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SUPERPLASTICITY - A FUNDAMENTAL INVESTIGATION ON DEFORMATION MECHANISM AND CAVITATION PHENOMENA

by



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ABSTRACT

Aluminum-Lithium alloys, mechanically alloyed and also SiC_{whisker} reinforced aluminum alloy--all have considerable potential for structural application in aerospace industry. These three alloy systems are being investigated in this research program. The Al-Li alloy was tested in tension at elevated temperature to establish the optimum condition for maximum elongation. The double logarithmic plot of stress vs strain rate exhibited the sigmoidal shape that is characteristic of superplasticity. The grain boundary sliding associated with superplasticity and the localization of plastic flow at strain rates away from optimum condition were investigated. The ongoing work is directed towards a better understanding of the cavitation phenomenon during superplastic deformation and to examine the effect of hydrostatic gas pressure on cavitation.

The mechanical alloy IN90211 was tested in tension for enhanced plasticity at 425, 450 and 475°C at an engineering strain rate of 50,000% min. Both the neck stability and elongation values increased with increasing temperature at this strain rate. The mechanical data were analyzed using the rate equation for creep deformation using estimated values of temperature dependent threshold stress. This concept of threshold stress will be investigated more rigorously using constant strain rate tests, load relaxation tested and also some low-stress creep experiments during the next year.

Part A

SUPERPLASTIC DEFORMATION AND FRACTURE IN A1-L1 ALLOYS

1. Introduction

Lithium additions to Al-based alloys lead to significant increases in the stiffness and to decreases in the density. Therefore, Al-Li alloys have considerable potential for applications in the aerospace industry and for other structural applications where weight considerations are important. The use of superplastic forming to manufacture components from Al-Li alloys can lead to additional benefits in cost savings and weight savings. Consequently, there is a considerable interest to develop superplasticity in Al-Li alloys.

Suitable thermo-mechanical processing can impart fine grain sizes to Al-Li alloys and this enhances the prospects for superplasticity in such alloys [1,2]. Recent experimental studies have shown that such alloys may exhibit very large elongations to failure under a limited range of temperatures and strain rates. However, extensive cavitation occurs during the superplastic deformation of Al-Li alloys [3]. Experimental studies have shown that even small levels of cavitation have a deleterious effect on the post-forming properties of superplastic alloys [4]. Therefore, there is a clear need to understand the deformation and fracture of Al-Li alloys, and to develop procedures to overcome the problem of concurrent cavitation. Previous studies in our laboratory have shown that cavitation in Al-Li alloys can be controlled by the superimposition of hydrostatic gas pressure during superplastic deformation [5]. However, the details of the processes involved in eliminating cavitation are not clear.

The overall goal of this study is to develop a fundamental understanding of deformation and fracture in superplastic Al-Li alloys. The role of hydrostatic gas pressure on cavitation is of particular interest. However, in order to understand the effect of hydrostatic pressure on cavitation, it is first necessary to characterize the deformation and fracture behavior at atmospheric pressure. This report essentially summarizes the progress made to date in characterizing the deformation of a superplastic Al-Li alloy at atmospheric pressure.

2. Experimental Material and Procedure

The Al-Li alloy chosen for this study had the following composition: Al-2.62% Cu-2.42% Li-0.20% Zr-0.07% Fe, where the constituents are given in weight percent. The material was provided by Dr. J. Wadsworth of Lockheed Palo Alto Research Lab, and it was supplied by Reynolds Metals Co. Tensile specimens with a gauge length of 6.3 mm were machined with their tensile axis parallel to the rolling direction.

Tensile experiments were performed in an Instron machine modified to operate at constant strain rates between 10^{-5} and 10^{-2} s⁻¹. The tensile specimens were annealed in air at 773 K for a period of 11 hours to allow for recrystallization and the stabilization of the initial microstructure. Individual specimens were tested to failure at 723 K to obtain the variation in flow stress with elongation.

