SYMPOSIUM HH

Superplasticity–Current Status and Future Potential

November 29 – December 1, 1999

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* Invited paper

SESSION HH1: SUPERPLASTICITY IN METALS
Chairs: Patrick B. Berbon and Zenji Horita
Monday Morning, November 29, 1999
Room 204 (H)

8:30 AM *HH1.1
COMPUTER SIMULATION OF GRAIN BOUNDARY CHARACTER IN A SUPERPLASTIC ALUMINUM ALLOY.
Terry R. McNelley, Department of Mechanical Engineering, Naval Postgraduate School, Monterey, CA; M. Teresa Perez-Prado, Applied Mechanics and Engineering Science, University of California - San Diego, La Jolla, CA.

The presence of high-angle grain boundaries in the microstructure is generally agreed to be an essential feature of fine-grain superplasticity. Such boundaries are needed in order to support grain boundary sliding and its accommodation during superplastic deformation. Recent studies have indicated that moderately misoriented boundaries (5-15 degrees) may play a critical role in alloys that exhibit continuous recrystallization but relatively little has been reported on the relationship between the grain boundary misorientation distribution and fine-grain superplasticity. In a polycrystalline material the grain boundary character must be described in terms of a probability distribution, e.g., the distribution of misorientation angles, rather than by a single parameter. Recently developed computer-aided electron backscatter pattern analysis methods have be used to obtain ample grain-specific orientation data during the investigation of grain boundary misorientation distributions in several superplastic aluminum alloys. For alloys that exhibit continuous recrystallization results have shown that bimodal grain boundary misorientation distributions are present in as-processed material and persist during subsequent annealing. Results of computer simulations of the misorientation distribution will be presented. These simulations are based on a model of the microstructure which assumes that deformation banding has occurred during prior deformation processing. The higher angle boundaries (>30 degrees) are assumed to develop in association with deformation banding while boundaries of lower misorientation (<30 degrees) are assumed to develop by dislocation reaction within the bands. Improved understanding of the grain boundary types associated with various microstructural transformation mechanisms will aid the design of processes to produce superplastic microstructures.

9:00 AM *HH1.2
SUPERPLASTICITY IN MAGNESIUM ALLOYS AND ITS COMPOSITES.
T.G. Nieh, Lawrence Livermore National Laboratory, CA; Toshiji Mukai, Hiroyuki Watanabe, Osaka Municipal Technical Research Institute, Osaka, JAPAN; Kenji Higashi, Osaka Prefecture University, Osaka, JAPAN.

There exist many potential opportunities for the use of magnesium alloys in automotive components. This is not only because magnesium has a relatively low density, which can directly and substantially reduce vehicle weight, but also its good damping characteristics, dimensional stability, machinability, and low casting costs. Recent advances in manufacturing technology further resulted in the development of magnesium-based composites. These composites have yet greater potential to be used in high-performance aerospace and automobile applications because of their superior dimensional stability, strength, and wear resistance. Superplastic forming is a viable technique to manufacture hard-to-form materials, such as a metal-matrix composites, into complex shapes. Limited data on superplasticity in magnesium-based composites are currently available. In this paper, the superplastic properties of several magnesium alloys and magnesium composites will be reviewed and compared. The role of the reinforcement on superplastic deformation mechanisms will be addressed. Special emphasis will be on the relationship between microstructure and deformation. A magnesium alloy ZK60 and its composites will be used as examples to illustrate the underlining principles leading to the observation of superplasticity. In this paper, two of the most recent challenges- high strain rate superplasticity and low temperature superplasticity, will be discussed and demonstrated in ZK60 alloy and its composites.

9:30 AM HH1.3
NUCLEATION AND EARLY GROWTH OF CAVITIES DURING SUPERPLASTIC DEFORMATION.
A.K. Ghosh and D-H. Bae, Dept of Materials Science and Engineering, The University of Michigan, Ann Arbor, MI.

Detailed studies of the early stages of cavity growth has revealed that cavities originate from preexisting damage within the alloy. Because the overcoming of surface tension (or sintering) forces requires only very small applied stress contrary to prior belief, almost no damage can truly sinter under tensile stresses. Thus the wide range of cavity sizes from nanometer to micrometer scale grow, but at very different rates. At low strain rates, diffusional accommodation to grain boundary sliding is large enough to slow the growth of most nanoscopic cavities, and the larger cavities contribute primarily to the overall void growth. This is consistent with a state of high strain rate sensitivity which tends to promote strain uniformity in the deforming material and minimize the growth of small damages. Thus while grain boundary sliding can accelerate cavity growth rate, this is true only if creep strength of the grain is large enough to tear open the boundary region i.e. when the test temperature is low or accommodation effects are poor. Furthermore, when only a few larger voids are present (as at low applied strain rates), they must grow to a larger size for the same imposed strain than that for high strain rates when many cavities are present. A new cavitation model explains such results and delineates the range of applicability of diffusional void growth model. The statistics of cavity size distribution points to continuous cavity nucleation by making yet finer cavities visible in the microscope.

9:45 AM HH1.4
SUPERPLASTIC DEFORMATION BEHAVIOR OF ZINC-ALUMINUM ALLOYS.
Tae Kwon Ha, Tae Shin Eom and Young Won Chang, Pohang University of Science and Technology, Dept of Materials Science and Engineering, Pohang, Kyungbuk, KOREA.

The superplastic deformation behaviors of a quasi-single phase Zn-0.3wt%Al and a microduplex Zn-22wt%Al eutectoid alloy were investigated in this study. A series of load relaxation and tensile tests were conducted at various temperatures ranging from RT (20°C) to 180°C. The investigation of deformed surface in a scanning electron microscope was also carried out and the result was compared with those of mechanical testing. Recently proposed internal variable theory of structural superplasticity has been applied. The flow curves obtained from load relaxation tests on superplastic Zn-22wt%Al eutectoid alloy were shown to consist of the contributions from interface sliding (IS) and the accommodating plastic deformation of grains. The IS behavior could be described as a viscous flow process characterized by the power index value of $M_g = 0.5$, suggesting the onset of intense phase boundary sliding (PBS). In the case of quasi-single phase Zn-0.3wt%Al alloy with the average grain size of 1μm, a large elongation of about 1400% was obtained at room temperature and the strain rate sensitivity parameter was about 0.4. Although relatively large-grained (10 μm) single phase alloy showed high value of strain rate sensitivity comparable to that of fine-grained alloy at very low strain rate range, grain boundary sliding was not expected from the analysis based on the internal variable theory of structural superplasticity. Careful examination on the surface of a specimen deformed at the strain rates ranging from 10^{-6}/s to 10^{-2}/s showed no evidence of the onset of grain boundary sliding.

10:30 AM *HH1.5
SUPERPLASTICITY IN Nb_3Al/Nb IN-SITU COMPOSITES.
S. Hanada, W. Fang, Tohoku University, Institute for Materials Research, Sendai, JAPAN.

Superplasticity in refractory intermetallic Nb_3Al has been investigated in this study, focusing on phase stability and microstructure. According to the Nb-Al binary phase diagram, the phase field of a Nb solid solution (Nbss) is broadened with increasing temperature; Nb-15.8 at%Al alloy consists of a single phase Nbss at 1900C and two phases of Nbss and Nb_3Al below about 1800C. When the alloy is homogenized at 1900C and annealed at 1300C, Nb_3Al precipitates preferentially into oriented directions, which produces lamellar microstructures. In contrast, equiaxed grain microstructures are evolved during isothermal forging at 1300C after homogenization at 1900C. Further annealing leads to decomposition from supersaturated Nbss to Nb_3Al without appreciable change in equiaxed grain microstructure and grain size. In tensile test at 1300C, fully annealed Nb-15.8 at%Al alloy exhibits no superplasticity, although it consists of small equiaxed grains of about several micron, while the as-isothermally forged alloy shows superplastic elongation over 900%. Microscopic observation of superplastic samples deformed to various strains revealed that volume fraction of Nb_3Al increases with increasing strain, and equiaxed grain microstructure is maintained after superplastic deformation. It is suggested from these observations that phase transformation plays an important role for accommodation of incompatibilities caused by grain boundary sliding in superplasticity in Nb_3Al/Nbss in-situ composites.

11:00 AM HH1.6
SUPERPLASTICITY OF NICKEL-BASED ALLOYS WITH MICRO- AND SUBMICROCRYSTALLINE STRUCTURES.
O.A. Kaibyshev, V.A. Valitov and Sh. Kh. Mukhtarov, Institute for Metals Superplasticity Problems, Ufa, RUSSIA; B.P. Bewlay and M.F.X. Gigliotti, General Electric Corporate Research and Development, Schenectady, NY.

