes different regions for diamond deposition, non-diamond deposition and no deposition from the vapor phase. This construction is based on the steady state computations of gas phase and gas-surface chemistry for the data points presented in the original C-H-O ternary diagram by Bachmann et. al.1 based on feed gas compositions. The analysis of the computational domain indicates that the radical species composition do distinguish the three regions and helps explain the contradicting experimental data points with non-typical feed gas mixtures. The analysis also shows that the revised diagram based on radical species composition works only when carbon monoxide (CO) is treated as a neutral species. The effects of temperature and pressure on this new ternary diagram are also explained. Furthermore, the new ternary diagram is shown to be useful when analyzing the diamond deposition from the vapor phase inside trenches. Financial support came from NSF through CAREER award #CTS 9876251 and from KY NASA-EPSCoR program through KSGC fellowship (Sally Eaton). Reference:1. P.K. Bachmann, D. Leers and H. Lydtin, Diamond and Related Materials, 1, 1 (1991).

4:20 PM

An Analysis for Capillary Penetration Kinetics in Reactive Couples: *R. Asthana*[1]; [1]University of Wisconsin-Stout, Manu. Eng. Pgm., Menomonie, WI 54751 USA

The spreading kinetics in selected high-temperature reactive couples are revisited to identify flow regimes from plots of ln (droplet radius) versus ln (time) that are formally consistent with the classical models for simple liquids. It is suggested that while such a presentational approach could allow for identification by exclusion of complex flow regimes that are limited by interfacial reactions or diffusion, empirical models provide a more convenient approach to analyze practical spreading problems. It is shown that complex flow behavior of several reactive couples is compatible with an empirical relationship of the form: q(t) = q0 + q0 exp (B-At), where q(t) and q0 are instantaneous and equilibrium values of the contact angle, and B and A are system specific constants. This relationship is used in an analysis for capillary penetration kinetics for several cases of practical interest, such as, a homogeneous capillary, a stripwise binary capillary with or without gravitational effects, and a homogenous capillary with shrinking pores due to reaction. Model calculations for the penetration by molten Si of C and Si3N4 capillaries (single phase or binary) show that time-dependent contact angles retard the penetration kinetics. With a binary capillary, the analysis yields stepped infiltration profiles that match the predictions based upon Cassie's effective contact angles for both gravity-free and gravity-inclusive situations. The computational outcomes for the combined effects of shrinking pore and time-dependent contact angles are assessed in light of recent experimental work in reactive systems.

Ultrafine Grained Materials: Severe Plastic Deformation Processing: II

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; S. L. Semiatin, Wright Laboratory, Materials Directorate, Dayton, OH 45440 USA; C. Suryanarayana, Colorado School of Mines, Department of Metal and Materials Engineering, Golden, CO 80401 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM   Room: Polk A/B
March 14, 2000   Location: Opryland Convention Center

Session Chair: S. Lee Semiatin, Air Force Research Laboratory, Wright-Patterson Air Force Base, OH 45433-7817 USA

2:00 PM Invited

Grain Refinement of Titanium Aliminides by Equal Channel Angular Extrusion: *Shankar M.L. Sastry*[1]; Rabindranath Mahapatra[2]; Dennis Hasson[3]; [1]Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]Naval Air Warfare Center, Aircraft, Matls. Lab., Patuxent River, MD 20670 USA; [3]U.S. Naval Academy, Mech. Eng., 590 Holloway Rd., Annapolis, MD USA

With the objectives of producing sub micron grains and determining the beneficial effects of such sub micron grained microstructures on mechanical properties, several gamma+alpha2 based Ti-Al-X (X= Nb, Cr, Mn,Mo) alloys were processed by equal channel angular extrusion (ECAE) at 1200-1500K. 6-12 mm diameter cylindrical specimens were produced by multiples passes at different temperatures and were annealed at 1300-1600K. Microstructures of as processed and annealed specimens were determined by transmission and scanning electron microscopy. Hardnesses, bend strengths, and bend ductilities were evaluated as functions of the temperature, deformation rate, and number of passes. Whereas in conventionally processed materials, the grain sizes were 10-50 micrometers, the grain sizes were < 2 micrometers in ECAE processed and annealed conditions. Furthermore there was ample evidence for the break up and conversion of gamma|alpha2 lamellar morphology to equaixed gamma and alpha2 grains. The effects of microstructural modifications on mechanical properties were determined.

2:25 PM Invited

Ultrafine Grained Materials by Equal Channel Angular Processing (ECAE): *Gary E. Korth*[1]; Thomas M. Lillo[1]; Jenya Macheret[2]; John E. Flinn[3]; [1]Idaho National Engineering and Environmental Laboratory, Met. and Cer., P.O. Box 1625, Idaho Falls, ID 83415-2218 USA; [2]U.S. Department of Energy, Idaho Operat. Off., 850 Energy Dr., Idaho Falls, ID 83415-1225 USA; [3]University of Idaho, 3450 S. 35 West, Idaho Falls, ID 83402 USA

Ultrafine grains were obtained in several alloys by processing with ECAE. Materials processed were Copper Alloy 101 (OFHC), Copper Alloy 260 (70/30 brass), Nickel Alloy 270 (99.97% pure), high purity aluminum (99.999%), and Aluminum Alloy 1100. In all cases, ultrafine grains (<1um) were produced with multiple passes through the ECAE die. Mechanical property and microstructural analysis of as-processed and annealed material showed the grain stability to be much less in the high purity materials than was observed in the more "dirty" alloys. ECAE processing produces a very high level of stored energy in the microstructure which provides a strong driving force for grain growth at relatively low annealing temperatures if grain boundaries are relatively clean. Any dispersions or impurities tend to pin the grain boundaries and stabilize the ultrafine grains.

