Microstructural and Physical Basis for Superplastic Cavitation in Aluminum Alloys

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Executive Summary

This report on Microstructural and Physical Basis for Superplastic Cavitation in Aluminum Alloys embodies work carried out jointly between PhD student Donghyun Bae and Professor Amit Ghosh and in direct collaboration with General Motors R&D Center, Pacific Northwest National Laboratory and Air Force Research Laboratory. Considerable new information on the initiation of nanoscopic damage in Al alloys, and the growth of voids through the microscopic and mesoscopic scales has been developed in this work. New information on the role of stress-state on void growth has been obtained. It has been shown that underpredictions of previous models of void growth are related to the lack of recognition of local stress-concentration effects surrounding the voids, persisting even at elevated temperatures.

The report is presented in the form of several papers, which also formed the chapters in the doctoral thesis by Dr. Bae. Some of the published papers are attached in the back of the report, and others are still in print. A total of ten research papers were generated as a result of this work.

GRAIN SIZE AND TEMPERATURE DEPENDENCE OF SUPERPLASTIC DEFORMATION IN AN Al - Mg ALLOY UNDER ISOSTRUCTURAL CONDITION

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ABSTRACT

The mechanical behavior of a superplastic Al-4.7%Mg-0.8%Mn-0.4%Cu alloy has been characterized by a new type of step strain-rate test which preserves the initial microstructure of the alloy (i.e. an isostructural test). Four different grain sizes of the alloy (8 to 30 μ m), prepared by variations in thermomechanical processing practice were examined. A sigmoidal relationship between $\log \sigma$ and $\log \dot{\epsilon}$ is observed for each isostructural condition. The value of maximum m ($= d \log \sigma / d \log \dot{\epsilon}$) increased with increasing temperature and with decreasing grain size. The isostructural $\log \sigma$ vs. $\log \dot{\epsilon}$ data are evaluated using the grain-mantle based quantitative model proposed by Ghosh. In the dislocation creep region ($\dot{\epsilon} > 10^{-1} \text{s}^{-1}$), the stress exponent is 4.55 and activation energy is close to that for lattice self-diffusion, but grain size exponent is non-zero (~ 0.37). In the grain mantle deformation region ($\dot{\epsilon} < 10^{-3} \text{s}^{-1}$), the value of stress exponent based on effective stress ($\sigma - \sigma_0$, where σ_0 is threshold stress) is ~ 1.7 , and grain size exponent is 2.3; but interestingly activation energy is the same as that for dislocation creep. Grain mantle creep is now believed to be controlled also by dislocation glide and climb processes, but its rate is enhanced many times due to a high concentration of vacancies near grain boundaries. σ_0 computed based on the model shows that it increases with increasing grain size and with decreasing temperature.

Keywords

superplasticity, aluminum alloy, thermally activated glide and climb, stress exponent, constitutive equation, grain mantle creep.

NOMENCLATURE

m	strain-rate sensitivity, $d \log \sigma / d \log \dot{\epsilon}$
σ	flow stress
σ_o	threshold stress
ϵ	strain
$\dot{\epsilon}$	strain-rate (s^{-1})
$\dot{\epsilon}_M$	grain mantle strain-rate (s^{-1})
$\dot{\epsilon}_C$	grain core strain-rate (s^{-1})
N	stress exponent for dislocation creep, $\log \dot{\epsilon}_C / \log \sigma$
$M (=1+q)$	stress exponent for grain mantle creep, $\log \dot{\epsilon}_M / \log(\sigma - \sigma_o)$
v	grain size exponent for dislocation creep, $-(d \log \dot{\epsilon}_C / d \log d)_{\sigma, T}$
μ	grain size exponent for grain mantle creep, $-(d \log \dot{\epsilon}_M / d \log d)_{f(\sigma), T}$
$f(\sigma)$	stress function in grain mantle creep, $(\sigma - \sigma_o)^M$
Q	activation energy
R	gas constant
T	absolute temperature in Kelvin
K, K', K''	constants in dislocation creep equation
B', B''	constants in grain mantle creep equation
d	grain size
t	time
L	length
A	specimen area
w_1, w_2, w_3, w_4	widths of different positions of tensile specimen
f_1, f_2	geometrical factors in equation (A1.2)
r	radius
<i>Subscripts:</i>	
avg	average
L	longitudinal direction
T	transverse direction
S	short transverse direction
a	region a
b	region b
c	region c
o	initial dimension