This paper will describe the generation of micro- and submicro-crystalline structures in several highly alloyed Ni-based

alloys that are strengthened by phases, such as gamma prime, gamma double prime, and delta. The relationship between the superplastic behavior and the microstructure will be discussed. It was established that high strain deformation processing in the range of 0.9 to 0.6 of the homologous temperature results in the progressive reduction of the initial coarse-grained structure (>100 microns) to a range of finer structures including a microcrystalline (MC) structure (<10 microns), a submicrocrystalline (SMC) and a nanocrystalline (NC) structure (< 50nanometres) with increasing deformation. The influence of chemical and phase compositions on both dynamic and static recrystallization is considered, and their effect on grain refinement is described. The thermal stability of alloys with MC, SMC and NC structures has also been investigated. Further refinement of the microstructure to the SMC condition expands the deformation conditions under which superplastic behavior is observed. It was established that in dispersion-hardened alloys containing gamma prime, or gamma double prime plus delta, with SMC structure, superplasticity can be observed at temperatures 200-250 degrees centigrade lower than in alloys with MC structure. High-strain rate superplastic behavior was observed in Ni-based superalloys with a SMC structure. Features of the superplastic behavior that were observed in alloys with SMC structure will be described. Examples of the manufacture of complex shaped parts by local superplastic.

11:15 AM HH1.7
A TEM STUDY OF DYNAMIC RECRYSTALLIZATION IN A SUPERPLASTIC AL-4MG-0.3SC ALLOY. Lisa M. Dougherty, Ian M. Robertson, Univ of Illinois at Urbana-Champaign, Dept of Materials Science and Engineering, Urbana, IL; John S. Vetrano, Pacific Northwest National Laboratory, Richland, WA.

A thermomechanical treatment has been developed to produce an unrecrystallized microstructure in an Al-4Mg-0.3Sc alloy that will dynamically recrystallize when strained at 733 K. Under specific forming conditions, initially unrecrystallized tensile specimens have been deformed to engineering strains exceeding 300%. This superplastic response is attributed to dynamic recrystallization during the early stages of deformation and subsequent sliding along the grain boundaries. Since the strain-rate during dynamic recrystallization can be faster than typical superplastic forming rates, a high strain-rate can be used until recrystallization is complete and a lower rate afterwards to allow grain boundary sliding. The result is an overall reduction in forming time. This transformation from an unrecrystallized to a recrystallized state during superplastic forming has been studied by deforming initially unrecrystallized tensile specimens at 733 K to true strains of 0.1, 0.2, 0.4 and 0.8 and analyzing the resultant microstructures in each using TEM and orientation image mapping. In all specimens, a bimodal size distribution of fine (i.e., 15 to 30 nm), coherent, Al_3Sc particles and large (i.e., 0.1 μm diameter or greater), semi-coherent, Al_3Sc particles is present. The larger particles have been observed to pin dislocations and interact extensively with subgrain and grain boundaries. At true strains less than 0.4, the microstructure is dominated by subgrain boundaries. As strain increases, the density of subgrain boundaries decreases while the density of matrix dislocations increases and the dislocation distribution becomes more complex. To understand how these microstructural changes occur and the role they play in the superplastic behavior of this alloy, samples prepared from the incrementally strained specimens are being deformed at elevated temperature *in-situ* in the TEM. The results of these *in-situ* experiments will be presented and the processes observed will be related to the microstructure and mechanical behavior of the bulk tensile specimens.

11:30 AM HH1.8
EFFECT OF SN ADDITIONS ON SUPERPLASTICITY IN Al-Mg-Mn-Sc ALLOYS. C.H. Henager Jr., J.S. Vetrano, V. Gertsman and S.M. Bruemmer, Pacific Northwest National Laboratory, Richland, WA.

Minor additions of Sn, in the amount of 0.03 w/o, made to an Al-4Mg-1Mn-0.3Sc (w/o) alloy were observed to have substantial effects on the flow stress, strain rate sensitivity (m-value), elongation at failure, and failure mode during constant strain rate testing at 573K to 823K. Tensile test data is analyzed and combined with SEM, TEM, and OIM to show the effects of Sn on flow and fracture in these alloys. Sn additions were not observed to affect starting grain sizes, which were about 6 microns, but did have measurable effects on elongation and strain rate sensitivity at all test temperatures. Elongations increased from a strain of 1.96 to 2.16 at 823K and m-values were higher for the Sn-containing alloys in comparison to alloys without Sn additions. At all other temperatures, alloys containing Sn exhibited reduced elongations and slightly lower m-values. Companion testing of similar alloys but with larger grain sizes (about 30 μm) to prevent grain boundary sliding showed that Sn additions promoted cavitation processes at boundaries. The beneficial aspects of Sn additions to the fine-grained alloys at 823K are discussed from the perspective that Sn promotes increased grain boundary diffusion rates.

11:45 AM HH1.9
TEXTURE EVOLUTION DURING LOW TEMPERATURE SUPERPLASTICITY IN 5083 AND 5052 Al-Mg ALLOYS. S.W. Su, I.C. Hsiao and J.C. Huang, National Sun Yat-Sen University, Inst. of Materials Science and Engineering, Kaohsiung, TAIWAN.

Low temperature superplasticity (LTSP) was observed in the 5083 and 5052 Al-Mg base alloys after thermomechanical treatments (TMT). The highest LTSP elongation was 400%, occurring at 250°C and $1x10^{-3}s^{-1}$. The subgrain structures formed during TMT transformed to better defined subgrains to ~0.5 μm upon heating to 250°C. Further static annealing or superplastic straining at 250°C would produce well defined fine grains to 0.7-2 μm, depending on the annealing time or strain level. The textures in the as-TMT sheets contained a mixed texture components of Cu $\{112\}\langle111\rangle$, brass $\{110\}\langle112\rangle$ and S $\{123\}\langle412\rangle$. The grain misorientation consisted of a bimodel distribution. The portion of low angle boundaries (<15°) could be used as an index for the behavior of LTSP. For materials exhibiting LTSP over 250%, only 31% of the boundaries in the as-TMT condition was low-angeled, compared with 62% for materials failed at an elongation less than 150%. Static annealing at 250°C could sharpen but not alter the existing rolling textures. Superplastic loading would accelerate grain evolution rate. The as-TMT textures evolved into a random orientation distribution after LTSP straining to 50% at 250°C. After 100% elongation, around 10% of the boundaries were low-angled. It appears that grain boundary sliding has been active after 50% elongation at the low temperature of 250°C, not much different from the case of high temperature superplasticity occurred in some dynamically recrystallized Al alloys. There were consistently around 10-15% of the boundaries that were classified as coincident site lattices independent of the as-TMT, statically annealed or superplastically strained specimens; ~5% was related to $\Sigma 3^n$ twin types.

SESSION HH2: SUPERPLASTICITY IN INDUSTRY

Chairs: Amit K. Ghosh and Ruslan Z. Valiev
Monday Afternoon, November 29, 1999
Room 204 (H)

1:30 PM *HH2.1
THE DEVELOPMENT OF TITANIUM SUPERPLASTIC FORMING MANUFACTURING TECHNOLOGY FOR COMMERCIAL JETLINERS. Daniel G. Sanders, The Boeing Company, Seattle, WA.

The commercialization of Superplastic Forming (SPF) and Superplastic Forming with concurrent Diffusion Bonding (SPF/DB) has been the result of a partnership between researchers within worldwide universities, governments and industry. This paper will explore an airframe manufacturers perspective of today's SPF technologies and provide insight into future areas of development for commercial aviation.
The majority of the academic studies in this field have focused on the development of new metal alloys and other materials that exhibit superplastic behavior. Many aluminum and titanium alloys are now available for use on SPF products. A considerable effort has also been made to understand the mechanics involved in superplastic deformation. This has led to the formulation of accurate constitutive equations that can be used for Finite Element Analysis (FEA) to predict thinning. The FEA models provide the industrys manufacturing engineers with the necessary forming parameters to fabricate complex shaped SPF components.
The Boeing Companys SPF program has been primarily concentrated on the development of SPF as a new manufacturing process, the creation of manufacturing specifications, automation of the production equipment and innovative monolithic titanium structural designs for weight and cost advantages over built-up assemblies. This program has been very successful and the variety/complexity of new applications is increasing with every new airframe derivative. Although successful in many military applications, the industrialization of SPF/DB as both a design and manufacturing technology has been relatively slow. As we enter into the year two thousand, the next big challenge for aerospace metal forming researchers will be to solidify a robust SPF/DB process and integrate SPF/DB designs into our airframes.

2:00 PM *HH2.2
INDUSTRIAL APPLICATIONS OF ALUMINUM SUPERPLASTIC FORMING. Anthony Barnes, Superform USA, Inc., Riverside, CA.

Over the past 10 years, superplastic aluminum forming has advanced from a laboratory curiosity to a well established manufacturing process. Applications are now found in many and diverse markets including medical systems, specialist automobiles, railcars, architectural panels and all types of aircraft. This paper examines the reasons for success in these markets and explores future prospects including the potential for fast forming alloys currently under development.