After characterizing the mechanical properties, an experiment was performed to examine the possibility of grain boundary sliding during superplastic deformation. The surface of a tensile specimen was polished to a smooth finish and a series of marker lines was inscribed on the surface, parallel to the tensile axis, by rubbing the surface once with a lens tissue containing $3\mu m$ diamond paste immersed in alcohol. The specimen was tested under the optimum strain rate of $3 \times 10^{-4} \text{ s}^{-1}$ at 723 K to an elongation of 15%. The surface of the specimen was subsequently examined in a Scanning Electron Microscope (SEM) to characterize the movement of grains during superplastic deformation.

The profiles of the fractured specimens were characterized quantitatively by measuring the variation in the cross-sectional areas with distance from the fracture tip.

3. Experimental Results and Discussion

3.1. Mechanical properties

Inspection of the true stress-elongation curves revealed that the Al-Li alloy does not exhibit any significant strain hardening or strain softening except during the early stages of deformation and near failure. The alloy typically exhibits steady-state stresses beyond elongations of 20%. These results suggest that the microstructure does not change significantly during superplastic deformation.

Figure 1 is a logarithmic plot of the steady-state stresses against the corresponding strain rates. The curve has a sigmoidal shape which is typical of superplastic alloys. The σ - \dot{c} curve is divisible into three distinct regions, each of which can be represented in the following form:

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

where m is the strain rate sensitivity and B is a constant incorporating the effect of grain size and temperature on the flow stress. From the definition of the strain rate sensitivity, it follows that m can be determined from the slope of the curve in Fig. 1. Under optimum conditions, 10^{-4} s⁻¹ < $\dot{\epsilon}$ < 10^{-3} s⁻¹, the Al-Li alloy exhibits an m value of 0.45. At both lower and higher strain rates, m tends to decrease to values less than ~0.3. This value of m compares favorably with the value of m in other superplastic Al-Li alloys [2].

Figure 2 is a plot of the variation in the elongation to failure with strain rate. The maximum elongation to failure of ~560% was obtained at a strain rate of 3×10^{-4} s⁻¹. A comparison of Figs. 1 and 2 reveals that the variation in the elongation to failure closely follows the variation in the strain rate sensitivity with the strain rate. This can be rationalized in terms of the influence of strain rate sensitivity on flow localization. Theoretical analyses predict that a high value of m leads to large elongations to failure by retarding flow localization [6].

3.2. Grain boundary effects during superplastic deformation

Grain boundaries are recognized to play two important roles in superplastic deformation [7]. On the one hand, grain boundary sliding

facilitates the large elongations occurring during superplastic flow, while on the other hand, they promote cavitation which may cause premature failure. Grain boundary sliding has been observed during the superplastic deformation of several microduplex alloys [7,8]. However, very little information is available on the occurence of GBS during the superplastic deformation of commercially important quasi-single phase materials such as Al-Li alloys.

The offsets at grain boundaries in the initially straight marker lines, as shown in Fig. 3, are clear evidence for the occurrence of GBS during superplastic deformation in Al-Li alloys. In addition to GBS, grain rotation also occurs during superplasticity, as indicated by grain A in Fig. 4.

The micrographs shown in Figs. 3 and 4 suggest that the Al-Li alloy deforms superplastically in a manner similar to other superplastic alloys. The observation of GBS is important because the occurrence of GBS can lead to cavity nucleation by developing stress concentrations at grain boundary particles.

3.3. Flow localization during superplastic flow

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Figure 5 shows the profiles of Al-Li specimens pulled to failure at strain rates ranging from 10^{-5} to 10^{-2} s⁻¹. It is clear from an inspection of Fig. 5 that, under the optimum conditions illustrated by specimen D, the deformation is very uniform and there is no evidence of flow localization. At strain rates away form the optimum conditions, as illustrated by specimens B and F, there is significant flow localization and there is clear evidence of necking near the fracture tip. These

results are consistent with the mechanical properties shown in Fig. 1 such that a high value of m retards flow localization.

Quantitative information on flow localization was obtained by measuring the variation in the cross-sectional area with distance from the fracture. The experimental data are summarized in Fig. 6 as a plot of the percentage reduction in area with the fractional length of the fractured specimens. The vertical arrows indicate the positions of the fracture tips. At a strain rate of $1.3 \times 10^{-2} \text{ s}^{-1}$, the specimen has a sharp neck as seen from the shape of the data in Fig. 6. Under optimum conditions, the specimen exhibited very little flow localization and the deformation was uniform over a large region of the gauge length. These experimental results are consistent with the observed variation in m with strain rate.

