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ANNUAL TECHNICAL REPORT

AFOSR GRANT NO. AFOSR-86-00575

SUPERPLASTICITY - A FUNDAMENTAL INVESTIGATION ON DEFORMATION MECHANISM AND CAVITATION PHENOMENA

by

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Submitted to

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February 15, 1988



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ABSTRACT

Aluminum-lithium alloys and mechanically alloyed aluminum alloy have considerable potential for structural application in aerospace industry. These alloy systems are being investigated in this research program. The constitutive equation for superplasticity suggests that the strain rate is very sensitively dependent on the grain size. However, one usually encounters a range of grain sizes in the microstructure. The current investigation reveals that under such circumstances, in the classical superplastic domain i.e. region II, both coarse and fine grains deform in the typically superplastic mode, i.e., they do not reveal too much of intracrystalline slip activity in the grain interior. However, in the higher strain rates (region III), the coarse grains were shown to deform by transcrystalline mode, whereas the fine grains were found to deform by intercrystalline mode. This will have important implications on the theoretical modelling for superplasticity.

The mechanically alloyed IN90211 showed tensile elongations in excess of 500 percent at the fast strain rates between 1 and 10 per second at 475°C. This superplasticity-like behavior at high strain rate appears to be associated with the fine grained (0.5 micron) microstructure of this alloy. Grain boundary sliding was clearly observed. A stress exponent of three was obtained in the superplastic-like regime. Current theories of superplasticity do not fully account for the observed stress exponent. This continuing investigation involving measurement of threshold stress and transmission electron microscopic observation of the deformed microstructure are expected to shed more light on the micromechanism.

PART A A TOPOLOGICAL STUDY OF SUPERPLASTIC DEFORMATION IN AN AI-LI ALLOY WITH A BIMODAL GRAIN SIZE DISTRIBUTION

1. Introduction

The ability of a class of fine-grained polycrystalline materials to exhibit extremely large strains to failure, termed superplasticity, has been demonstrated not only in metallic alloys¹⁻³ but also, increasingly, in composites^{4,5} and ceramics^{6,7}. The mechanical properties of these materials may be characterized in terms of the following expression:

$$\sigma = B \varepsilon^{m}$$
(1)

where $\dot{\epsilon}$ is the strain rate, σ is the flow stress, m is the strain rate sensitivity and B is a constant incorporating the effect of temperature and microstructure on the flow stress. Superplastic metallic alloys exhibit a sigmoidal relationship between the flow stress and the strain rate and this flow behavior may be divided into three regions: a superplastic region II with $m \ge 0.5$ at intermediate strain rates, and the non-superplastic regions I and III with $m \le 0.3$ at low and high strain rates, respectively. This flow behavior is generally interpreted in terms of three different rate mechanisms three regions of deformation. controlling for the The rate-controlling mechanism for deformation in the superplastic region II has not yet been identified unambiguously; the various mechanisms proposed have been reviewed in detail by Kashyap and Mukherjee.⁸

Ghosh and Raj⁹ developed a model for superplastic deformation by recognizing that polycrystalline materials generally have a distribution of grain sizes. Their model postulated that fine grains will deform by a Newtonian viscous Coble diffusion creep mechanism and that coarse grains will

deform by an intragranular dislocation power-law creep mechanism. It is to be noted that the Coble creep rates are inversely proportional to the cube of the grain size and that the power-law creep rates are independent of grain size. Assuming that the fine grains and the coarse grains deform at the same strain rate (iso-strain rate assumption), Ghosh and Raj^{9,10} were able to produce sigmoidal plots of the variation in stress with strain rate similar to those reported in superplastic alloys, and the model was shown to be in agreement with some of the available experimental results.⁹⁻¹¹ This is a simple and attractive model for superplasticity by which it is possible to obtain the three regions of flow based on a suitable combination of two creep mechanisms and an appropriate grain size distribution. However, Gifkins¹² raised an important objection to the validity of the iso-strain rate assumption. In addition, the Ghosh and Raj model does not take into account explicitly the observed microstructural aspects of superplastic deformation such as grain boundary sliding, grain switching and the emergence of new grains on the surface. The present study was undertaken with a view to examining the possibility that, in a superplastic alloy, fine grains and

coarse grains may deform by different mechanisms.

2. Experimental Material and Procedure

A superplastic Al-2.6% Cu-2.4% Li-0.2% Zr-0.07% Fe (constituents by wt. %) was obtained from Reynolds Aluminum Co. in the form of sheets of thickness ~2.2 μ m. The alloy had been processed to statically recrystallize prior to superplastic deformation. Annealing the alloy for 11 hours at 773 K in air led to the development of a bimodal grain size distribution at the specimen surfaces, with coarse grains having dimensions of ~50 μ m and fine grains

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having dimensions of $-5 \ \mu$ m. This microstructure is well suited to studying the effect of grain size distribution on the deformation mechanisms operating in superplasticity.