1 INTRODUCTION

Significant interest currently exists in the application of lightweight aluminum alloys in automotive body panels. With good corrosion resistance, a medium level of strength and a modest degree of superplasticity, an Al-4.7%Mg-0.8%Mn alloy has great potential for this application [1-3]. According to Watanabe *et al.* [4], the addition of a small amount of copper to this alloy was found to improve its superplastic formability. Such an alloy modified by the addition of 0.4%Cu was produced by Kaiser Aluminum Company. For this study, this alloy is utilized. The alloy contains dispersoid particles of an Al-Mn base intermetallic compound (e.g. Al_6Mn) containing Cu [4], which act as grain boundary pinning agents.

The understanding of the rate-controlling mechanism for superplastic deformation behavior is important not only from a scientific viewpoint but also for the practical application of such an alloy. However, the development of an accurate description of constitutive behaviors (stress vs. strain-rate) has posed a major challenge for researchers for many years because microstructural changes such as grain growth occurring during superplastic deformation may continuously influence flow stress, and such concurrent grain growth is dependent on strain-rate [5, 6]. During an incremental step strain-rate test, dynamic grain growth leads to increasing stress on the specimen at slower strain-rates, thus making it impossible to attain a level of steady state stress. Other difficulties for high temperature tensile test are the flow of soft superplastic material from the grip region into the gauge region [7-9] and the development of a non-uniform cross-section within the gauge region [10]. Certain alloys are more prone to these problems. Thus, all sections of a specimen experience different strain-rates, causing considerable difficulty in maintaining a constant strain-rate in the gauge region. Consequently, the measurement of accurate steady-state stress for constant microstructure as a function of strain-rate has been a major challenge. In this paper, an improved test method is used, which avoids incremental

loading steps for the slower strain-rates. To further improve the accuracy of test data, constant true strain-rate is maintained via control of cross-head speed (CHS) according to a recent model [7, 8].

The purposes of improved isostructural tests are to ascertain material behavior to a degree suitable for verifying relevant superplastic mechanisms and also to determine precisely the constants needed to fit the appropriate mechanistic model. This would permit predicting sheet forming behavior with greater confidence than previously possible. To set the stage for this study, we start with a brief review of the superplasticity mechanisms. The sigmoidal $\log \sigma$ vs. $\log \dot{\epsilon}$ curve is generally divided into three strain-rate regions to represent the different mechanisms [11]. A generalized creep equation is often used for each of three strain-rate regions [12]. This equation can be given by:

$\dot{\epsilon} = B \sigma^n d^{-p} \exp(-Q/RT)$ where B is a constant, d is grain size, σ is stress, and n , p , and Q are stress exponent, grain size exponent, and activation energy, respectively for each of the specific creep mechanisms. While there is controversy regarding the deformation mechanism operative in Region I ($\dot{\epsilon} < 10^{-5} \text{s}^{-1}$), it is sometimes interpreted as diffusional creep which may be modified by a threshold stress for grain boundary sliding (GBS) [11, 13] or GBS accommodated by a diffusion controlled process [14, 15]. The stress exponent in this region, as experimentally observed, is in the range of 3 to 4.8 for materials which exhibit good superplastic characteristics [16, 17] but even higher values are observed. In Region II ($10^{-5} \text{s}^{-1} < \dot{\epsilon} < 10^{-2} \text{s}^{-1}$), the dominant deformation mechanism is considered to be GBS, accommodated by diffusion or dislocation processes [14, 15, 18, 19], in which the stress exponent is usually between 1 and 2, and the activation energy is often quoted to be that for grain boundary diffusion [14, 16, 17]. Also higher activation energy similar to that of lattice diffusion or pipe diffusion has been reported [20, 21]. In Region III ($\dot{\epsilon} > 10^{-2} \text{s}^{-1}$), deformation is generally believed to be by dislocation climb with a stress exponent in the range of 4 to 5.5 (sometimes higher), and an activation energy which equals that for lattice self-diffusion [22].