2:50 PM

Multiaxis Deformation Methods to Achieve Extremely Large Strains and Ultrafine Grain Steels: *Wayne C. Chen*[1]; David E. Ferguson[1]; Hugo S. Ferguson[1]; [1]Dynamic Systems Inc., 323 Route 355, P.O. Box 1234, Poestenkill, NY 12140 USA

Ultrafine grain size is often achieved by severe plastic deformation. A few techniques have been developed to achieve severe plastic deformation, such as equal channel angular (ECA) pressing/extrusion, torsion straining, and accumulative roll bonding (ARB) techniques. This paper will introduce multiaxis deformation techniques which can achieve extremely large strains with constant deformation volume. Two different types of hot deformation methods, each with a different number of deformation axis, are studied. They are two-axis deformation and three-axis deformation. The two-axis deformation can be fully restrained or unrestrained lengthwise. The three-axis deformation has no restraint. The bulk volume of multiaxis full restraint compression specimens can be easily machined into mechanical test samples for mechanical property measurements and other studies. A plain carbon steel (AISI 1018) was studied using the Multi-Axis Restraint Compression system developed at Dynamic Systems Inc.. One micron grain size was achieved with the plain carbon steel. The ultimate tensile strength measured doubled, when compared to that of the conventionally hot rolled material.

3:10 PM

Fabrication of Ultra Fine Grained Bulk Iron through Mechanical Milling of Iron Powder: *Setsuo Takaki*[1]; Yuji Kimura[2]; [1]Kyushu University, Dept. of Matls. Sci. and Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan; [2]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Mechanical milling of metallic powders is a useful technique for giving ultimate severe deformation to the metal and producing nanosized grains within the powder particles. If the mechanically milled metallic powders were successfully consolidated to bulk without loosing the fine grained microstructure, we ought to be able to obtain ultra fine grained bulk materials. In this paper, the MMC process (Mechanical Milling & Consolidation process) will be introduced for iron as a technique to fabricate ultra fine grained bulk materials and be discussed on the Hall-Petch relationship of iron materials in the ultra fine grained region.

3:30 PM

Grain-Size Evolution in Nanocrystalline Fe during Mechanical Arttrition: *Michael Atzmon*[1]; Huahang Tian[2]; [1]University of Michigan, Dept. of Nucl. Eng. & Rad. Sci. and Matls. Sci. & Eng., Cooley Bldg./North Campus, Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109-2136 USA

We present a study of the evolution of grain size and strain in Fe powder during low-energy ball milling. For this method of inducing mechanical attrition, local heating by impact is negligible, so that the temperature and milling intensity can be controlled independently. Using the Warren-Averbach method, the grain size and RMS strain were determined as a function of milling time for temperatures between 298K and 523K and varying milling amplitudes. Average grain diameters as small as 7 nm were observed. A model was developed to describe the evolution of the grain size, based on a rate equation which includes grain refinement and simultaneous grain growth. The contribution to grain growth by nonequilibrium vacancies generated by deformation was incorporated, using concepts developed to model radiation-enhanced diffusion. The model expression fits well the temporal curves for all conditions, and the behavior of the fitting parameters as a function of temperature and milling intensity is consistent with the assumptions made. In particular, the activation energy for the kinetic coefficient for grain growth is negligible below 473K, in agreement with the dominance of nonequilibrium defects. A thermally activated contribution to the grain-growth term is noticeable above 473K. The fraction of the impact energy which is converted into grain boundaries or point defects is observed to decrease with the milling amplitude. An application of the results to observed non-monotonic precipitation in ball-milled supersaturated solid solution will be presented.

3:50 PM Break

4:00 PM Invited

Mechanisms of Ultrafine Grain Formation during Severe Plastic Deformation: *R. Kaibyshev*[1]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Present work is an overview of experimental data dealt with microstructural evolution during severe plastic deformation. Two techniques were used to deform bulk materials. A magnesium alloy Mg-5.8%Zn-0.65%Zr, aluminum alloy 2219 and 15Cr25Ti steel were strained at ambient temperature by using of Bridgeman anvil. Few aluminum alloys were processed by ECAE at enhanced temperatures. Evaluation of mechanical properties, X-ray examination and TEM observations were performed to reveal operating mechanisms of ultrafine grain formation. It was shown that stacking fault energy (SFE) and temperature strongly influence mechanism of nanocrystalline and submicrocrystalline grain formation. In general the microstructural evolution consists of three stages. At first stage the subdivision if initial grains into fragments occurs. In materials with low values of SFE a deformation twinning provides this subdivision at ambient temperature. In material with high values of SFE a formation of low energy dislocations structures results in grain separation. Dynamic recrystallization may occur at moderate temperatures and refinement of initial structure takes place. At second stage of microstructural evolution a single slip occurs in fine crystallites and a strong increase of lattice dislocation density is observed. A specific mechanism for ultrafine grain formation is operative. This mechanism is considered in details. Formed structure is highly non-equilibrium due to high density of extrinsic dislocations into grain boundaries. At third stage a recovery process occurs into boundaries of ultrafine grains. It leads to decrease of internal stresses. Effects of initial grain size and secondary phases on the mechanisms of ultrafine grain formation are discussed.