Tensile specimens with gage lengths of 6.4 mm were machined with the tensile axes parallel to the rolling direction. Tensile tests were conducted in air at 723 K in an Instron machine modified to operate at constant true strain rates. The variation in flow stress with strain rate was determined by testing specimens over a range of strain rates between 10^{-5} and 10^{-1} s⁻¹. Two additional tests were conducted to examine the topological changes occurring during superplastic deformation at strain rates of 3×10^{-4} and 1.3 x 10^{-2} s⁻¹, corresponding to the optimum strain rate for s⁻¹ deformation in region II and in region III, respectively. For the topological study, the specimens were polished to a smooth finish and marker lines were inscribed on the surfaces parallel to the tensile axes by rubbing the specimen surfaces with a lens tissue containing 3 μ m diamond paste. The specimens were tested to strains of ~15% and removed from the Instron They were examined in a scanning electron microscope (SEM) and a machine. few grains on the specimen surface were identified. The specimens were then re-introduced into the Instron machine, pulled further and examined by SEM to study the topological changes occurring in the grains identified previously.

3. Results and Discussion

Figure 1 illustrates the experimentally determined variation in flow stress with strain rate on a logarithmic scale. This Al-Li alloy clearly exhibits a sigmoidal relationship between the flow stress and the strain rate with a maximum strain rate sensitivity of ~0.45 at strain rates between ~ 10^{-4} and 10^{-3} s⁻¹. The vertical arrows in Fig. 1 indicate the strain rates used in the topological study described below. At the optimum superplastic strain rate of 3 x 10^{-4} s⁻¹ in region II and at a strain rate of 1.3 x 10^{-2} s⁻¹ in region III, the specimens exhibited elongations to failure of 560% and 180%, respectively.

Figures 2 a-c show SEM micrographs for the same region in a specimen deformed at a strain rate of 3 x 10^{-4} s⁻¹ to elongations of 16. 45 and 78%. respectively. These photomicrographs show the topological changes occurring in the coarse-grained region of the specimen with the grains typically having dimensions of 50 μ m. The sharp offsets in the marker lines at grain boundaries is indicative of the occurrence of grain boundary sliding. An examination of these micrographs reveals clearly that the coarse grains elongate during superplastic deformation. In addition, a careful inspection of these micrographs reveals the bending of the marker lines within the coarse grains. Both of the above observations are consistent with the occurrence of intraganular dislocation creep in the coarse grains during superplastic deformation. Measurements indicate that the relative elongation of the coarse grains is essentially identical to the relative elongation of the specimen. Figure 3 is a micrograph of a fine-grained region of the specimen deformed to an elongation of 16% at a strain rate of 3 x 10^{-9} s⁻¹. The fine grains typically have dimensions of $-5 \mu m$. There is clear evidence of the occurrence of grain boundary sliding; however, it is important to note that the marker lines remain straight within the grains. Figure 4 shows a fine-grained region of the specimen at an elongation of 78%. A careful inspection of Fig.4 indicates that, while there is extensive grain boundary sliding and grain rotation, there is no evidence for the bending of marker lines within the grains. An examination of several regions in the specimen

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indicated that the fine grains did not elongate along the tensile axis. These micrographs suggest that there is very little intragranular deformation in the fine grains during superplastic deformation. These results are consistent with previous topological studies on fine-grained superplastic alloys. $^{13-16}$

The above observations on topological changes occurring during superplastic deformation indicate that, in a material with a bimodal grain size distribution, the coarse grains may deform largely by <u>intragranular</u> dislocation creep and the fine grains may deform largely by an <u>intergranular</u> creep mechanism.

Figures 5 a and b show the topological changes occurring in a specimen deformed at a strain rate of $1.3 \times 10^{-2} \text{ s}^{-1}$ to elongations of 15 and 42%, respectively. These micrographs show the changes occurring in the coarse grains during deformation in region III and they provide clear evidence for the elongation of the coarse grains along the tensile axis. Careful inspection also shows that the marker lines bend within the coarse grains and that the relative elongation of the coarse grains is similar to the relative elongation of the specimen.

Figures 6a and b illustrate the changes occurring in the fine-grained region of a specimen deformed at a strain rate of $1.3 \times 10^{-2} \text{ s}^{-1}$ to elongations of 15% and 42, respectively. It is apparent that the fine grains marked as A and B in Figs. 6 a and b have elongated during deformation in region III. In addition, the initially straight marker lines are clearly bent within the grains A and B in Fig. 6b. These observations indicate that the fine grains also undergo intragranular deformation in region III. The observations illustrated by Figs. 5 and 6 suggest that, in region III, both the fine grains and the coarse grains undergo intragranular deformation.

The present study provides experimental support for the theoretical model proposed recently by Ghosh and Raj.¹⁰ In this model, it was assumed that the coarse grains and the fine grains may deform by different mechanisms. An analysis of the model indicated that, in region III both the coarse and the fine grains will deform by intragranular dislocation power-law creep whereas in region II, the coarse grains will deform by power-law creep and the fine grains will deform by Coble diffusional creep. The experimental results obtained in region III are in agreement with the analysis by Ghosh and Raj. In region II, the experimental results show that the coarse grains deform by an intragranular creep process. These results are consistent with the analysis developed by Ghosh and Raj¹⁰; however, it is to be noted that the present results do not necessarily indicate that the fine grains deform by a Coble diffusional creep mechanism.