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.

Specimens that were pulled to failure at different strair rates will be examined microstructurally for cavitation. The fracture tips will be characterized using SEM.

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 the 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. Therefore, measurements of grain boundary sliding under atmospheric pressure and under superimposed hydrostatic pressure provide a convenient means of determining indirectly the effect of hydrostatic pressure on cavity nucleation.

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Fig. 3. Scanning electron micrograph illustrating the occurrence of grain boundary sliding. The tensile axis is horizontal.



Fig. 4. Scanning electron micrograph illustrating the occurrence of grain rotation at the grain marked A. The tensile axis is horizontal.

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Fig. 5. Profiles of the specimens pulled to failure at different strain rates.

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The variation in the cross-sectional area with the normalized gauge length, where ΔA is the change in cross-sectional area and A_0 is the original gauge length. Fig. 6.

PART B

ENHANCED PLASTICITY IN MECHANICALLY ALLOYED AND WHISKER-REINFORCED ALUMINUM ALLOY SYSTEMS

The objective of this work is to understand the micromechanical mechanisms that allow extended ductility of two aluminum alloys at unusually high rates compared to superplastic deformation. If these mechanisms can be understood and controlled, then formation of aluminum parts using superplastic methods at rates similar to forging may be accomplished with confidence.

1. Past Work on Aluminum 2124/SiC_ and IN90211 Mechanical Alloy

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Silicon Carbide whiskers (SiC_w) have been added to aluminum alloys to obtain increased stiffness without a weight penalty. Some of these metals have been shown to behave in a superplastic fashion. In particular, the work done at Lockheed in recent years has indicated that $2124/SiC_w$ will undergo extended ductility at higher strain rates than are commonly used for superplastic alloys. This can be partly rationalized by the small grain size, but the role the whiskers play in the deformation is not yet clear. Similarly, developments at Lockheed on mechanical alloys have also shown extended ductility, at even higher rates than the 2124/Si alloy [1]. These results are now being investigated in detail.

Similarly, Gregory and Nix [2] have investigated the superplastic behavior of a mechanically alloyed nickel based alloy called MA6000. The rates of deformation have also been unusually high, and they show that a diffusion based mechanism (rather than dislocation slip) can account for the high ductility observed. The diffusion mechanism is favorable due to the effect the mechanically alloyed particles have on pinning grain boundaries. This pinning effect produces very fine grain size in the microstructure which is stable as a function of strain and temperature. This is one of the major benefits of mechanical alloying.

2. Experiments of the Past Year

During 1986, preliminary experiments on some roughly machined IN90211 mechanically alloyed specimens were performed to determine whether the trend of increasing elongation continued beyond Lockheed's data with increasing strain rate. These experiments resulted in higher elongations than any recorded at Lockheed, so clearly it is prudent to investigate this more carefully. Thirty carefully machined specimens are in hand for future testing.

In addition, two $2124/SiC_{W}$ specimens were tested at higher strain rates. These resulted in less elongation than at lower strain rates. The reasons for this difference are not yet known and are being investigated at present.

A. Results

The data from Lockheed were obtained with a screw driven Instron machine, which could deform at a maximum of 1.3/sec (8000 %/min.) on 1/4 inch gauge length specimens. The recent data were obtained on an MTS servohydraulic machine operated at constant stroke rate, that could deform at rates up to 8.3/sec (50,000 %/min.). The data recently obtained are plotted upon the data from Lockheed in Figures 1 and 2 to illustrate the trends. Specimen gauge length was shorter in the case of the mechanically alloyed specimens, which may account for some of the increase in elongation. Elongation in the mechanically alloyed specimens was not uniform as shown in Figure 3, where the reduction in area is plotted with final gauge length. This non-uniformity may be enhanced by a temperature gradient along the gauge length, resulting from insufficient time for temperature equilibration. Measurements indicate that temperatures differed from center to end by as much as 15°C. In the current configuration, it is possible to reduce the differential from top to bottom to within 4°C by allowing a longer time to heat up. At lower temperatures, there is greater necking instability. The three data resulting from the highest strain rate (50,000%/min., 8.3/sec) show increasing elongation and neck stability with increasing temperature.