4:25 PM Invited

Severe Plastic Deformation of Ti-6Al-4V via Equal Channel Angular Extrusion: *David P. DeLo*[1]; S. Lee Semiatin[2]; [1]Extrude Hone Corporation, 1 Industry Blvd., Irwin, PA 15642 USA; [2]Air Force Research Laboratory, Matls. Process. and Process. Sci., Matls. and Manufact. Direct., AFRL/MLLM, Wright-Patterson Air Force Base, OH 45433-7817 USA

The equal channel angular extrusion process (ECAE) imposes severe plastic deformation throughout a bulk section of material without changing the overall dimensions of the workpiece. Incremental deformation occurs in the form of simple shear confined to a narrow zone. Under ideal conditions, the simple shear deformation occurs uniformly throughout the workpiece refining the microstructure and affecting crystallographic orientations. Physical models were used to study the effects of ECAE processing on Ti-6Al-4V billets having either a lamellar or an equiaxed alpha preform microstructure. Microstructural features resulting from ECAE performed at various temperatures and using various processing routes are compared. The relationships between material flow properties, processing conditions, macroscopic deformation during ECAE, and the resulting microstructures are described.

4:50 PM

Effect of Large-Strain Deformation Prior to Austenitization on Austenite Grain Size in 0.3%C-9%Ni Steel: *Tomoyuki Yokota*[1]; Tetsuo Shiraga[1]; Masakazu Niikura[1]; Kaoru Sato[2]; [1]Ferrous Super Metal Consortium of Japan, NKK Corp. Matls. & Processing Rsch. Ctr., 1-1 Minamiwatarida-cho, Kawasaki-ku, Kawasaki-shi 210-0855 Japan; [2]Ferrous Super Metal Consortium of Japan, NKK Corp. App. Tech. Rsch. Ctr., 1-1 Minamiwatarida-cho, Kawasaki-ku, Kawasaki-shi 210-0855 Japan

Fundamental research project to pursue ultra fine grain size below 1micron in steel is currently under way as “Ferrous Super Metal” project supported by NEDO (New Energy and Industrial Technology Development Organization). One effort within this national project is austenite grain size refinement, which is one of the most important factors for microstructural control in heat treatment. Cold or warm deformation prior to austenitization is known to have a beneficial effect on austenite grain size, and in this paper, the effect of large-strain deformation at a temperature range just below Ac1 transformation temperature on austenite grain size after austenitization was studied using 0.3%C-9%Ni steel. Because this steel has high hardenability, once reverse transformation occurs, austenite transforms martensite with extremely high hardness. Deformation up to 70% was done in a single pass by compression of cylindrical specimens using laboratory deformation simulator. Increase in reduction ratio up to 50% continuously refined austenite grain size down to 2 micron after subsequent reheating to austenitization temperature (700°C) by induction rapid heating. Drastic change in reverse transformation behavior was found for the reduction above 70%. Spontaneous reverse transformation was induced by such a large strain deformation even without subsequent reheating. It was revealed by TEM observation that austenite grain size of the specimen was remarkably refined down to around 0.5 micron, and it showed hardness of fully quenched martensite. Adiabatic heating due to deformation was suggested to contribute to such a spontaneous transformation. Transformation mechanism and required metallurgical condition for the spontaneous transformation will be discussed.

5:10 PM

Thermal Stability of Ultrafine Grained Ferritic Structure of Iron with Oxide Particles: *Yuuji Kimura*[1]; Satoru Nakamyo[2]; Hideyuki Hidaka[2]; Hideto Goto[2]; Setsuo Takaki[2]; [1]National Research Institute for Metals, Frontier Rsch. Ctr. for Struct. Matls., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Kyushu University, Matls. Sci. and Eng., Grad. School of Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan

Mechanical milling is very effective for charging a large strain into metals, because endless cyclic deformation can be performed through kneading reaction. Sufficient milling treatment finally causes an ultimate work-hardening based on the ultra grain refining to about few ten nm or less (charged true strain is more than 10). Moreover, we confirmed in ferritic stainless steel powders that a large amount of oxide particles, which are thermodynamically so stable, were not only dispersed but also decomposed during ultra grain refining of the matrix and then re-precipitated very finely on annealing. In this paper, iron powders with oxide particles were mechanically milled and consolidated to bulk materials. The thermal stability of ultra fine ferrite grain structures were then investigated in relation to the structural change and the Zener pinning effect of the oxide particles.