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Finally, it is important to note that both the observations of grain elongation and the bending of marker lines within coarse grains of the Al-Li alloy with a bimodal grain size distribution are contrary to typical observations in uniform fine-grained microduplex superplastic alloys.¹³⁻¹⁶ Thus, for example, Miller <u>et al.</u>¹⁴ examined the changes occurring in the surface grains during superplastic deformation in a fine-grained Pb-Sn eutectic alloy. Their careful surface study up to elongations of 800% indicated that the fine grains do not elongate and that the initially straight marker lines remain straight within the grains during superplastic deformation. Thus, while the Ghosh and Raj⁹ model may provide a reasonable explanation for deformation in some alloys, additional theoretical models may be necessary to rationalize the deformation characteristics of other superplastic alloys.

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4. Research Planned for the Follow-On-Year

The major emphasis for research in the following year will be to develop an understanding of cavitation during superplastic deformation and to examine the effect of hydrostatic pressure on cavitation.

Hydrostatic pressure may lead to decreases in the total level of cavitation by decreasing the level of cavities nucleated, or by decreasing the rate of growth of cavities or both. It is well known that grain boundary sliding leads to stress concentrations at particles, and cavities may nucleate if this stress concentration is not relieved sufficiently rapidly. The time to nucleate a cavity from classical theory (for assumed shape factor) will be compared to the time it takes to relax the stress concentration at grain boundary features (i.e., ledges, particles at boundary) by diffusional processes. Such analysis will give us an indication of the probability of nucleating a cavity under the given experimental condition.

REFERENCES

- 1. E.E. Underwood, J. Metals, (1982) 14, 914-919.
- K.A. Padmanabhan and G.J. Davies, <u>Superplasticity</u>, Springer Verlag, Berlin (1980).
- 3. O.D. Sherby and J. Wadsworth, Mater. Sci. Tech., (1985), 1, 925-936.
- 4. M.Y. Wu and O.D. Sherby, Scripta Metall., (1984) <u>18</u>, 773-776.
- 5. T.G. Nieh, C.A. Henshall and J. Wadsworth, Scripta Metall., (1984) <u>18</u>, 1405-1408.
- 6. C. Carry and A. Mocellin, <u>Superplasticity</u>, B. Baudelet and M. Suery eds., Centre Nationale de la Recherche, Paris, 1985, pp. 16.1-16.19.
- 7. C. Carry and A. Mocellin, J. Amer. Cer. Soc., (1986) <u>69</u>, C125.
- 8. B.P. Kashyap and A.K. Mukherjee, <u>Superplasticity</u>, B. Baudelet and M. Suery, eds., Centre Nationale de la Recherche, Paris, 1985, pp. 4.1-4.31.
- 9. A.K. Ghosh and R. Raj, Acta Metall., (1981) <u>29</u>, 283-292.
- 10. A.K. Ghosh and R. Raj, Acta Metall., (1981) 29 607-616.
- 11. A.K. Ghosh and R. Raj, <u>Superplasticity</u>, B. Baudelet and M. Suery eds., Centre Nationale de la Recherche, Paris, 1985, pp. 11.1-11.19.
- 12. R. C. Gifkins, <u>Superplastic forming of structural alloys</u>, N. E. Paton and C.H. Hamilton eds., TMS-AIME, Warrendale, PA, 1982, pp. 3.
- 13. A.E Geckinli and C.R. Barrett, J. Mater. Sci., (1976) <u>11</u>, 510-521.
- 14. D.A. Miller and T.G. Langdon, <u>Microstructural Science</u>, G. Petzow, R. Paris, E.D. Albrecht and J.L. McCall eds., Elsevier Science, New York, 1981, vol. 9, pp. 249-256.
- P. Shariat, R.B. Vastava and T.G. Langdon, <u>Microstructural Science</u>, W.E. White, J.H. Richardson and J.L. McCall eds., Elsevier Science, New York, 1982, vol.10, pp. 337-345.
- Z.-R. Lin, A.H. Chokshi and T.G. Langdon, <u>Microstructural Science</u>, M.E. Blum, P. M. French, R. M. Middleton and G. F. Vander Voort, eds., ASM, Ohio, 1987, vol. 15, pp. 323-334.



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The variation in flow stress with strain rate for the Al-Li alloy. Fig. 1

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Fig. 3.

Scanning electron micrograph of a fine-grained region in the specimen tested at a strain rate of $3 \times 10^{-4} \text{ s}^{-1}$ to an elongation of 16%.



Fig. 4.

Scanning electron micrograph of a fine-grained region in the specimen tested at a strain rate of 3×10^{-4} s⁻¹ to an elongation of 78%.



a



b

Fig. 5 Scanning electron micrographs of the same regions in the specimen tested at a strain rate of 1.3 x 10^{-2} s⁻¹ to elongations of (a) 15 and (b) 42%, respectively.