B. Microstructure

The fracture surfaces of the three data at high strain rate are illustrated in Figure 4. At the lowest temperature (425°C), the fracture mode was ductile failure, as the cup and cone geometry is evident. At 450°C, the fracture surface is intragranular, indicating a different

mechanism. Compared to the specimen at 475°C, the 450°C fracture surface is a bit softer in appearance. This softness is not due to surface diffusion following fracture, since the higher temperature fracture surface was sharper. In all cases, the specimen was exposed to ambient air within a minute of the fracture. The fracture surface of the specimen at 450°C at lower strain rate of 10,000%/min (1.67/sec) also appeared as soft as the one at higher rate, and also more regular in topography, as well. Typical cavity size of the intergranular fracture surface is greater than 3μ m. The intergranular surface feature suggests a grain size between 0.4 and 1μ m.

The fracture surface of the $2124/SiC_{W}$ had very similar features to those described above. The fibers are about $0.5\mu m$ in diameter, and were protruding out of the fracture surface without much contact with the aluminum particles. The grain size appears to be between 1 and $2\mu m$ in Figure 5.

C. Analysis of data

The stress and strain rate data have been analyzed to obtain an estimate of the apparent activation energy of the micromechanism producing the extended ductility observed in the mechanical alloy. From the sparse data in Figure 1, values of n = 12.5 and 3.8, for the intermediate and high strain rate region respectively, were used with the usual formulation of the creep equation.

$\dot{\epsilon} = A\sigma^{n} exp(-Q/RT),$

where Q is the activation energy describing the process, RT has its usual meaning, σ is stress, n is the stress exponent, and A is a structure parameter containing grain size. From the strain rate at constant stress

and inverse temperature, the activation energy can be determined from the slopes in Figure 6. Given a value for n, activation energies can be obtained from the values of stress at constant strain rate, indicated by the boxes in Figure 6. This energy is considerably larger than the lattice and grain boundary diffusion values which are 142 and 84 kJ/mol, respectively.

The work of Gregory and Nix indicated a threshold stress was operative, below which deformation was described as a slip mechanism, and above which Coble creep could adequately describe the behavior. The lack of agreement in the activation energies above the diffusion based deformation mechanisms suggested that a similar approach as Gregory and Nix may explain the behavior of the mechanical alloy. To evaluate this, a temperature dependent threshold stress σ_0 , was chosen from an extrapolation of the lower m region of Figure 1. Using these values in the following equation resulted a reduction of the activation energy.

$\dot{\epsilon} = A(\sigma - \sigma_0)^n \exp(Q/RT)$

This does not adequately explain the results, so other considerations involving microstructural aspects or the energetics of the deformation process are being explored.

3. Discussion

As this research effort has just started, there are a number of unanswered questions, and paths that ought to be explored. Some of these are discussed below, and based upon this, experiments are proposed.

It is curious that the mechanical alloyed material appears to have some porosity before deformation, as shown in Figure 7. It is not clear whether the observed porosity in deformed specimens is a remnant of the

initial state, or whether it evolves during deformation. There is as yet insufficient data to answer this question, much less its implications.

The tests done on the mechanical alloy and the $2124/SiC_{W}$ to date have been at constant crosshead rate. In determining the optimum strain rate for maximum elongation, true strain rate test condition would not have the varying strain rate that results in engineering, i.e., constant crosshead rate tests. For example, at 400% strain the true strain rate is 40% of the initial rate. Analysis of the data using kinetic theory is easier when the strain rate as well as structure are held constant as much as possible.

Experiments thus far have indicated that pencil marks on the surface of specimen gauge survive the effect of strain and temperature, allowing an easy way to observe the uniformity of elongation. In future experiments, specimen preparation will include these marks on one side, and polished on the other, which may allow effects such as grain boundary sliding, to be observed.

A load relaxation experiment conceivably allows one to obtain the entire stress vs strain rate curve for a given temperature in one experiment. However, this becomes technically more difficult as the strain rate decreases, such that it would be prudent to do a few creep experiments at loads below the minimum obtained with stress relaxation, in order to determine if the postulated threshold stress is a reasonable way to describe the operating mechanism of the mechanical alloy.