2:30 PM HH2.3
MANUFACTURING ALUMINIUM SANDWICH STRUCTURES BY MEANS OF SUPERPLASTIC FORMING. Petteri Impio, Leinovalu Oy, FINLAND; Joe Pimenoff, Hannu Hanninen, Helsinki University of Technology, Laboratory of Engineering Materials, Espoo, FINLAND; Matti Heinakari, Kvaerner Masa-Yards Oy, FINLAND.

The present paper examines the possibilities of manufacturing large scale aluminium sandwich structures using superplastic forming. The materials tested were Mg-alloyed production quality aluminiums (Al 5083-O, Al 5083-H321) and Aluminium 1561. Tensile tests at elevated temperatures were performed in order to establish the suitability of the test materials for superplastic forming. The microstructural changes in the test material specimens were examined. Thereafter numerical simulation of the Al 5083-O forming process was conducted based on the tensile test results. The numerical simulation results were subsequently used to estimate forming parameters and the feasibility of manufacturing large scale structures by superplastic forming. The final results indicate that higher strains can be reached at higher temperatures for the test materials. Aluminium alloy 1561 exhibited the largest strains and AL 5083-H321 the smallest. The strains appeared to be temperature dependent but not much affected by the strain rate. Al 5083-O and Al 5083-H321 showed susceptibility for cavity forming whereas Aluminium 1561 formed relatively small numbers of voids. The numerical simulation indicates that Al 5083-O can be superplastically shaped using relatively low forming pressure.

2:45 PM HH2.4
THE MICROSTRUCTURE AND SUPERPLASTIC FORMING PROPERTIES OF CLEAN MECHANICALLY ALLOYED TITANIUM - TITANIUM BORIDE ALLOYS. A.P. Brown, R. Brydson, C. Hammond, Dept of Materials, School of Process, Environmental and Materials Engineering, University of Leeds, Leeds, UNITED KINGDOM; A. Wisbey, T.M.T. Godfrey, Structural Materials Centre, Defence Evaluation Research Agency, Farnborough, Hants, UNITED KINGDOM.

The most commonly used titanium alloy for commercial superplastic forming (SPF) is Ti -6% Al -4% V (Ti-6-4). Improved superplastic properties of this alloy are expected by grain refinement. Fine-grained material may be SPFd at lower temperatures and higher strain rates, whilst particulate reinforcement of the alloy matrix may control the significant grain growth observed during SPF, and limit the loss in tensile strength of the post SPF'd material. Mechanical alloying is a possible route to producing nanocrystalline material that would be an ideal precursor to such fine-grained, particulate reinforced Ti-6-4. The clean mechanical alloying of gas atomized Ti-6-4 with 0, 0.1 and 0.5 wt.% boron and the consolidation of this milled material by hot isostatic pressing (HIPing) at temperatures of 600 - 900°C has been carried out at the Defence Evaluation Research Agency (UK). It has already been shown that this process leads to the precipitation of a needle-shaped ceramic phase (TiB) within the matrix of the HIPd alloys and results in some grain-size refinement.
The present study has focused on the microstructural development of the Ti - TiB composite materials. The consolidated material has been analysed using X-ray diffraction, SEM and analytical TEM (EDX and PEELS). Mechanical testing has been undertaken at DERA.

3:30 PM *HH2.5
ORIGIN OF PRE-EXISTING CAVITIES IN SUPERPLASTIC SINGLE PHASE ALUMINUM MATERIALS. Z. Chen, N. Chandra, Florida State University, Dept of Mechanical Engineering, Tallahassee, FL.

The role of initial pre-existing cavities on the cavitation of superplastic material has been controversial in nature. Some researchers have reported the presence of pre-existing cavities due to the severe plastic deformation during thermo-mechanical processing of superplastic raw materials. Others argued that pre-existing cavities do not exist in superplastic sheet metals since they could not observe cavities in as-received materials. In this work, we present direct observations of pre-existing cavities in two Aluminum material systems, Al 5083 and Al 7475 alloys. Cavities were usually associated with inclusions and second phase particles, especially those with larger size. Optical and SEM micrographs of the cavities are presented and the results show that cavities are more prone in the direction of rolling, where the particles are aligned.
A finite element based simulation of the thermo-mechanical process of the base material reveals that cavities can originate during rolling around the particles. A single pass of rolling is numerical simulated and the results clearly show that cavities can orginate along the rolling direction in the front and back end of the particles and size of the cavity increases with the reduction ratio. Experimental obseravation corroborate the numerical results. A parametric numerical study of the shape and size of the particle on cavities are also presented.

4:00 PM HH2.6
SUPERPLASTIC-LIKE PHENOMENA IN HIGH VELOCITY DEFORMATION. Glenn S. Daehn, Hemant Panshikar, Subhrangshu Datta, Keith Crane and Vincent Vohnout, Department of Materials Science and Engineering, The Ohio State University, Columbus, OH.

Superplasticty is a means to an end. It simply facilitates the fabrication of hard-to-form component shapes. Superplastic forming comes only with significant economic and technical costs: specially processed materials that are seldom optimized for in-service performance must be used, and the cost of the forming operation is typically fairly high. Here we will show that high velocity forming (usually as accomplished by electromagnetic forming at velocities on the order of 100 m/s) produces many effects similar to superpalstic forming: formability can be dramatically improved, one sided dies can be used and multiple strategies exist for using high velocity forming in fabricating sheet metal components. This presentation will focus on the current state of understanding with respect to using inertial effects to improve the formability of sheet materials. There are three basic factors that should be accounted for that may work together: 1) inertia tends to make deformation more uniform in sheet material stretched beyond the Considere limit, 2) die wall impact can produce an ironing-like effect that can improve formability in a wide range of geometries, and 3) electromagnetic and inertial forces can alter the effective deformation anisotropy of the material. Strategies for using these effects within the constraints of realistic sheet component shapes will be introduced.

4:15 PM HH2.7
REVERSE MODELLING OF SUPERPLASTIC DOME FORMING OF MACHINED SHEETS. Nihat Akkus, Toshihiro Usugi, Masanori Kawahara, Ken-ichi Manabe, Hisashi Nishimura Tokyo Metropolitan University, Faculty of Engineering, Hachioji-shi, Tokyo, JAPAN.

An analytical and experimental work on the superplastic bulge forming of machined sheets is presented in this study. Unlike previously employed incremental-iterative method, a reverse deformation model was used to estimate initial thickness distribution of the machined sheets from which a constant final thickness can be obtained when the bulged sheet shape is hemisphere. The reverse deformation model was obtained by modifying previously known models which were based on the axisymmetric membrane and the incremental strain theory. Bulge forming experiments were conducted for machined sheets of Al alloy, A5083, at about 530°C under constant pressure control mode. Result of simulation to estimate final constant thickness distribution agreed well with the experiment and it was confirmed that the reverse deformation model can be successfully applied to optimize the thickness distribution of the starting sheets to obtain the desired final thickness distribution of the free bulged hemispherical product. Key words: superplastic forming, reverse deformation model, hemispherical bulging, machined blank, constant thickness distribution

SESSION HH3: SUPERPLASTICITY IN CERAMICS

Chairs: T.G. Nieh and Taketo Sakuma
Tuesday Morning, November 30, 1999
Room 204 (H)

8:30 AM *HH3.1
GRAIN BOUNDARY ENGINEERING OF HIGHLY DEFORMABLE CERAMICS. Martha L. Mecartney, Department of Chemical and Biochemical Engineering and Materials Science University of California, Irvine, CA.

Grain boundaries in ceramics play a key role in the deformation process for superplastic ceramics at high temperatures. This presentation will highlight two approaches to creating highly deformable ceramics with a stable fine grain size using cubic zirconia as a model material. The first approach utilizes high viscosity grain boundary phases which provide for relatively easy grain boundary sliding but preserve grain boundary adhesion. The second approach employs second phase particles as pinning agents to minimize grain growth while allowing for a significant degree of grain boundary sliding. Results from compression and tensile tests at high temperatures along with room temperature mechanical data from these two-phase ceramics indicates some interesting anomalies. Enhanced rates of deformation do not correlate with enhanced ductility at high temperatures. Use of grain boundary compositions that enhance high temperature deformation can change the room temperature fracture mode from transgranular to intergranular and decrease the fracture toughness. However, for certain intergranular compositions, primarily those containing high valence cations, the mechanical properties are not significantly impacted by small additions of intergranular second phases. The mechanical properties of highly deformable ceramics can be maintained after 100% deformation at high temperatures.

9:00 AM HH3.2
CREEP DEFORMATION IN A 3MOL%Y_2O_3-STABILIZED TETRAGONAL ZIRCONIA. Koji Morita, Keijiro Hiraga Yoshio Sakka National Research Institute for Metals, Tsukuba-shi, Ibaraki, JAPAN.