SUPERPLASTIC-LIKE BEHAVIOR IN A MECHANICALLY ALLOYED ALUMINUM

1. Introduction

PART B

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Superplastic behavior is characterized by large tensile elongations [1]. Typically, values of about 500 to 1000% are possible although values of over 5000% have been documented [2]. The phenomenon of superplasticity is of both commercial and scientific interest. For commercial applications, elongation of only several hundred percent are necessary; the uniformity of deformation that usually accompanies superplastic flow is of equal importance.

One of the drawbacks of superplastic forming of commercial alloys is the relatively slow strain rates at which optimum superplastic elongations are found. In general, this strain rate is closely related to the principal microstructural feature governing superplastic flow, i.e., grain size. Thus, the 7000 series aluminum alloys are superplastic at relatively slow strain rates of 0.0002/s and have a relatively coarse grain size (about 15 μ m). By comparison, SUPRAL and many Al-Li alloys [2] exhibit optimum superplastic behavior at strain rates faster than the 7000 series alloys, i.e., about 0.003/sec, and this improvement is related to their fine grain size of 2-4 μ m.

It was recently discovered [3] that composites of Al-2124 containing 20 vol% of SiC whiskers behave in a superplastic-like manner (up to 300% elongation) at a relatively high strain rate of 0.3/s. This strain rate is two orders of magnitude faster than that for the best of the aluminum alloys described above. The observation of superplastic like behavior at these unusually high strain rates is nonetheless believed to be consistent with the extremely fine (submicron) grain sizes in these complex composites and

alloys. Furthermore, a similar high strain rate superplasticity phenomenon was recently observed in an ultrafine grained, mechanically alloyed IN9021 alloy [4]. In this latter case, maximum elongations were found at yet faster strain rates up to 1/s. To the authors' knowledge, this type of high strain rate phenomenon has only once been previously observed. Gregory, et al., [5] noted high ductilities (300%) at high strain rates (0.3/s) in a study of the mechanically alloyed, nickel-based superalloys, MA 754 and IN 6000.

In the present paper, new experimental data are presented from tests carried out on IN90211, an alloy that is very similar to IN9021, at strain rates of as high as 340/s, which is over two orders of magnitude higher than previously examined for IN9021 [4].

2. Experimental Procedures

Specimens of IN90211 were produced by Novamet Aluminum. The alloy was manufactured by mechanical alloying and contains oxide and carbide dispersions of approximately 30 nm in diameter that have an interparticle spacing of about 60 nm [6]. The material had been subjected to a rather complex thermomechanical processing procedure to reduce extruded plate of 127 mm thickness to thin sheet 2.5 mm thick. Specimens with a 2.5 mm square cross section and a gage length of 6.7 mm were machined from processed sheets. The tensile axis was selected to be parallel to the rolling direction. Both IN9021 and IN90211 were processed similarly, but differ slightly in composition: by wt.%, IN9021; 4.0 Cu, 1.5 Mg, 1.1 C, 0.8 0, bal. Al, IN90211; 4.4 Cu, 2.0 Mg, 1.1 C, 0.8 0, bal. Al.

Constant true strain rate tensile tests were carried out over the temperature range 425 to 475°C, at strain rates from 0.0001 to 1/s, on a computer-controlled, servo-hydraulic, testing machine. Tensile tests at

strain rates between 2 and 340/s were carried out at constant engineering strain rates (constant crosshead velocity). The testing temperature was normally controlled to \pm 2°C. During testing at high strain rates, however, specimens were observed to increase in temperature by about 5°C near the ends of the gage length following deformation. Calculations reveal that adiabatic heating can lead to temperature increases of up to 17°C, after 500% elongation at 475°C. Local high strains in the center of the specimen, therefore, could result in temperatures that exceed the incipient melting temperature (492°C) in specimens deformed at 475°C.

Selected specimens were examined using optical, scanning and transmission electron microscopy prior to and after deformation. In addition, some microstructural features, e.g., initial porosity (15 to 500 μ m diam.), stringers (of Fe-rich particles, 5-30 μ m diam.), and small (0.3-3 μ m) copperrich particles were identified.

3. Results and Discussion

The elongation-to-failure for IN90211 alloy, as a function of strain rate, is shown in Fig. 1a for several testing temperatures. Over the temperature range from 425 to 475° C, and at the strain rates at which most aerospace aluminum alloys exhibit superplasticity, i.e., 0.0001 to 0.003/s, IN90211 shows modest elongation of 30-40%. This type of elongation is similar to that found in most pure metals and alloys at elevated temperatures and intermediate strain rates. The values of elongation-to-failure for IN90211, however, are observed to increase continuously as the strain rate increases. An apparent maximum in elongation-to-failure is found at a strain rate between 1 to 10/s, depending upon the testing temperature. In general, at a given strain rate, the value of the elongation-to-failure increases as the test temperature increases. A maximum elongation-to-failure of 505% was recorded at a test temperature of 475°C and a strain rate of 2.5/s. The elongation data obtained from IN90211 in this study are slightly higher than those from IN9021 [4]. This may be partially due to the slight difference in chemical compositions between these two alloys, but may also be due to the shorter gage length specimens used in the present study compared with the data of Nieh, et al [4]. In summary, the data of the present study on IN90211 are considered to be both qualitatively and quantitatively reasonably consistent with those obtained from IN9021.