In the grain neighbor exchange mechanism proposed by Ashby & Verrall [14], superplastic creep rate is viewed as a sum of two processes, dislocation creep (Region III) and GBS accommodated by diffusion creep (Regions I and II). The latter contribution is connected with shorter diffusion paths than those postulated by Nabarro-Herring and Coble creep [23-25]. This model assumes a linear relationship between "effective stress" ($\sigma - \sigma_o$) and strain-rate, where σ_o is threshold stress required to initiate the grain switching process. While their mechanism provides a reasonable explanation for the observed values of m ($= d \log \sigma / d \log \dot{\epsilon}$) in the three different regions, several basic problems in the details of the model are found. First, the grain switching event seems to end after a strain of 0.55. Second, evidence for grain extension followed by rapid return to their equilibrium shape is never seen experimentally. Third, identical diffusional flows must occur on each side of the grain boundary in an array of identical hexagonal grains, which prevents any boundary movements except for uniform translations [26]. Furthermore, experimentally measured threshold stress generally increases with increasing grain size and with decreasing temperature [27], which contradicts a basic feature of their model.

Alternative models of accommodation to GBS by dislocation climb along grain boundaries has been proposed, based on experimentally observed dislocation activity during superplastic deformation. These models are based on relief of stress concentrations at the ledges and protrusions on grain boundaries during the GBS process by injecting these dislocations into grains, and allowing them to climb along opposite boundaries [18, 19, 21]. These models always predict $m = 0.5$ over a wide range of strain-rates, which is not always observed in many studies on superplastic behavior.

A different view was postulated by Gifkins [28]. He proposed that within the grain mantle region where grain boundary dislocations pile up at triple point folds, it can provide a different response from that of the grain core. The stress concentration near grain boundaries may be relaxed by the dissociation of these dislocations into other grain boundary dislocations or into lattice dislocations, which accommodates sliding. He

assumed that accommodation in Region II may be either by dislocation climb-glide with a stress exponent of 2 or by grain boundary diffusion with a stress exponent of 1. However, the sigmoidal shaped $\log \sigma$ vs. $\log \dot{\epsilon}$ data cannot be expected directly from his model.

Inspired by Gifkins's core-mantle model, a constitutive model was proposed by Ghosh [29], in which the total strain-rate is also viewed as the sum of the strain-rate contributed from the grain mantle region and that within the grain core. As in Refs. 18 and 19, stress concentration is believed to arise at triple points, grain edges, particles on the grain boundaries when GBS tends to occur. Also as in the other models, dislocation glide initiates at these locations but emitted in multiple directions depending on the orientation of the active slip planes. They do not generally traverse across the entire grain to climb along opposite boundary. Local climb near the regions of stress concentrations can relieve the glide dislocations when the applied stress (or strain-rate) is low, but with increasing stress glide dislocations have sufficient velocity to spread out through a large part of the crystal before their relief by climb may occur. Thus, with increasing applied stress there is a gradual transition from climb control in the mantle region to increasing degree of glide-climb creep within the grains, all the way to Region III dislocation creep (e.g. Weertman creep), when dislocations traverse the entire grain. This transition is modeled in Ref. 29 by adding the localized mantle based creep process with background dislocation creep to predict a sigmoidal $\log \sigma$ vs. $\log \dot{\epsilon}$ curve. The exact shape of $\log \sigma$ - $\log \dot{\epsilon}$ curve depends on how the transition between the two extreme mechanisms occurs for a given material, and thus the predicted peak m values can approach 1, or as low as 0.4 or 0.3.

The grain size dependence of flow stress in this model arises from the number of grain boundary corners (and edges) per unit volume where glide activation can occur. Thus grain size dependence of mantle creep is large in Regions I and II for superplastic materials, but low for dislocation creep (as in Region III, or for coarse grain alloys, or at low test temperatures). Threshold stress in this model arises from the stress necessary to move

pinned glide dislocations at the grain boundaries, and it increases with increasing particle pinning.

Good agreement has already been seen between carefully developed $\log \sigma$ vs. $\log \dot{\epsilon}$ data and this model over a wide range of strain-rates [29]. However, a detailed study on the effects of grain size, temperature, and strain-rate has not been carried out to test the details of the model. In this study, $\log \sigma$ vs. $\log \dot{\epsilon}$ data were developed using the new step strain-rate test method. Using the fit between experimental data and the model, constants for the model equation, activation energy, stress exponent, grain size exponent, and threshold stress have been evaluated. The reason for selecting this method for comparison with data is the apparent flexibility of the constitutive equation over a wide range of grain sizes and temperatures, and its apparent realism.