The high temperature deformation behavior of a high purity yttria-stabilized tetragonal zirconia (Y-TZP) was examined by constant load creep tests in tension at 1673 K, at initial stresses of 15-80 MPa. During loading, the true tensile displacement was monitored optically between the targets made on the both ends of gauge section. The monitored creep rates were corrected against the instantaneous stress and grain size. At a stress range of 15-80 MPa, the correction yielded a stress exponent of 2.7. TEM observation for deformed specimens, which were rapidly cooled without the removal of creep load, revealed the evidence of intragranular dislocation activities. The dislocations were frequently observed at around the multiple grain junctions. The stress exponent and the TEM observation suggest that dislocation activities may play an important role in accommodation process. On the basis of these results, the deformation behavior in a high purity Y- TZP will be discussed.

9:15 AM HH3.3
SUPERPLASTICITY AND JOINING OF ZIRCONIA BASED CERAMICS. F. Guitérrez-Mora, A. Domínguez-Rodríguez, M. Jiménez-Melendo, University of Sevilla, Dept Física Materia Condensada, Sevilla, SPAIN; R. Chaim, Technion, Dept Materials Engineering, Halfa, ISRAEL; J.L. Roubort, Energy Technology Div, Argonne National Laboratory, Argonne, IL.

The high temperature mechanical behavior of alumina-zirconia composites with alumina volume fractions of 20, 60 and 80% and with different phase distributions has been studied as a function of the strain rate (10^{-7}-$10^{-4}s^{1}$), stress (5 300 MPa), and temperature (1250-1500°C). The results were analyzed using the standard creep equation. The stress exponent was between 1 and 2 and the activation energy was between 650 and 700 kJ/mol. Microstructural observations of samples deformed indicated that deformation was achieved mainly by grain boundary sliding (GBS). This microscopic characteristic has been used to join pieces of these materials. Two pieces of these materials are compressed at temperatures ranging from 1250 to 1350°C at crosshead speeds of 5 μm/mn to achieve a final strain of 5 to 10%. Scanning electron microscopy observations of the interface indicated that GBS was activated and the junction contained no cavities or cracks. Vickers indentations at the interface indicated that the interface was as strong as the matrix.

9:30 AM HH3.4
SUPERPLASTIC DEFORMATION OF HIGH PURITY YTTRIA STABILIZED TETRAGONAL ZIRCONIA CONTAINING 2 AND 3 MOL% YTTRIA. Min Z. Berbon, Terence G. Langdon, University of Southern California, Los Angeles, CA.

Two-step sintering was used to fabricate yttria stabilized tetragonal zirconia containing 2 and 3 mol% yttria with submicron grain size. High temperature deformation of both materials was investigated over a wide strain rate range. The deformation was found to be superplastic and characterized by a stress exponent dependence on stress. The results are shown to be in agreement with available literature data on similar materials, and consistent with a model proposed earlier that contribute the superplastic flow to interface-controlled Coble diffusion creep with the movement of cation ions as rate controlling.

9:45 AM HH3.5
CREEP BEHAVIOR OF A SUPERPLASTIC Y-TZP/Al_2O_3 COMPOSITE. Siari S. Sosa and Terence G. Langdon, Dept. of Materials Science, University of Southern California, Los Angeles, CA.

Creep characteristics of a high purity superplastic Y-TZP/Al_22O_3 composite were studied. The samples had a uniform initial grain size and shape distribution, and the phase distribution was also uniform. Creep tests were conducted in tension. The grain size and grain shape were measured after the creep tests. Results revealed the existence of a primary steady state region with a transition to steady state stage. The steady state creep rate decreased after large strains. Scanning Electron Microscopy observations revealed grain elongation to an extent determined quantitatively by the grain shape measurements. The results were compared with earlier studies found in the literature and analyzed using the interface reaction-controlled diffusion creep model. Some grain growth was observed after testing at the lower stresses.

10:30 AM *HH3.6
SOME ASPECTS OF PLASTIC FLOW IN SILICON NITRIDE. Tanguy Rouxel, Laboratoire "Verres et Céramiques", Université de Rennes I, FRANCE; Jacques Rabier and Xavier Milhet, Laboratoire de Métallurgie Physique, Université de Poitiers, FRANCE; Sébastine Testu, ENSCI, SPCTS, Limoges, FRANCE.

The different scales of plastic flow in silicon nitride were investigated either by indentation experiments and compression under hydrostatic pressure in the 20-850°C temperature range and by stress relaxation and creep above 1350°C. 1/3<11-20> and 1/3<11-23> dislocations were evidenced by Transmission electron Microscopy in the low temperature range. Elastic anisotropic effects were invoked to interpret the observations and the possibility of a/b phase transformation by cooperative shear of the structure has been discussed. Cross-slip events in {10-10} prismatic planes were observed at temperature as low as 20°C by Atomic Force Microscopy on micro-hardness indents. By increasing the temperature, the deviation plane becomes {11-20} prismatic planes. Above 1350°C, the creep strain could be fitted by the sum of a transient component. $\epsilon_1 = \epsilon^{[1-exp-(t/\tau_c)^{b_c}]}$, where τ_c reflects the duration of the transient creep stage and b_c is between 0 and 1, and a stationary component, ϵ_s-$A\sigma^n t$, where σ is the stress and n is the stress exponent. The increase of ϵ with temperature is interpreted on the basis of the formation of liquid intergranular phases above 1400°C by progressive melting of the some grains. A creep exponent of 1.8 was determined. A single value could hardly be given to the activation energy since an S-shape curve was observed in the In $_s$ versus 1/T plot, as for most glasses over large temperature ranges. The stress relaxation kinetics was found to follow the Kohlrausch-Williams-Watt expression: σ/σ_o-exp[-t/$\tau_r)^{b_r}$], where b_r ranges between 0 (solid state) and 1 (liquid state) and τ_r is a characteristic relaxation time constant. As in the case of glasses, τ_r decreases rapidly whereas b_r increases from about 0.2 to 0.7 as the temperature increases trom 1400 to 1650°C. But again, it is very difficult to get a single value for the activation energy from the in τ_r versus 1/T plot.

11:00 AM *HH3.7
GRAIN BOUNDARY STRUCTURE AND SLIDING OF ALUMINA BICRYSTALS. Y. Ikuhara, T. Watanabe, T. Yamamoto, T. Saito*, H. Yoshida and T. Sakuma, Department of Materials Science, Faculty of Engineering, The University of Tokyo, Tokyo, JAPAN. *Japan Fine Ceramics Center, Nagoya, JAPAN.

Alumina ceramics are expected as one of structural ceramics, and its mechanical properties are closely related to the grain boundary atomic structure which depend on the grain boundary characteristics. Therefore, an investigating of the grain boundary behavior using bicrystal experiments is really needed to understand the nature of the mechanical properties even in such ceramics. In the present study, alumina bicrystals with several kinds of grain boundary characteristics ($\Sigma3$, $\Sigma7$, $\Sigma13$, $\Sigma21$, $\Sigma19$, high and small angle grain boundaries) were fabricated by a hot joining technique. The grain boundary structures were then examined by high resolution electron microscopy (HREM) and analytical electron microscopy (AEM) to reveal their atomic structure and chemical composition. The stability of grain boundary structure was also evaluated by DV-Xα molecular orbital method, calculating the bond overlap population (BOP) and net charge (NC) as a function of the interplanar distance between two adjacent grains across the grain boundaries. The calculated result agrees well with the atomic grain boundary structure experimentally obtained from HREM analysis. In order to evaluate the grain boundary sliding behavior, creep tests were conducted to apply pure shear stress along the grain boundaries in the respective bicrystals by the compression test at high temperatures between 1470 and 1670K. It was found that the grain boundary sliding takes place, depending on the degree of coincidence site lattice (CSL) of the respective bicrystals. In the present paper, the relationships of grain boundary sliding, atomic structure, chemical composition, chemical bonding state and energy will be discussed in detail.

11:30 AM HH3.8
COMMONALITY AND SPECIALTY OF GRAIN BOUNDARIES IN OXIDES AND NON-OXIDES CERAMIC SYSTEMS WITH AND WITHOUT AMORPHOUS FILMS. Hui Gu, JST Corporation, Ceramics Superplasticity Project, Nagoya, JAPAN.

Spatially-resolved EELS analysis has given quantitative information on grain boundary structure, composition and chemical bonding in three ceramic systems which have demonstrated superplasticity in decreasing degrees: 3Y-TZP, sintered Si_3N_4 and HIPed SiC, all of sub-micron grain size. Although only Si_3N_4 reveals clear amorphous films at grain boundaries by HREM, the detected amount of silicon, yttrium and oxygen segregants in 3Y-TZP are sufficient to form a layer of a half nm all by themselves, and it is likely that them also form a film at elevated temperatures. This picture is supported by the correlation of deformation and segregation behaviors by SiO_2 doping. On the other hand, no silicate phase was detected at grain boundary in SiC system while it is B, C that play the major role for grain boundary sliding. The liquid phase sintered SiC which did not show superplasticity does have films at grain boundaries, but such alumina based films were not stable in structure and/or composition at high temperature and thus hardened the material. In drawing common picture from these systems, it is concluded that a stable amorphous structure at grain boundary, preferably the flexible silicate structure, is crucial to enable superplasticity to occur in ceramic systems.