There is a curious correlation in the mechanical alloy datum at 475°C. Adiabatic heating increases the temperature as strain is accumulated, at the rates used. A conservative estimate indicates that the temperature could increase by 10°C for every 100% of true strain.

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The elongation behavior at 500°C is comparitively poor, presumably due to being above the incipient melt of 492°C. Did the specimen at 475°C fracture due to the specimen reaching the incipient melt temperature, since it fractured at 1.7 true strain, which corresponds to conservatively reaching 492°C?

Moreover, why does the $2124/SiC_{W}$ have the best elongations above incipient melt temperature of 502°C? These issues need to be answered yet in a self-consistent manner.

4. Proposed Work for the Follow-on Year

Given the above motivation, the following experiments with the mechanical alloy are proposed in the coming year:

1. A series of experiments, at constant true strain rate, will expand the available data for analytical work. Four temperatures will be used, 475, 462, 450, and 425°C, and experiments will be conducted at one per decade between 100/sec and .001/sec strain rates at each temperature.

2. Load relaxation experiments will be conducted at the above four temperatures 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 strain-enhanced grain growth occurs during the test.

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

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

5. Pending the above results, elongation below failure will be conducted in order to aid in the determination of the micromechanism of plastic flow.

Investigation of 2124/SiC will continue in a similar fashion at a lower level of effort.

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Figure 3 Reduction of area for three specimens tested at three temperatures at 8.3/sec strain rate indicate that the necking stability increases with test temperature.



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diameter are visible, and are not intimately bonded to the metal. SiC whiskers approximately 5 um long and 0.5 um in Figure 5



Figure 6 The effect of a threshold stress (x's on graph) is not evident in the apparent activation energy at higher rates, but it does reduce the energy at lower rates, where the threshold stress is a larger proportion of the total stress. These apparent values are larger than the aluminum self diffusion activation energy (142 kJ/mol).



Figure 7 A polished, undeformed specimen has porosity prior to leformation. Small white dots are copper rich particles, larger white lots in stringers have been identified to be iron rich particles.

PART C

1. <u>Cumulative List of Publications Resulting from AFOSR Support</u>

- 1980 1. LOW STRESS AND SUPERPLASTIC CREEP BEHAVIOR OF Zn-22% A1 EUTECTOID ALLOY, A. Arieli, A. K. S. Yu, and A. K. Mukherjee, <u>Metallurgical Transactions</u>, Vol. 11A, pp. 181-191, 1980.
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- 1984 9. MECHANICAL AND MICROSTRUCTURAL ASPECTS FOR OPTIMIZING THE SUPERPLASTIC DEFORMATION OF TI-6A1-4V ALLOY Β. Hidalgo-Prada, G. Gurewitz and Α. Κ. Mukherjee. Proceedings, Intn' Amer. Conf. on Mat. Tech., San Juan, Puerto Rico, Ed. D. Black, p.18-1, 1984.
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- 1986 18. 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.
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- 1. ON THE INTER-DEPENDENCE OF GRAIN BOUNDARY PHENOMENON AND SUPERPLASTIC DEFORMATION, B. P. Kashyap and A. K. Mukherjee, <u>Proceedings of Seiken Symposium</u>, Japan 1985.
- EFFECT OF HYDROSTATIC PRESSURE ON HIGH TEMPERATURE FAILURE IN SUPERPLASTIC ALUMINUM ALLOYS, A. H. Chokshi, J. E. Franklin and A. K. Mukherjee, Fifth International Conference on Mechanical Behavior of Materials, Beijing, China.

PART C Continued

2. List of Dissertations

- 1. Ph.D. Dissertation: "Hydrostatic Pressure Effects on Superplastic 7475 Aluminum Alloy," by Jerrold Franklin.
- 2. M.S. Dissertation: "Effect of Dynamic Microstructural Evolution During Superplastic Deformation in Ti-6Al-4V," by Michael Meier.

3. List of Personnel Involved in the Research

| ٦. | 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 _W and mechanically alloyed aluminum system). |
| 4. | Ms. Shara Hinote-Williams: | Report and Manuscript preparation. |

4. List of Cooperative Activities

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