A recent model by Fukuyo *et al.* [21] also addresses the cases of $m = 0.5$ and $m = 1.0$ by considerations of GBS accommodated by glide and climb as in Ref. 19 and also by glide alone. The details of their glide based model are not entirely clear because glide is generally considered to be thermally activated and has a nonlinear stress dependence. Furthermore, in the results to be presented in this work, the grain size and temperature effects on a family of $\log \sigma$ vs. $\log \dot{\epsilon}$ curves are found to change gradually, unlike the two distinct modes of behavior found in Ref. 21.

2 MATERIAL AND EXPERIMENTAL PROCEDURE

2.1 Material and Thermomechanical Processing

The test material for this study was an Al-Mg-Mn-Cu alloy, which was provided by Kaiser Aluminum Company in the as-cast condition. Its chemical composition is shown in Table 1. This ingot piece was given a homogenization heat treatment at 500°C for 12 hours in an air-circulating furnace. The homogenized material (larger than 30mm in thickness)

was forged to about 13mm at 250°C. To obtain sheets having different grain sizes, the forged material was cut into several different thicknesses and cold rolled to a final gauge of 2mm. The different levels of cold rolling reduction were 20, 56, 70, and 90%, respectively. The rolled sheets were recrystallized by static annealing at 500°C for 0.5 hour, followed by water quenching. These test materials were cut in three orthogonal sections, mechanically polished, and then etched with Graf Sergent etchant (15.5ml HNO₃, 0.5ml HF, 3g CrO₃, and 84ml water). Aging for 12 hours at 150°C prior to etching proved to be helpful to successfully reveal the grain structure by decorating grain boundaries by precipitates. Grain size was measured by the linear intercept method. Grain sizes of L (longitudinal), T (transverse), and S (short transverse) directions were measured separately. Average grain size was determined by the following relationship:

$$d_{avg} = \sqrt[3]{d_L \times d_T \times d_S} \quad (1)$$

where d_L , d_T , and d_S are the mean linear intercept grain sizes along L, T, and S directions, respectively.

Resulting micrographs are shown in Fig. 1. The grains were somewhat brick shaped rather than equiaxed. Fig. 2 shows the change in average grain size as a function of cold rolling reduction. The average grain sizes achieved by this process were 8.07, 10.2, 13.5, and 30.8μm respectively, for the different rolling reductions mentioned above. As the amount of cold rolling increases, the recrystallized grain size monotonically decreases.

2.2 Step Strain-rate Test

Incremental step strain-rate tests have generally been used in the past to determine the stress vs. strain-rate relationship in superplastic metals. Since such tests start with an initially slow strain-rate, dynamic grain growth and resulting strain hardening can occur at low $\dot{\epsilon}$, causing difficulty in attaining a steady state stress even after a significant amount of strain is applied to the specimen. Thus, it was decided that decremental step strain-rate tests might be more efficient to reach steady state stress and keep grain growth to a minimum. A

new schedule of step strain-rate decrements has been developed as shown in Fig. 3. The testing is performed in three stages; the first set of steps covers the lowest strain-rate range ($10^{-6}\text{s}^{-1} < \dot{\epsilon} < 10^{-3}\text{s}^{-1}$), the second set covers the intermediate strain-rate range ($10^{-3}\text{s}^{-1} < \dot{\epsilon} < 10^{-1}\text{s}^{-1}$), and the last set covers the highest strain-rate range ($\dot{\epsilon} > 10^{-1}\text{s}^{-1}$). The initial loading is nearly elastic, and is carried out at a rapid rate for a very short duration ($\approx 0.5\text{sec}$, at 10^{-2}s^{-1}). The purpose of this loading is to exceed the steady state stress levels for several of slower strain-rates on the basis of prior trial experiments. Immediately following this rapid loading, progressively slower strain-rates are applied to the specimen (e.g. $5 \times 10^{-4}\text{s}^{-1}$, 10^{-4}s^{-1} , 10^{-5}s^{-1} , etc.) to achieve the steady load level for each individual $\dot{\epsilon}$ within a short time duration. (Constant true strain-rate during each step is maintained via cross-head speed (CHS) control according to Refs. 7 and 8, which is described in Appendix 1). Attainment of steady load level for each $\dot{\epsilon}$ is assumed to indicate a near steady state condition for stress, and when this is achieved, the next decrement to lower strain-rate is initiated. In the intermediate strain-rate range, again rapid loading at $\dot{\epsilon} = 10^{-2}\text{s}^{-1}$ was initiated and maintained for 1.5 s to obtain a stress level high enough to exceed the steady state stresses for $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$ to $\dot{\epsilon} = 5 \times 10^{-3}\text{s}^{-1}$. Now decremental $\dot{\epsilon}$ steps are started immediately to obtain the steady loads corresponding to these strain-rates. Finally the last $\dot{\epsilon}$ range is investigated in a similar manner. The entire data now cover over 5 orders of magnitude of strain-rates with an accumulated strain of about 0.1. Grain size measured after this test is found to be the same as the initial material*. Thus, this method was deemed satisfactory for our new isostructural tests.