11:45 AM HH3.9
Abstract Withdrawn.

SESSION HH4: OTHER TECHNIQUES INCLUDING SEVERE PLASTIC DEFORMATION

Chairs: Oscar A. Kaibyshev and M. L. Mecartney
Tuesday Afternoon, November 30, 1999
Room 204 (H)

1:30 PM HH4.1
CAVITATION AND FAILURE OF Ti AND Ti-6Al-4V DURING TRANSFORMATION SUPERPLASTIC STRAINING.
Christopher Schuh, David C. Dunand, Northwestern University, Department of Materials Science and Engineering, Evanston, IL.

During thermal cycling through the α/β phase transformation under the action of a small external biasing stress, Ti and its alloys exhibit a deformation stress exponent of unity and achieve superplastic strains. We report tensile experiments on Ti and Ti-6Al-4V aimed at understanding the failure processes in these materials. The development of cavities is assessed as a function of superplastic strain, and compared with data acquired during isothermal creep under otherwise similar conditions. Differences in the cavity development of unalloyed Ti and the Ti-6Al-4V alloy are discussed in light of the transformation characteristics, and implications for superplastic forming by transformation superplasticity are highlighted.

1:45 PM HH4.2
EFFECTS OF CRYSTALLOGRAPHIC TEXTURE ON INTERNAL STRESS SUPERPLASTICITY INDUCED BY ANISOTROPIC THERMAL EXPANSION. K. Kitazono, E. Sato and K. Kuribayashi, The Institute of Space and Astronautical Science, Kanagawa, JAPAN; R. Hirasaka and T. Motegi, Chiba Institute of Technology, Chiba, JAPAN.

Polycrystalline materials having crystallographic anisotropy show internal stress superplasticity under thermal cycling conditions. It happens since the internal stress induced by the mismatch of the thermal strain between the neighboring grains accelerates the macroscopic creep deformation by an externally applied stress, and the resulting average strain rate is proportional to the applied stress. Some qualitative models enabled to explain the appearance of the linear creep at low stresses, but no quantitative estimation of the thermal cycling creep rate has been carried out. The authors developed a new quantitative model based on continuum mechanics including the effects of crystallographic texture. The proposed equation was experimentally verified through the thermal cycling creep tests using polycrystalline zinc having the deformation texture, and agrees well with the experimental results.

2:00 PM *HH4.3
GRAIN REFINEMENT OF ALUMINUM USING EQUAL-CHANNEL ANGULAR PRESSING. Zenji Horita, Kyushu Univ, Dept of Materials Science & Eng, Fukuoka, JAPAN; Minoru Furukawa, Fukuoka University of Education, Dept of Technology, Munakata, JAPAN; Minoru Nemoto, Kyushu Univ, Dept of Materials Science & Eng, Fukuoka, JAPAN; Terence G. Langdon, Univ of Southern California, Dept of Materials Science, Los Angeles, CA.

Equal-channel angular (ECA) pressing is a procedure whereby the grain size of polycrystalline materials may be reduced to the submicrometer level. Therefore, this processing method has the potential for producing materials which may exhibit superplasticity. This paper discusses the various factors which affect the development and evolution of the microstructure produced by ECA pressing. Specifically, it examines (i) the influence of the total strain which is determined by the number of passes through the die, (ii) the effect of the strain imposed on each passage through the die which is determined primarily by the angle between the two parts of the channel and (iii) the effect of rotation of the sample between repetitive pressings. As will be demonstrated, these various factors provide an opportunity to optimize the development of a homogeneous microstructure consisting of an array of equiaxed grains separated by high angle grain boundaries.

2:30 PM *HH4.4
HIGH RESOLUTION EBSD ANALYSIS OF SUBMICRON GRAINED SEVERELY DEFORMED ALUMINIUM ALLOYS.
Phil B. Prangnell, Ali Gholinia, Jacob R. Bowen, Jody S. Hayes, Manchester Materials Science Centre, UMIST, Manchester, UNITED KINGDOM.

It is well known that severe deformation, using techniques like ECAE and reciprocating extrusion, can lead to alloys with a fine grain size and improved superplastic properties. Such methods often have non-conventional strain paths. Previously the deformation structures of severely deformed alloys have been studied in the TEM. However, the deformed state can be inhomogeneous on a scale of the original grain size, and with this technique it is very difficult to obtain statistically significant quantitative date over representative areas. It has been variously reported that severely deformed alloys can recrystallise, discontinuously and continuously, and undergo enhanced grain growth on annealing. This range of observations is probably related to the poor current understanding of the effect of different processing methods on the homogeneity of the deformed state, prior to annealing. The introduction of FEG-SEM's has recently lead to an improvement in the resolution of EBSD systems and means that this powerful technique can now be applied to analysing submicron cellular deformation structures. EBSD allows the misorientations and the distribution of high and low angle boundaries to be determined. During severe deformation it has been found that submicron grains are formed by the process of grain subdivision on the scale of the cell, or Taylor lattice, blocks. Even after very high plastic strains ($\sim$ 10) the deformation structure is generally inhomogeneous, on a larger scale than has previously been reported and is a strong function of the strain path, processing temperature, and alloy composition. For example, alloys deformed with cyclic redundant strains typically develop a stable deformation pattern within the grains, resulting in bimodal grain structures comprised of submicron and larger 4-10mm grains. It has been shown that in general the most effective way of forming a submicron-grained alloy by severe plastic deformation is to maintain a constant strain path, lower the processing temperature, and increase the solute content.

3:30 PM *HH4.5
SPD PROCESSING AND SUPERPLASTICITY IN ULTRAFINE-GRAINED ALLOYS. Ruslan Z. Valiev, Ufa State Aviation Technical University, Institute of Physics of Advanced Materials, Ufa, RUSSIA.

Methods of Severe Plastic Deformation processing, namely, equal channel angular pressing, severe torsion straining and others, can lead to a strong refinement of microstructure and formation of ultrafine-grained (UFG) nanostructures in metallic materials; therefore, there is a potential to achieve their enhanced superplastic properties. However, attaining such properties is a complex problem, which depends on different processing and microstructural parameters. This paper focuses on the relationship: SPD processing-UFG structures-new superplastic properties, for several alloys and intermetallics. It has been shown that for microstructures of SPD materials it is typical to have a presence of not only very small grain sizes, but also specific defect structures, high internal stresses, crystallographic texture and often a change of a phase composition. From the other hand, these microstructural parameters are associated with details of SPD processing (applied method, processing routes, temperature, strain and strain rates and others). The examples of attaining enhanced superplasticity at relatively low temperatures and high strain rates in SPD alloys are shown. The origin and mechanisms of superplasticity in the processed ultrafine-grained alloys are considered and discussed as well.

4:00 PM HH4.6
MODEL OF DEFORMATION DURING EQUAL-CHANNEL ANGULAR PRESSING. Patrick B. Berbon, Rockwell Science Center, Structural Metals Division, Thousand Oaks, CA; Minoru Furukawa, Fukuoka Univ of Education, Dept of Technology, Munakata, JAPAN; Zenji Horita, Kyushu Univ, Dept of Materials Science and Engineering, Fukuoka, JAPAN; Minoru Nemoto, Sasebo National College of Technology, Sasebo, JAPAN; Terence G. Langdon, Univ of Southern California, Dept of Materials Science and Mechanical Engineering, Los Angeles, CA.

In the past few years, the equal-channel angular (ECA) pressing technique has allowed remarkable achievements in the production of submicrocrystalline (SMC) materials with superb superplastic properties, particularly at low temperatures and high strain rates. Although some of the observed mechanical properties can be explained by the small grain size resulting from the process, other results are more puzzling. It appears they are caused by the specific mode of deformation occurring during ECA processing. In this paper,

we are reviewing the specificity of this metal working technique, and we are proposing a possible deformation mechanism to explain the observed microstructures, their thermal stability, and the superb superplastic properties.

4:15 PM HH4.7
SUPERPLASTIC BEHAVIOR OF AN ALUMIUM-BASED ALLOY AFTER EQUAL-CHANNEL ANGULAR PRESSING. Jingtao Wang, Dept of Metallurgy, Xi'an Univ of Architecture & Technology, P.R. CHINA; Patrick B. Berbon, Dept of Materials Science, University of Southern California, Los Angeles, CA. (Now at Structural Metals Division, Rockwell Science Center, Thousand Oaks, CA); Yuzhong Xu, Lizhong Wang, Dept of Metallurgy, Xi'an Univ of Architecture & Technology, P.R. CHINA; Terence G. Langdon, Depts of Materials Science & Mechanical Engineering, University of Southern California, Los Angeles, CA.