The peak true stress is plotted as a function of strain rate in Fig. 1b. Each of these regions has a particular value of stress dependence upon strain rate. For superplastic studies it is convenient to use the simple equation σ = $k\epsilon^{in}$ where σ is the true flow stress at a true strain rate, ϵ , k is a constant, and m is the strain rate sensitivity exponent. For ideally superplastic materials, m = 1 (Newtonian viscous fluids), whereas for most superplastic metals, m = 0.5. Conventional metals usually have values of m =In the low stress or low strain rate regime, for IN90211 m < 0.1, 0.2. whereas at high stresses or strain rates (above 0.1/s) the m is about 0.3 In addition, preliminary data indicate that there may exist a third regime with m < 0.3 at extremely fast strain rates (above 50/s). The apparent transition of flow behavior from m < 0.1 to m = 0.3 at high strain rates is also consistent with previously reported high strain rate data [3-5]. For specimens showing high elongations, the stress-strain curves typically exhibit an extensive stress plateau region, indicating a uniform elongation prior to necking. It is interesting to note that the elongation values of

about 300-500% for m = 0.3 are consistent with prediction based on correlation of m with elongations-to-failure by Woodford [7].

It is generally believed that low strain rate sensitivity exponents (or high stress exponents, n, where n=1/m) are normally observed in ODS materials during creep in low stress regimes and are a consequence of the existence of a threshold stress [8-10]. A special effort was made to measure the presence of a threshold stress in the present study by conducting a stress relaxation After a long period (approximately 8×10^4 s) of experiment at 450°C. relaxation an apparent steady state was reached. Despite the fact the relaxation curve was subjected to a small ambient temperature fluctuation (about 3°C) during the experiment (which resulted in a slight variation in the flow stress measurement) the final result suggests an apparent threshold stress of 9 MPa. This value is not consistent with calculated values based on the Orowan bowing theory [11]. The apparent threshold stress can also be estimated from Fib. 1b using approaches suggested by Mohamad [8] and Nardone et al [9]. Following Mohamad [8], a threshold stress was derived from a plot of the cube root of strain rate versus stress, as shown in Fig. 2, using only data from the high strain rate range of Fig. 1b. The extrapolated values for threshold stress using this method are 5 and 16 MPa for temperatures of 475 and 425°C, respectively. These values are consistent with the 9 MPa value obtained from the stress relaxation measurement at 450°C. The introduction of this threshold stress (to replace the flow stress with an effective stress) in the normalized plot of Fig. 3 does not appear to significantly affect the general behavior of the strain rate-stress curve, contrary to the results of others [8,9]. This replacement does, however, result in a better defined strain rate-stress curve, i.e., a change in stress exponents of n =

20 at low strain rates to n = 3.0 at high strain rates. By using these threshold stresses, an activation energy of approximately 350 kJ/mol was obtained from the low strain rate data which is about three times the value for lattice diffusion in aluminum. This result is consistent with the observation that the apparent activation energy for creep of a nickel-base ODS alloy is about 3 times that of the activation energy for bulk diffusion of the matrix material [12].

A transmission electron micrograph of the microstructure of an untested sample is shown in Fig. 4. In addition to fine oxides and carbides, S phase (CuMgAl₂) precipitates are also sometimes found in the microstructure, especially along grain boundaries. The grain size was determined to be approximately 0.5 μ m. The microstructure of samples after deformation appeared to be similar to the undeformed one. This illustrates the high degree of thermal stability of the microstructure. Although most grain boundaries are observed to be high angled, grains with low-angle boundaries are observed. These grain clusters may result from coalescence of the low-angle boundary regions.

A typical fracture surface from a superplastically deformed IN90211 specimen is shown in Fig. 5a. The fracture surface exhibits a granular appearance indicative of an intergranular fracture mode. Also, cavities of several μ m in size and several μ m apart are observed. It is interesting to note that the sizes of the granular features are almost identical to the grain size of the untested material as observed by TEM. Failure in the specimen was caused by grain boundary separation. In the case of fracture samples from low strain rate tests (nonsuperplastic region), the fracture

appearance is different. Macroscopically, the samples exhibit local necking and, microscopically, the fracture surface exhibits overall ductile cup-andcone type features, with cups of about 10-20 μ m in size, as shown in Fig. 5b. Interestingly, this fracture surface appearance is guite different from the observation in IN9021 by Shaw [13], in which a fibrous fracture surface was observed. These differences are difficult to explain since the testing temperature used by Shaw was 400°C which is close to the temperature selected in the present study. Although Shaw emphasized the importance of strain rate, the strain rate used by Shaw is uncertain. Special attempts were made to study grain boundary sliding during superplastic deformation of IN90211. Markers were placed on highly polished specimens surfaces and the deformed specimen was examined using SEM. Evidence of grain boundary sliding and rotation is apparent as shown in Fig. 6. At an estimated local strain of 500% or greater, porosity between grains was observed. Some grains as well as grain clusters are visible in Fig. 6. The grain clusters are believed to be related to the low-angle grain boundaries observed in the TEM microstructure, discussed earlier.