Using this method, step strain-rate tests were conducted on the test materials having four different grain sizes, in the temperature range of 450 to 550°C. The tests were controlled by computer through a digital interface board on a 4505 Instron machine fitted

* For example, after one complete strain-rate cycle (at 500°C), the average grain size was measured as 8.12 μm for an initial grain size of 8.07 μm , and was 10.5 μm for an initial grain size of 10.2 μm , which is well within the scatter band of grain size data.

with a clamshell furnace with three independent heating zones (temperature control within $\pm 1^\circ\text{C}$).

3 RESULTS AND DISCUSSION

3.1 Stress versus Strain-rate Relationship

Stress vs. strain-rate relationships at several different temperatures are plotted in a double-logarithmic scale in Fig. 4 for three different grain sizes: (a) $8.07\mu\text{m}$, (b) $13.5\mu\text{m}$, and (c) $30.8\mu\text{m}$. For a grain size of $8.07\mu\text{m}$ (a), the sigmoidal shaped curves in $\log\sigma$ versus $\log\dot{\epsilon}$ plots are observed. However, as the temperature decreases, the level of flow stress increases and the curve shape changes toward a flatter configuration. This type behavior is also observed for other grain sizes ((b) and (c)). For the largest grain size ($d = 30.8\mu\text{m}$), however, $\log\sigma - \log\dot{\epsilon}$ plot at 450°C is nearly a straight line over the entire range of strain-rate.

The grain size dependence of this relationship at a temperature of 550°C is shown in Fig. 5. As the grain size increases, again the sigmoidal curve shape becomes flatter and the level of flow stress increases over the entire range of strain-rate (similar to the reduced temperature effect). The increase in the logarithmic value of flow stress is greater at lower strain-rates, and a smaller increase in the logarithmic value of flow stress is seen at higher strain-rates. The increase in flow stress with increasing grain size in the high strain-rate range ($\dot{\epsilon} > 10^{-1}\text{s}^{-1}$) is not usually reported but has been observed in this work as well as those by Shei and Langdon [17] and Barrett *et al.* [30].

3.2 Constitutive Parameters for Mechanism Based Equation

To obtain an improved understanding of the mechanisms involved and to determine the strain-rate sensitivities for different regions of superplastic behavior, it is best to fit the isostructural data by a physically based yet mathematically flexible constitutive relation. A constitutive equation developed by Ghosh [29] is applied to the stress vs. strain-rate data shown in Fig. 4 and 5. As mentioned in the Introduction, this constitutive relation was based on a modified mantle-core concept [28], in which the total strain-rate ($\dot{\epsilon}$) is viewed as a sum of (i) grain mantle region contribution ($\dot{\epsilon}_M$) accommodated by climb in the mantle region and (ii) dislocation creep contribution ($\dot{\epsilon}_C$) within the grain core. The detail of the mantle process (local climb) is entirely different from Gifkins's model, and the total strain-rate according to this model is given by

$$\dot{\epsilon} = \dot{\epsilon}_M + \dot{\epsilon}_C = B' (\sigma - \sigma_o)^M + K \sigma^N \quad (2)$$

where $B' (= B/d^2, d$ is the grain size, B is a constant) and K are temperature and structure dependent constants, M is the stress exponent for the grain mantle deformation ($M = 1+q$ in Ref. 29), N is the stress exponent for the grain core creep (normally referred to as n for dislocation creep), and σ_o is the threshold stress. For this alloy, insoluble dispersoid particles or triple junctions lead to a threshold stress effect, which is typical of most (if not all) superplastic alloys [29, 31, 32] and will be discussed later. The parameters based on Eq. (2) which describe the constitutive behavior for data presented in Fig. 4 and 5 are determined by a curve-fitting method (see Appendix 2). By applying the analytical method to the experimental results for all test temperatures and grain sizes, the various constitutive parameters (B', σ_o, M, K , and N) were calculated, and are listed in Table 2. As the grain size increases, B' and K are found to decrease, while M and σ_o increase, which will be discussed in section 3.4.