It is recognized that superplastic deformation requires a very small and stable grain size (typically $<$ 10 micrometers). A further reduction in grain size, to submicron level or lower, offers the potential advantages of increasing the strain rate and/or decreasing the temperature associated with superplastic flow. This paper reports investigations on microstructure and tensile behavior at elevated temperatures of a submicrometer grained Al-3Mg-0.5Zr alloy processed by Equal-Channel Angular pressing (ECAP) up to an equivalent true strain of ~8.

4:30 PM HH4.8
THE EFFECT OF EQUAL-CHANNEL ANGULAR PRESSING ON THE SUPERPLASTIC PROPERTIES OF COMMERCIAL ALUMINUM ALLOYS. Sungwon Lee, Terence G. Langdon, Univ of Southern California, Dept of Materials Science and Mechanical Engineering, Los Angeles, CA.

Equal-channel angular (ECA) pressing was used to develop very fine grain sizes in two commercial aluminum alloys, 2024 and Supral-100 alloy. ECA pressing was performed at room temperature and elevated temperatures for both alloys and new pressing routes were also considered. Tensile tests were conducted on both pressed and unpressed samples for each alloy to evaluate the effect of ECA pressing. For both alloys, it was revealed that the optimum superplastic conditions are changed by ECA Pressing, i.e. the optimum superplastic temperature decreases and the optimum superplastic strain rate increases. In addition, high strain rate superplasticity was obtained for the Supral-100 alloy regardless of the variation of pressing condition.

4:45 PM HH4.9
PROCESSING BY EQUAL-CHANNEL ANGULAR PRESSING: POTENTIAL FOR ACHIEVING SUPERPLASTICITY. Terence G. Langdon, Univ of Southern California, Dept of Materials Science, Los Angeles, CA; Minoru Furukawa, Fukuoka Univ of Education, Dept of Technology, Munakata, JAPAN; Zenji Horita, Minoru Nemoto, Kyushu Univ, Dept of Materials Science & Eng, Fukuoka, JAPAN.

Equal-channel angular (ECA) pressing is a processing procedure whereby a very severe plastic strain is imposed on a sample without any change in the cross-sectional dimensions of the material. This processing method leads to a substantial grain refinement, producing grains which are within the submicrometer or even the nanometer scale. This paper discusses the potential for using this method to prepare materials for superplasticity. As demonstrated in this report, it is possible both to achieve superplastic deformation in selected materials after ECA pressing and also to extend the superplastic region so that it occurs at very high strain rates.

SESSION HH5: FUNDAMENTAL ASPECTS OF SUPERPLASTICITY

Chairs: Min Z. Berbon and Kenji Higashi
Wednesday Morning, December 1, 1999
Room 204 (H)

8:30 AM *HH5.1
NEWTONIAN FLOW IN BULK AMORPHOUS ALLOYS. J. Wadsworth, T.G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA.

Bulk amorphous alloys have recently attracted great attention because of their unique properties, e.g., superior strength and hardness, excellent corrosion resistance, reduced sliding friction and improved wear resistance, and easy formability in a viscous state. These properties, and particularly easy formability, should lead to applications in the fields of near-net-shape fabrication of structural components. Whereas large tensile ductility has generally been observed in the supercooled liquid region in metallic glasses, the exact deformation mechanism, and in particular whether such alloys deform by Newtonian viscous flow, remains a controversial issue. In this paper, existing data are discussed and an interpretation for the apparent controversy is offered. In addition, results from our recent study on an amorphous alloy (composition: Zr-10Al-5Ti-17.9Cu-14.6Ni, in at.%) are presented. Structural evolution during plastic deformation is characterized. It is suggested that the appearance of non-Newtonian behavior is caused by the concurrent crystallization of the amorphous structure during deformation.

9:00 AM *HH5.2
RECENT DEVELOPMENTS IN SUPERPLASTICITY. S.X. McFadden, R.S. Mishra, and A.K. Mukherjee, University of California, Dept of Chemical Engineering and Material Science, Davis, CA.

The recent developments in superplasticity in metals and intermetallics are emphasized in this presentation. The superplastic behavior of technologically significant titanium based intermetallic alloys as well as several nickel-base ordered alloys will be reviewed. The high strain rate superplasticity (HSRS) for various powder metallurgy based aluminum and non-aluminum alloys has been analyzed to determine the mechanism. The parametric dependencies of HSRS in composites are different from those observed in conventional aluminum alloys and mechanically alloyed alloys. The investigation on superplasticity has been extended to nanocrystalline metals, alloys, and intermetallics. An intriguing question has been whether the deformation mechanisms scale with grain size to nanocrystalline range or whether there are fundamental changes/ transitions. Superplasticity has been observed in a number of nanocrystalline materials processed by severe plastic deformation and electrodeposition at low temperatures. The important features include high strain rate superplasticity in an aluminum alloy, extensive strain hardening, and high flow stresses. A comparison of the experimental results with existing models shows that the deformation mechanisms for microcrystalline materials are not simply scaleable to nanocrystalline range. This research was funded by Grant No. NSF-DMR-9903321.

9:30 AM HH5.3
MODIFIED CONSTITUTIVE EQUATION OF SUPERPLASTICITY INCLUDING GRAIN GROWTH RATE. Hiroyuki Miyazaki, Takayoshi Iseki, Tokyo Institute of Technology, Dept of Inorganic Materials, Tokyo, JAPAN; Toyohiko Yano, Tokyo Institute of Technology, Research Laboratory for Nuclear Reactors, Tokyo, JAPAN.

Application of the well-known constitutive equation of superplastic behavior is limited since the equation can not analyze the deformation behavior associated with grain coarsening. Here we present a modified constitutive equation for superplasticity by incorporating the rate of grain-growth to predict superplastic behavior associated with grain growth at high temperature. With the equation, dependence of flow stress of superplastic material upon the test condition was calculated and was plotted against both strain rate and deformation temperature. To testify the modified equations, alumina composite with titanium carbide as minor phase was deformed at temperature from 1450 to 1650°C and at strain rate of 10^{-4}-$10^{-3}s^{-1}$. By elevating the test temperature, both the strain rate and the elongation of the Al_2O_3-TiC composite were increased, whereas the flow stress was decreased, even though grain coarsening was observed after the deformation. The plot of calculated flow stress explained the acceleration in strain rate of the composite without increment in the flow stress and implied that the stability of grain size during the deformation was not a necessary condition for superplastic deformation of the composite. The plot of calculated flow stress of 3Y-TZP could also analyze the increment in the stress exponent in slow strain rate region that is reported by other researchers. The validity of the plot was also confirmed with the superplastic SiC. The plot of the observed flow stress for liquid-phase sintered SiC agreed with the calculated tendency. Application of the plot of the calculated flow stress was presented in order to design superplastic ceramics.

9:45 AM HH5.4
ROLE OF VACANCIES AND SOLUTE ATOMS ON GRAIN BOUNDARY SLIDING. J.S. Vetrano, C.H. Henager, Jr., E.P. Simonen and S.M. Bruemmer, Pacific Northwest National Laboratory, Richland, WA.

It is necessary for grain boundary dislocations to slide and climb during the grain boundary sliding process that dominates fine-grained superplastic deformation. The process of climb requires either an influx of vacancies to the grain boundary plane or a local generation of vacancies. Transmission electron microscopy (TEM) observations of grain boundaries in superplastically deformed Al-Mg-Mn alloys quenched under load from the deformation temperature have revealed the presence of nano-scale cavities resulting from a localized supersaturation of vacancies at the grain boundary. Compositional measurements along interfaces have also shown an effect of solute atoms on the local structure. This is shown to result from a coupling of vacancy and solute atom flows during deformation and quenching. Calculations of the localized vacancy concentration indicate that the supersaturation along the grain boundary can be as much as a factor of five. The effects of the local supersaturation and solute atom movement on deformation rates and cavity nucleation and growth will be discussed in light of recent computer simulations of grain boundary sliding. *Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830

10:30 AM *HH5.5
GRAIN BOUNDARY DYNAMICS IN SUPERPLASTICITY AND GRAIN GROWTH. Fumihiro Wakai, Tokyo Institute of Technology (Japan Science and Technology Corporation), Yokohama, JAPAN; Naoya Enomoto, Tokyo Inst Tech , Mater Struc Lab, Yokohama, JAPAN, Hiroshi Ogawa, Natl Ind Res Inst Nagoya, Struc Form Dept, Nagoya, JAPAN.

The term Grain Boundary Dynamics refers to the motion of grain boundary. The grain boundary dynamics in grain growth occurs by grain boundary migration driven by interface energy. The grain boundary dynamics in superplasticity occurs by the relative motion of grains driven by the external force. In the former case grain switching is induced by annihilation of small grains. In the latter case grain switching is induced by relative motion of grains. The 3-dimensional simulation illustrates the grain boundary dynamics in both processes.