The exact deformation mechanisms that operate at high strain rates in materials like MA alloys, such as ODS materials, are still uncertain. Shaw [13] has suggested that the phenomenon may not be related to a fine grain size, but may be a result of dynamic recrystalization from the high strain rate of testing. His theory however, cannot explain the observation of grain boundary sliding and the retention of grain size distribution before and after superplastic deformation. Gregory, et al., [5] have argued that a combination of slip with Coble creep (in which the Coble creep exhibits a threshold stress) can explain the phenomenon. From a microstructural point

of view, it is expected that the maximum strain rate at which superplasticity is observed will increase as the grain size decreases. This is because of the inverse grain size dependence of the strain rate for superplastic flow [4]. However, a fine grain size is a necessary but not sufficient condition to produce superplasticity [1]. For example, in our study another mechanically alloyed aluminum alloy, IN9052 has been examined. This alloy is also fine-grained but has exhibited a relatively poor ductility up to strain rates of 1/s. Although the two alloys appear to have similar grain sizes, other microstructural features that may explain the difference in properties between the two alloys IN90211 and IN9052 have not yet been determined.

4. Conclusions

Tensile elongation in excess of 500% were obtained with IN90211 mechanically alloyed aluminum at strain rates between 1 and 10/s at 475°C. This superplastic-like behavior at high strain rate appears to be associated with the fine grained (0.5 μ m) microstructure in the alloy. Grain boundary sliding was clearly observed. A stress exponent of 3 was obtained in the superplastic-like regime. Current theories of superplasticity do not fully account for the observed stress exponent change at high strain rate.

5. Research Planned for the Follow-On-Year

Based on the above mentioned results, the following experiments with the mechanical alloy are proposed in the coming year:

A. Load relaxation experiments will be conducted at the four temperatures (475, 462, 450, 425°C) starting at a rate of 10/sec. These same specimens can then be used to obtain another value of elongation in a subsequent test, which will indicate how time at temperature affects elongation behavior. The

shoulder regions of the specimens will be used to investigate whether strainenhanced grain growth occurs during the test.

B. Creep experiments will be done at loads below the lowest observed load relaxation value, to determine whether there is indeed a "threshold stress" operative.

C. Microscopy (TEM, SEM and optical) will be used to investigate the role of cavitation and dislocation motion in deformation process.

D. Attempts will be made to use x-ray line-width broadening technique to ascertain if there are large differences in the dislocation density in the deformed and undeformed samples. Micro-beam x-ray spectroscopy will be used to identify the chemistry of the inert dispersions.

E. Results of both mechanical and transmission electron microscopy will be combined in order to ascertain the true origin of the threshold stress in the microstructure.

<u>References</u>

- O.D. Sherby and J. Wadsworth, <u>Deformation</u>, <u>Processing</u>, <u>and Structure</u>, Krauss, G., (ed.), ASM, Metals, Park, OH, (1983), 355.
- J. Wadsworth, T.G. Nieh, and A.K. Mukherjee, <u>Aluminum Alloys: Their</u> <u>Physical and Mechanical Properties</u>, Starke, E.A., and Sanders, T.H., (eds.) Chameleon Press Ltd., London, United Kingdom, (1986), 1239.
- 3. T.G. Nieh, C.A. Henshall and J. Wadsworth, Scripta Metall., <u>18</u>, (1984), 1405.
- T.G. Nieh, P.S. Gilman and J. Wadsworth, Scripta Metall., <u>19</u>, (1985), 1375.
- J.K. Gregory, J.C. Gibeling and W.D. Nix, Metall. Trans., <u>16A</u>, (1985), 777.
- 6. P.S. Gilman and W.D. Nix, Metall. Trans., <u>12A</u>, (1981), 813.
- 7. D.A. Woodford, Trans. ASM, <u>62</u>, (1969), 291.

8. F.A. Mohamed, J. Mat. Sci., <u>18</u>, (1983), 582.

H-S-S-H all horsesses browned

- 9. V.C. Nardone, D.E. Matejczyk, and J.K. Tien, Acta Metall., <u>32</u>, (1984), 1509.
- 10. A.H. Clauer and N. Hansen, Acta Metall., <u>32</u>, (1984), 269.
- 11. E. Orowan, <u>Internal Stresses in Metals and Alloys</u>, Inst. Metals, London, (1948), 451.
- 12. J.J. Stephens and W.D. Nix, Metall. Trans. <u>16A</u>, (1985), 1307.
- 13. W.J.D. Shaw, Mater. Lett., <u>4</u>, (1985), 1.



Fig. 1 Elongations-to-failure (a) and peak true stresses (b) as a function of strain rate. The maximum elongation-to-failure occurs at strain rates between 1 and 10s⁻¹.