3.3 Strain-rate Sensitivity

The curve fits provide a method to describe the experimental results in the form of a single equation (Eq. (2)) and microscopic mechanisms involved. The important rheological parameters related to the mechanisms of superplastic flow can be obtained by differentiating the fitted constitutive equation. This differential ($d \log \sigma / d \log \dot{\epsilon}$), commonly recognized as strain-rate sensitivity (m) is calculated from the constitutive equation by a simple incremental computation. This calculation involves taking small increments in the value of σ (from σ_1 to σ_2), calculating the corresponding values of $\dot{\epsilon}$ ($\dot{\epsilon}_1$ and $\dot{\epsilon}_2$) from Eq. (2), and then determining the ratio: $[\log(\sigma_2/\sigma_1) / \log(\dot{\epsilon}_2/\dot{\epsilon}_1)]$ as m . Fig. 6 shows plots of m vs. $\log \dot{\epsilon}$. The temperature dependence of m at a grain size of $8.07 \mu\text{m}$ is shown in Fig. 6(a), and the grain size dependence of m at a temperature of 550°C is shown in Fig. 6(b). The peak value of m is about 0.6 at 550°C and $\dot{\epsilon} = 4 \times 10^{-3} \text{s}^{-1}$ for the $8 \mu\text{m}$ material. The main trends for data on m are as follows:

- i) The value of m approaches between 0 and 0.1 at very low strain-rates, and about 0.22 at high strain-rates, with maximum m occurring at an intermediate strain-rate (10^{-3} - 10^{-2}s^{-1} for the $8 \mu\text{m}$ material).
- ii) The strain-rate corresponding to peak m is displaced to lower strain-rates as the temperature decreases. For increasing grain size, the peak m location is displaced from higher $\dot{\epsilon}$ toward lower $\dot{\epsilon}$ (i.e. from $4 \times 10^{-3} \text{s}^{-1} \rightarrow 6 \times 10^{-5} \text{s}^{-1}$) at a fixed temperature.
- iii) The magnitude of maximum m increases with increasing temperature, and decreasing grain size.

3.4 Temperature and Grain Size Dependence of Constitutive Parameters

Since the isostructural $\log \sigma$ - $\log \dot{\epsilon}$ data have been analyzed in this study over a wide range of temperature and strain-rate, it is possible to determine the activation energies

for creep in the two different mechanistic regimes discussed in section 3.2. In addition, the grain size and temperature dependencies of stress exponent and threshold stress can be analyzed.

3.4.1 High Strain-rate Region

In the high strain-rate region ($\dot{\epsilon} > 10^{-1} \text{s}^{-1}$), deformation behavior follows dislocation creep with the constitutive equation of the form: $\dot{\epsilon}_c = K\sigma^N$ as in Eq. (2). Fig. 7(a) shows plots of K as a function of grain size and temperature. K increases with increasing temperature and decreasing grain size. The grain size dependence of K may be expressed approximately by $K = K' d^{-\nu}$ where K' and ν are constants. Further, the thermal activation of K' may be introduced, so $\dot{\epsilon}_c$ may be expressed in an exponential form [33-35] as

$$\dot{\epsilon}_c = K'' d^{-\nu} \exp(-Q/RT) \sigma^N \quad (3)$$

where K'' a constant within K' , R is the gas constant, and T is the absolute temperature, and Q , N , and ν are respectively activation energy, stress exponent, and grain size exponent for the dislocation creep mechanism. Generally Weertman's creep model [36] for coupled dislocation glide-climb process is widely used to explain dislocation creep behavior. This model predicts a value of N as 4.5, but no grain size dependence exists in this model (i.e. $\nu = 0$). Q in this model is the activation energy for lattice self-diffusion. In this study, the general methods for the determination of the values of N , Q , and ν in Eq. (3) were used as follows.