11:00 AM HH5.6
MICROSTRUCTURAL RELAXATION VIA GRAIN GROWTH DURING SUPERPLASTIC DEFORMATION. John R. Seidensticker, Sheldon M. Wiederhorn, National Institute of Standards and Technology, Gaithersburg, MD; Merrilea J. Mayo, The Pennsylvania State University, University Park, PA.

It has been widely recognized that superplastic deformation causes dynamic grain growth - an excess of grain growth over and above that which would occur during a similar anneal without deformation. This interrelation between grain growth and superplastic deformation suggests that the processes responsible for the two phenomena may be similar. In fact, the linear dependence of the dynamic grain growth rate on the applied strain rate can be predicted based solely on strain occurring as grain growth which is directionally biased due to the applied strain or stress. The present work takes this idea a step further, and considers that superplasticity occurs due to the coupling of grain deformation due to solid state diffusional creep with structural relaxation occurring as the recovery of an equiaxed grain shape due to normal grain growth processes. This theory not only explains the relationship between superplasticity and dynamic grain growth, but also explains the slight grain aspect ratios and the large anelastic back strains observed following superplastic deformation. Furthermore, combined with grain boundary migration rates which are limited due to solute drag or second phase pinning (factors which are almost always present in materials exhibiting superplasticity), both an apparent threshold stress and stress exponents greater than unity can be predicted as well.

11:15 AM HH5.7
SIMULATION OF MICROSTRUCTURAL EVOLUTION DURING SUPERPLASTIC DEFORMATION. Byung-Nam Kim, Keijiro Hiraga, National Research Institute for Metals, Mechanical Properties Division, Ibaraki, JAPAN.

The microstructural evolution during superplastic deformation is simulated for 2-dimensional polycrystalline aggregates. The deformation mechanism used in the simulation is the grain boundary diffusion and Lifshitz sliding. By analyzing the matter transport between grain boundaries, we calculated the dynamics of grain boundary movement under applied stresses. An atomic jump model for grain boundary migration is also employed in order to consider the effect of static grain growth during deformation. The relationship between mechanical properties and microstructural changes are investigated by the simulation. The simulation is carried out for three different loading conditions, that is, constant stress, constant strain rate and constant displacement rate. Under the condition of constant stress, the strain rate decreases gradually due to grain growth and the grain growth exponent is found to be 3 at low stress level. The aspect ratio increases initially and decreases gradually after maximum due to the decreasing strain rate. At high stress level, the stress exponent becomes slightly smaller than 1 by the influence of the increasing aspect ratio. The other microstructural features such as grain boundary sliding and grain rotation are also investigated.

11:30 AM HH5.8
ON THE RELATIONSHIP BETWEEN GRAIN BOUNDARY SLIDING AND INTRAGRANULAR SLIP DURING SUPERPLASTIC DEFORMATION. Askar Sheikh-Ali, Jerzy Szpunar, McGill Univ, Dept of Metallurgical Engineering, Montreal, CANADA; Hamid Garmestani, Lab for Micromechanics of Materials, NHMFL, Tallahassee, FL.

There are two different concepts concerning the relationship between grain boundary sliding and intragranular (crystallographic) slip during superplastic deformation. According to the first concept the slip and sliding are closely connected with each other. The experimental results obtained on Zn-Cu-Mn and Al-Zn-Mg-Zr alloys by Matsuki et al can be considered as a proof of the first concept. Strain-rate sensitivity exponents for slip and sliding are close to each other in the optimum region of superplasticity and differed markedly at the higher strain rates. The other concept considers sliding and slip as independent and competing processes. This concept can be confirmed by the results obtained on superplastic zinc and magnesium alloys when the change in direction of deformation from favorable for slip to unfavorable one increases the strain-rate sensitivity coefficient. This paper examines grain boundary sliding under the conditions of plastic strain incompatibility that is the most frequent case in polycrystalline materials and presents experimental results obtained on incompatible zinc bicrystals. It is shown that sliding along an individual boundary can be separated into two components that are dependent and independent on intragranular slip.

11:45 AM HH5.9
INFLUENCE OF THE GRAIN BOUNDARY GAMMA SURFACE ON SLIDING RESISTANCE. Richard J. Kurtz, Pacific Northwest National Laboratory, Richland, WA; Richard G. Hoagland, Washington State University, Department of Mechanical and Materials Engineering, Pullman, WA.

Grain boundary (GB) sliding produces changes in the structure of the GB and accompanying changes in GB energy. The variation of the GB energy within the 2D periodic cell of nonequivalent displacements constitutes the GB gamma surface. The shape of the gamma surface depends on the GB structures produced during relative translation of the two grains and it determines the sliding resistance, which is the stress required to move the system over the lowest saddle point along a particular path. In this paper we present the results of an atomistic study of the gamma surfaces for two types of boundaries in aluminum, one a low energy boundary, Sigma 11$<110>\{131\}$ symmetric tilt, and a higher energy boundary, Sigma 11$<110>\{252\}|\{414\}$ asymmetric tilt boundary. The former is characterized by deep valleys separated by fairly high-energy saddles. The latter boundary possesses a much more open structure and has a relatively flat gamma surface with relatively narrow saddles which is more characteristic of a large period boundary. However, the sliding resistance, as determined by application of an elastic band method, is found to be about the same for both and quite high. Consequently, GB dislocations are needed to enable sliding at low shear stresses. The gamma surface also plays a role in defining the GB dislocation core structure as we also discuss. Work supported by Materials Sciences Branch, Basic Energy Sciences, U.S. Department of Energy, under Contract DE-AC06-76RLO 1830.

SESSION HH6: HIGH-STRAIN-RATE SUPERPLASTICITY

Chairs: Terence G. Langdon and Tanguy Rouxel
Wednesday Afternoon, December 1, 1999
Room 204 (H)

1:30 PM *HH6.1
MATERIALS DESIGN AND PROCESSINGS FOR INDUSTRIAL HIGH-STRAIN-RATE SUPERPLASTIC FORMING. Kenji Higashi, Osaka Prefecture University, Department of Metallurgy and Materials Science, Sakai, Osaka, JAPAN.

The most important requirement for high-strain-rate superplasticity is the refinement of grain size in matrix of the materials. However, another problem associated with microstructural aspect is the role of cavitation in superplastic materials. The majority of the cavities found in superplastic materials at least either pre-exist, being located around hard particles as a consequence of defects introduced during the thermo-mechanical processing necessary to evolve the superplastic microstructure, or generated by grain boundary sliding during superplastic flow as a result of too high an imposed deformation rate for the deformation temperature and the current grain sizes. The high-strain-rate superplastic materials contain a relatively large amount of the particles with high possibility that the matrix-particles interfaces offer a nucleation site for cavities during superplastic flow. Thus, in the case of mass production in the industrial materials, for example, the high-performance-engine pistons with near-net-shape of the PM Al-Si based alloys developed by the high-strain-rate superplastic forging technology, extensive cavitation around the large and hard particles of Si could cause the premature fracture. This perennial problem, and the role of cavitation in high strain-rate superplastic materials with particles, are undoubtedly topics that should be considered to achieve enhanced superplasticity at high strain rates. Recently, a new model for high-strain-rate superplasticity was proposed in which it is assumed that grain boundary sliding is the dominant deformation process, as in normal superplasticity, but with the present of accommodation helper such as a liquid phase at the interfaces or boundaries serving both to relieve the stress concentrations due to sliding and to restrict the build up of internal cavitation and subsequent failure by cavity interlinkage. In the present work, the optimum materials design in microstructural control for the distribution of the accommodation helpers as well as the grain size refinement will be discussed for the high-strain-rate superplastic materials.

2:00 PM *HH6.2
HIGH STRAIN RATE SUPERPLASTICITY AND MICRO-STRUCTURAL DEVELOPMENT IN A 2124Al/SiC MMC, AND COMPARISON WITH AN ECA PRESSED ALUMINIUM ALLOY.
R.I. Todd, University of Oxford, Department of Materials, Oxford, UNITED KINGDOM; J.S. Kim, G.H. Zahid, P.B. Prangnell, Manchester Materials Science Centre, Manchester, UNITED KINGDOM.

Both superplastic deformation and the accompanying microstructural development in a 2124Al/18vol%SiCp metal matrix composite have been investigated. Mechanical property results comparable to those found in similar materials have been achieved, the optimum superplastic elongation being 430% at a strain rate of almost 0.1s-1. The anomalously high activation energy which has been observed in other studies was also reproduced. Differential scanning calorimetry showed that the optimum temperature in the current material was significantly below the temperature at which melting began, however, in contrast to some previous work. Microstructural investigation showed that both the optimum elongation, and the onset of the high activation energy regime, coincided with the temperature above which the majority of the intermetallic particles present at lower temperatures started to dissolve. It is concluded that the high apparent activation energy is an artefact caused by microstructural changes. The superplastic deformation of an ECA pressed zirconium-containing aluminium alloy is also reported for temperatures around the solvus for the majority of the intermetallic particles, and compared with the results from the MMC.