Fig. 2 (Peak True stress) versus (true strain rate) ' plot. Extrapolated values for threshold stresses are 5 and 16 MPa for 475 and 425°C, respectively. Fig. 3 Normalized peak true stress, as a function of normalized strain rate, assuming lattice self diffusion for alumium, and a threshold stress (as discussed in the text).



100nm

Fig. 4 Transmission electron micrograph of the microstructure of IN90211. The S phase precipitates (arrowed) are often observed at grain boundaries.



5 µm

Fig. 6 Deformation of an initially polished surface (1/s, 425°C). Note evidence of grain boundary rotation and sliding.



1 0 µm



1 0 µm

b

Fig. 5 Fracture surfaces of IN90211 at strain rates of (a) 77/s (superplastic) (b) 0.0001/s (nonsuperplastic), 475°C.

PART C LOW STRESS AND SUPERPLASTIC CREEP BEHAVIOR OF Zn-22% A1 1. EUTECTOID ALLOY, A. Arieli, A. K. S. Yu, and A. K. Mukherjee. Metallurgical Transactions, Vol. 11A, pp. 181-191, 1980. 2. FACTORS AFFECTING THE MAXIMUM ATTAINABLE DUCTILITY IN A SUPERPLASTIC TITANIUM ALLOY, A. Arieli and A. Mukherjee, Materials Science and Engineering, Vol. 43, pp. 47-54, 1980. HIGH-TEMPERATURE DIFFUSION-CONTROLLED CREEP BEHAVIOR OF THE Zn-3. 22% Al EUTECTOID TESTED IN TORSION, A. Arieli and A. K. Mukherjee, Acta Metallurgica, Vol. 10, ppl 1571-1581, 1980. AN EVALUATION OF THE EFFECTS OF CONCUREENT GRAIN GROWTH DURING 4. SUPERPLASTIC FLOW OF THE Ti-6A1-4V ALLOY, A. Arieli, B. J. Maclean and A. K. Mukherjee, Proc. of the 4th Intnl. Conf. on Titanium, Kyoto, Japan, Ed. H. Kimura and O. Izumi, pp. 1047-1056, 1980. TWO-AND THREE-DIMENSIONAL DEFORMATION MECHANISM MAPS FOR HIGH 5. TEMPERATURE CREEP OF Zn-22% Al EUTECTOID ALLOY, A. Arieli and A. K. Mukherjee, Materials Science and Engineering, Vol. 47, pp. 113-120, 1980. REPLY TO "A CRITICAL EVALUATION OF THE CONCEPT OF A UNIVERSAL 6. PARAMETER TO UNIQUELY SPECIFY HIGH TEMPERATURE CREEP MECHANISMS," A. Arieli and A. K. Mukherjee, Scripta Metallurgica, Vol. 15, Sept. 81, p. 1053, 1980. THE EFFECT OF STRAIN AND CONCURRENT GRAIN GROWTH ON THE 7. SUPERPLASTIC BEHAVIOR OF THE Ti-6A1-4V ALLOY, A. Arieli, B. J. McLean and A. K. Mukherje, Res Mechanica, Vol. 6, pp. 131-159, 1983. ELEVATED TEMPERATURE CAVITATION IN CREEP AND SUPERPLASTICITY OF 8. Ti-6Al-4V ALLOY, G. Gurewitz, N. Ridley and A. K. Mukherjee, Proceedings of the International Conference on Fracture Mechanics, Nov. 1983, Pub. by the Chinese Soc. of Theoretical

MECHANICAL AND MICROSTRUCTURAL ASPECTS FOR OPTIMIZING THE 1984 9. SUPERPLASTIC DEFORMATION OF Ti-6A1-4V ALLOY, B. Hidalgo-Prada, G. Gurewitz and A. K. Mukherjee, Proceedings of Intn' Amer. <u>Conf. on Mat. Tech.</u>, San Juan, Puerto Rico, Ed, D. Black, p. 18.1, 1984

and Applied Mech., p. 898, Science Press, Beijing, China, 1983.