The stress exponent ($N = [\log \dot{\epsilon}_c / \log \sigma]_{T,d}$) is directly measurable in the $\log \sigma$ vs. $\log \dot{\epsilon}$ curve (see Appendix 2) and is plotted in Fig. 7(b). The experimentally measured N value is found to be approximately 4.55 over the range of investigation, in good agreement with Weertman creep [36].

The creep activation energy (Q) for core creep can be obtained for constant stress and grain size by differentiating Eq. (3) as given by

$$Q = -R \left[\frac{d \ln \dot{\epsilon}_c}{d(1/T)} \right]_{\sigma, d} \quad (4)$$

A value of Q for isostructural data can now be obtained from the slope of a logarithmic plot of $\dot{\epsilon}_c$ versus $(1/T)$. Fig. 8(a) shows such a plot for a constant stress of 40MPa for three different grain sizes. The slope of this plot ($= -Q / 2.303R$) yields a value of Q around 163(kJ/mol)* (see Table 3(a)), which is slightly higher than that of lattice self-diffusion for pure aluminum (142kJ/mol [37]). It is believed that this small increase in activation energy for the Al-Mg alloy could partly be due to the effects of interaction between dislocations and dispersed particles or solute atoms, and also partly a result of experimental scatter.

Again from Eq. (3), the grain size exponent (ν) for constant stress and temperature is expressed as

$$\nu = - \left[\frac{d \log \dot{\epsilon}_c}{d \log d} \right]_{\sigma, T} \quad (5)$$

It is found that ν is approximately 0.37 at 40MPa (see Table 3(b)) as shown in Fig. 8(b).

Reported creep data [38] generally show no grain size dependence for power-law creep.

Exceptions are: (i) Shei and Langdon [17] reported a value of $\nu = 1.2$ for copper alloy of 3

$\sim 11\mu\text{m}$ grain size and (ii) Barrett *et al.* [30] reported a value of $\nu = 1$ for pure copper of

* The activation energy can be also expressed as $Q = -R \left[\frac{d \ln(\dot{\epsilon}_c G^{N-1} T)}{d(1/T)} \right]$ by compensating for the temperature dependence of shear modulus using the creep equation: $\dot{\epsilon}_c = K''' \left(\frac{G}{T} \right) \exp \left(\frac{-Q}{RT} \right) \left(\frac{\sigma}{G} \right)^N$ where K''' is a material constant, G is the shear modulus. The values of G as a function of temperature were estimated using the data for pure aluminum: $G(\text{MPa}) = 29483.6 - 13.612T$ (K) [37]. The calculated activation energy for $\sigma = 40\text{MPa}$ was 155.1(kJ/mol) which is slightly lower than the above value but well within the range of data scatter.

less than 50 μm grain size. Both of the studies suggested that the responsible mechanism might be grain boundary sliding or grain boundary shearing. For the fine-grained materials the enhanced grain size dependency may be possible due to the local stress and strain concentration near grain boundary triple points, dispersoid particles and ledges on grain boundaries, which provide an additional deformation mechanism in addition to the ongoing grain boundary sliding strain [39]. If this mechanism were operative, the creep contribution from the grain corners would decrease with increasing grain size (as the number density of grain corners decreases). The magnitude of grain size dependence observed here differs somewhat from those in Refs. 17 and 30, possibly because the micromechanism for such effects is complex, producing varying dependencies as a function of alloy, test temperature, and strain-rate.

3.4.2 Low and Intermediate Strain-rate Region

In the low and intermediate strain-rate region ($\dot{\epsilon} < 10^{-3} \text{s}^{-1}$), deformation behavior is described as grain mantle creep with a constitutive equation [29]: $\dot{\epsilon}_M = B' (\sigma - \sigma_o)^M$ as in Eq. (2). The experimentally obtained B' term is plotted in Fig. 9(a). B' is also found to be a function of grain size and temperature - it increases with increasing temperature and decreasing grain size, thereby aiding the creep deformation process. We believe that the temperature and grain size dependencies of B' may be expressed as

$$B' = B'' d^{-\mu} \exp(-Q/RT) \quad (6)$$

where B'' is a constant, and Q and μ are respectively activation energy and grain size exponent for the grain mantle deformation mechanism. Thus, the aforementioned mantle creep equation may be rewritten as

$$\dot{\epsilon}_M = (B'' d^{-\mu} \exp(-Q/RT)) (\sigma - \sigma_o)^M \quad (7)$$

To obtain the values of the various constants and their temperature and grain size dependencies, the stress function in Eq. (7): $f(\sigma) = (\sigma - \sigma_o)^M$ is separated out first.