2:30 PM HH6.3
HIGH STRAIN RATE SUPERPLASTICITY OF Al_2O_{3f}/A6061 COMPOSITE PLATES.
A.B. Ma, J.Q. Jiang, J.H. Jiang, Southeast Univ, Dept of Materials Science and Engineering, Nanjing, P.R. CHINA; T. Imura, Aichi Institute of Technology, Dept of Mechanical Engineering, JAPAN; Y. Nishida, T. Imai, National Industrial Research Institute of Nagoya, Nagoya, JAPAN.

It was believed that fine grains are necessary for high strain rate superplasticity for metal matrix composites. Therefore, the materials used for superplastic tests are usually needed a hot extrusion process to fine the grains before producing tensile test samples. However, the size of the materials after a hot extrusion process is very small, so that it is difficult to produce a product by superplastic forming. The aim of this present work is to investigate the high strain rate superplasticity of Al_2O_3/A6061 composite plates produced via a hot rolling without a hot extrusion process. Before a hot rolling, a hot forging process was used. The results show that the composite plates appear superplastic behavior at three temperatures and correspondingly at three strain rates. And maximum total elongation of 121% of the composite plates along with the hot rolling direction was obtained at high strain rate 1.11 x 10^{-1} s^{-1} at 833 K. The relationship between tensile strain rate and test temperature is that the higher the temperature, the higher the optimum strain rate is.

2:45 PM HH6.4
FLOW STRESS AND ELONGATION OF SUPERPLASTIC DEFORMATION IN $La_{55}Al_{25}Ni_{20}$ METALLIC GLASS.
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Metallic glasses exhibit favorable engineering properties such as high strength, high hardness, excellent wear behavior and good corrosion resistance. Recently, many metallic glasses having high stability of the supercooled liquid against crystallization and high glass-forming ability have been discovered. We have previously reported that the new metallic glasses exhibit high-strain-rate superplasticity in the supercooled liquid state. The superplasticity of the metallic glasses made it possible to produce bulk parts by powder consolidation and to deform the bulk metallic glasses into near-net shapes. This contribution presents the mechanism of the superplasticity in metallic glasses on the basis of the experimental results in a $La_{55}Al_{25}Ni_{20}$ alloy.
The high-strain-rate superplasticity was ascribed to a Newtonian viscous flow where the strain-rate sensitivity exponent (m value) is unity. The Newtonian flow was observed at lower strain-rates in the supercooled liquid region above the glass transition temperature. However, the Newtonian flow transferred to a non-Newtonian one at higher strain-rates. The critical strain-rate increased with increasing temperature. The flow stress of the supercooled liquid was expressed very well by the stretched exponential function σ_{flow}=D$\dot{\epsilon}$ exp(H$^-$/RT) [1-exp(E/{$\dot{\epsilon}$ exp(H$^-$/RT)}$^{0.82}$)]. The Newtonian viscosity of the metallic glass exhibited an intermediate fragility between the strong glasses (SiO_2) and fragile glasses (O-terphenyl). Both the cystallization and the non Newtonian flow transition restricted the elongation to failure. The maximum elongation was obtained at the maximum strain-rate in the Newtonian flow region at each temperature. The ideal maximum elongation was estimated to be 10^5 to 10^7 % for the constant strain-rates and 10^3 to 10^4 % for the constant crosshead-speed, that were simulated by the incubation time of crystallization and the critical strain-rates of the Newtonian viscous flow.

3:30 PM *HH6.5
HIGH STRAIN RATE SUPERPLASTICITY OF ULTRA-FINE GRAINED Al-10wt%Ti AND Al-10wt%Ti-2wt%Fe ALLOYS.
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Ultra-fine microstructure consisting of equiaxed Al-grains and aluminide particulate was produced by a power metallurgy process using the gas atomized powders of Al-10wt%Ti-2wt%Fe alloy. High strain rate superplasticity(HSRS) has been investigated at 873 - 923K and strain-rates higher than 0.001 1/s in tension, and total elongation up to 500% was observed at strain-rate of 0.1 1/s. The strain-rate vs. flow stress behavior exhibited the typical aspects of HSRS, such as an increase of stra-rate sensitivity with incease in strain-rate and an apparent activation energy higher than that for lattice difusion in aluminum. The concept of threshold stress has been incorporated to illustrate the HSRS behavior, where the stress exponent of 3 is used to describe the experimental data. The determined threshold stress showed strong temperature dependency as in the case of a similarly processed Al-10wt%Ti alloy, which exhibited the stress exponent of 2 in the same testing condition. The solute drag mechanism has been postulated for the fine-grained Al-Ti-Fe alloy, and the role of Fe-addition to the Al-Ti alloy will be discussed.

4:00 PM HH6.6
CAVITATION BEHAVIOR OF COARSE GRAINED Al-4.5MG ALLOY EXHIBITING SUPERPLASTIC-LIKE ELONGATION.
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Cavitation behaviors have been investigated at a temperature from 613 K to 693 K and a strain rate from 10^{-5} to 10^{-2}/s for Al-4.5%Mg-0.05%Fe and Al-4.5%Mg-0.2%Fe alloys with grain size of 50 μm. The alloys exhibited large elongation-to-failure above 300% at a strain rate of 10^{-3}/s. Cavitation was increased by increasing iron content. Most cavities were nucleated at the interface between ferrous primary crystal and matrix and elongated parallel to the tensile direction. The critical diameter of the primary crystal, above which cavity is nucleated, was 1.5 μm at grain bundary and 0.5 μ at grain interior, which were very close to the theoretical results.

4:15 PM HH6.7
CAVITY FORMATION AND EARLY GROWTH IN A SUPERPLASTIC Al - Mg ALLOY.
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The evaluation of exact mechanisms of cavity formation and early growth is critically important in the prediction of internal damage. Because of the difficulty to ascertain them in superplasticity, the early stage of cavitation in a superplastically deformed Al-Mg-Mn-Cu alloy has been examined. Small cavities ($<$ 0.5 μm) were detected by scanning electron microscopy and the number of cavities per volume was monitored by image analysis through an optical microscope on an unetched surface. For conventionally processed superplastic alloy, pre-existing defects smaller in size compared to the resolution of optical microscope were present generally at the interface between particle and matrix. The results of tensile deformation in the temperature range of 450°C to 550°C at constant strain-rates of

$10^{-4}s^{-1}$ to $10^{-2}s^{-1}$ are presented. At the early stage of cavitation, most of the cavities are observed at the interfaces between particle and matrix and grow along the particle-matrix interface. This suggests that early cavity growth is by matrix/particle decohesion. The evolution of interfacial defect has rapid kinetics. The density of observable cavities increases with strain, suggesting that there is continuous "nucleation" of cavities. The number of cavities increases at higher strain-rates and at lower temperatures due to higher stresses which assist particle-matrix debonding in the regions of interfacial defects. Also the reduced strain-rate sensitivity and decreasing diffusional accommodation under these conditions cause rapid initial growth of nanoscale defects. The results are in good agreement with superplastic deformation mechanism.

4:30 PM HH6.8

ANALYSIS OF CAVITATION IN NEAR-γ TITANIUM ALUMINIDE DURING HIGH TEMPERATURE/SUPERPLASTIC DEFORMATION. Carl M. Lombard, Air Force Research Laboratory, Materials and Manufacturing Directorate, Processing Science Group, AFRL/MLLM, Wright-Patterson AFB, OH; Amit K. Ghosh, Univ of Michigan, Dept of Materials Science & Engineering, Ann Arbor, MI; S. Lee Semiatin, Air Force Research Laboratory, Materials and Manufacturing Directorate, Processing Science Group, AFRL/MLLM, Wright-Patterson AFB, OH.

The superplastic flow behavior of a near-γ titanium aluminide (Ti-45.5Al-2Cr-2Nb) was determined under uniaxial tension in the as-rolled or rolled-and-heat treated conditions (1177°C/4 hours or 1238°C/2 hours). Cavitation rates, failure modes and microstructural evolution were established via isothermal, constant strain rate tests conducted at 10^{-4} - 10^{-2} s^{-1} and temperatures between 900°C and 1200°C. This work examined the cavitation and its growth rate. After tension testing at a given temperature and strain rate, the as-rolled specimens developed fewer large size cavities than those in the heat treated specimens, possible due to the finer grain size in the as-rolled material. Cavities were found to predominately nucleate at γ/α_2 grain boundaries. Cavity growth was found to be largely plasticity controlled; the largest cavity size and the density of cavities increased with increasing strain or strain rate, and decreasing temperature. For all three initial microstructures, the optimum sheet forming temperature was identified as 1200°C, at which the lowest cavity growth rates and highest ductilities were observed. Experimentally derived cavity growth rates were compared with various equations that predict cavity growth rates as a function of strain rate sensitivity. Although the equations assume no coalescence and no nucleation of new cavities, which were experimentally observed, they were somewhat useful in predicting the actual cavity growth rates.