1. CUMMULATIVE LIST OF PUBLICATIONS RESULTING FROM AFOSR SUPPORT

1980

- A METALLOGRAPHIC STUDY OF CAVITATION IN Ti-6A1-4V ALLOY, G. Gurewitz and A. K. Mukherjee, <u>Advances in Fracture Research</u>, Ed., S. R. Valluri, et al., Pergamon Press, Oxford, p. 2319-2324, 1984.
- CREEP AND DISLOCATION SUBSTRUCTURE, L. Bendersky, A. Rosen and A. K. Mukherjee, Invited Review, <u>International Metals Review</u>, Vol. 30, p. 1, 1985.
 - THE STRAIN RATE SENSITIVITY VALUES IN SUPERPLASTIC DEFORMATION,
 G. Gurewitz and A. K. Mukherjee, <u>Materials Science and</u> <u>Engineering</u>, Vol. 70, p. 191-196, 1985.
 - ON THE NEW MODELS OF SUPERPLASTIC DEFORMATION, B. P. Kashyap and A. K. Mukherjee, <u>Proceeding of International Conference on</u> <u>Superplasticity</u>, Grenoble, France, pp. 4.1-4.31, Sept. 1985.
 - MICROSTRUCTURAL EVOLUTION DURING SUPERPLASTIC DEFORMATION IN A Ni-MODIFIED Ti-6A1-4V ALLOY, B. Hidalgo-Prada and A. K. Mukherjee, <u>Scripta Metallurgica</u>, Vol. 19, pp. 1235-1239, 1985.
 - SUPERPLASTICITY-CORRELATION BETWEEN STRUCTURE AND PROPERTIES, M. Suery and A. K. Mukherjee, <u>Creep Behavior of Crystalline Solids</u>, ed. B. Wilshire, Pineridge Series on Progress in Creep and Fracture, Pineridge Press, Swansea G>B>, pp. 137-200, 1985
 - 16. CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE IN A NI-MODIFIED SUPERPLASTIC Ti-6A1-4V ALLOY, B. Hidalgo-Prada and A.K. Mukherjee, <u>Proc. of Intnl. Conf. of Strength of Metals and Alloys-7</u>, Montreal Canada, ed. H.J. McQueen <u>et al.</u>, Vol. 2, pp. 835-840, 1985.
- 1986 17. CAVITY NUCLEATION IN 7475 AL ALLOY, M. K. Rao and A. K. Mukherjee, <u>Scripta Met.</u>, Vol. 20, pp. 411-414, 1986.

1985

•

- REVIEW: CAVITATION BEHAVIOR DURING HIGH TEMPERATURE DEFORMATION OF MICROGRAINED SUPERPLASTIC MATERIALS,
 B. P. Kashyap and A. K. Mukherjee, Invited review, <u>Res</u> <u>Mechanica</u>, Vol. 17, pp. 293-355, 1986.
- SUPERPLASTIC DEFORMATION BEHAVIOR IN INGOT AND POWDER METALLURGICALLY PROCESSED Al-Li BASED ALLOYS, M.C. Pandey, J. Wadsworth and A.K. Mukherjee, <u>Materials Science and Engineering</u>, Vol. 80, No. 2, pp. 169-179, 1986.
- 20. ON THE TRANSITION FROM SUPERPLASTIC TO NON-SUPERPLASTIC DEFORMATION AT HIGH STRAIN RATES, Atul Chokshi and Amiya Mukherjee, <u>Scripta Met</u>, Vol. 20, pp. 1771-1774, 1986.
- 21. ON THE TRANSITION FROM SUPERPLASTIC TO NON-SUPERPLASTIC DEFORMATION AT HIGH STRAIN RATES, Atul Chokshi, Amiya Mukherjee, <u>Scripta Met</u>, 1986.

- 22. HIGH STRAIN RATE SUPERPLASTICITY IN MECHANICALLY ALLOYED ALUMINUM, T. R. Bieler, T. G. Nieh, J. Wadsworth and A. K. Mukherjee, <u>Proceedings of IX Inter-American Conference on Materials</u> <u>Technology</u>, Santiago, Chile, p. 213-217, 1987.
- 23. MICROSTRUCTURAL EVIDENCE FOR DYNAMIC RECRYSTALLIZATION DURING SUPERPLASTIC DEFORMATION, Atul H. Chokshi, Jeffrey Wadsworth and Amiya K. Mukherjee, <u>Scripta Met.</u>, Vol. 21, p 1347-1351, 1987.

IN PRESS

- 1. ON THE INTER-DEPENDENCE OF GRAIN BOUNDARY PHENOMENON AND SUPERPLASTIC DEFORMATION, B.P. Kashyap and A.K. Mukherjee, <u>Proceedings of Seiken</u> <u>Symposium</u>, Japan 1985.
- 2. EFFECT OF HYDROSTATIC PRESSURE ON HIGH TEMPERATURE FAILURE IN SUPERPLASTIC ALUMINUM ALLOYS, A. H. Chokshi, J. E. Franklin and A.K. Mukherjee, <u>Fifth International Conference on Mechanical</u> <u>Behavior of Materials</u>, Beijing, China.
- 2. <u>List of Dissertations</u>
 - 1. "Superplastic Forming Under Biaxial Stress State" by H.S. Lee.
 - 2. Ph.D. Dissertation: (In preparation): Enhanced Plasticity in Mechanically Alloyed Aluminum at High Strain Rates by T. R. Bieler.
- 3. List of Personnel Involved in the Research

1.	Prof. Amiya K. Mukherjee:	Principal Investigator
2.	Dr. Atul Chokshi:	Post Doctoral Fellow (emphasis on Al-Li alloy system and elevated temperature plasticity.)
3.	Mr. Thomas Bieler:	Ph.D. Candidate and Research Assistant, (emphasis on Al-SiC and mechanically alloyed aluminum system).
4.	Ms. Shara Hinote-Williams:	Report and Manuscript preparation.

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Dr. J. Wadsworth, Dr. T.G. Nieh and Ms. C. Henshall of Lockheed, Palo Alto Laboratory. Cooperation on Al-Li; 2124 Al-SiC and mechanically alloyed aluminum systems.

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