Thus, in the following section, the values of M and σ_o are examined first, then those of Q and μ , which are calculated by the methods shown in Appendix 3 and 4, respectively.

The stress exponent ($M = [\log \dot{\epsilon}_M / \log(\sigma - \sigma_o)]_{T,d}$) is obtained from curve fitting to data (see Appendix 2) and is plotted in Fig. 9(b). The value of M is found to be in the range of 1.63 - 1.82 for this alloy, its value decreasing slightly from 1.82 as the test temperature increases. Generally M is expected to be a constant larger than 1, its value being related to the exponent of stress describing the number of activated glide sources on the grain boundaries [29]. (Discontinuities such as ledges, non-deformable particles, and triple junctions on grain boundaries act as dislocation sources.) The number of activated sources was postulated to increase with increasing effective stress according to a power law, thus the decay in its exponent may be due to recovery effects causing M to drop slightly.

In the low $\dot{\epsilon}$ region, the role of threshold stress, σ_o , is important, which is estimated by the curve fitting method (see Appendix 2), and is plotted as a function of temperature in Fig. 10. As the test temperature increases, the threshold stress decreases almost linearly, approaching zero at the incipient melting temperature ($\sim 580^\circ\text{C}$). As for comparison, in the work of Mohamed [31], the threshold stress was calculated by the extrapolation method using $M = 2$, which produced an exponential relationship with $(1/T)$ in a Zn-22%Al alloy. In our case, the value of the relevant M is based on actual data, rather than an assumed value of M .

The threshold stress also increases with increasing grain size. The present findings on temperature and grain size dependencies of threshold stress are in agreement with the data [27], but in contradiction with the model proposed by Ashby & Verrall [14]. As stated above, the threshold stress is related to initiation of glide at triple junctions, ledges, or particles at the grain boundaries, and not diffusion. However, diffusion and recovery effects can reduce this "yield strength" via thermal activation. As the temperature increases, thermal vibration of pinned dislocations enhances their mobility; these dislocations can then

overcome obstacles more easily to initiate the glide process. On the other hand, for smaller grain sizes, the number of grain corners as well as particles per unit volume lying on grain boundaries or triple junctions are statistically more. Thus, if more glide sources are activated in the fine grain metals, a greater creep strain can be expected in comparison to coarse grain metals. This leads to lower flow stress in the fine grain metals.

The determination of activation energy for grain mantle creep has been made by analyzing $\dot{\epsilon}_M$ data as a function of $(1/T)$ for constant values of the stress function, $f(\sigma) = (\sigma - \sigma_o)^M$, and grain size, d , as shown in Appendix 3. The average value of Q for grain mantle creep is found to be 166(kJ/mol) (see Table 4(a)), which is about the same as the activation energy for dislocation creep found in this study. This result suggests that the mass transport for creep in the grain mantle region is by climb, similar to intragranular creep, but its rate is significantly higher possibly due to the presence of a large number of vacancy sources and sinks near the grain boundary which increase the pre-exponential factor in the diffusion equation. This was postulated by Clark and Alden [5]. Thus, superplastic deformation in metals may not involve true grain boundary diffusivity (the activation energy for grain boundary diffusion for pure aluminum is about 84 (kJ/mol) [37]), but simply vacancy-enhanced climb creep near the grain boundaries [5] which modifies the parameter, B'' , in Eq. (6). The concept of glide and climb occurring near grain boundary region is also supported by TEM observation of dislocation density [40, 41], arising due to strain concentration near grain boundaries and corners. While our finding of the activation energy for both grain mantle creep and dislocation creep being similar might appear surprising to some, there are findings to support such results. For example, Fukuyo *et al.* [21] also obtained similar results but not due to a correction for threshold stress, nor due to isostructural test data.

Finally the grain size exponent for grain mantle creep has been estimated by analyzing the grain size dependency of $\dot{\epsilon}_M$, again for constant values of the stress function, $f(\sigma) = (\sigma - \sigma_o)^M$, and temperature as shown in Appendix 4. The grain size exponent (μ)

is found to be 2.3 (see Table 4(b)). This value is fairly close to that for Nabarro-Herring creep ($\mu = 2.0$).

Superplastic deformation behavior, therefore, may be interpreted on the basis of the same rate-controlling glide-climb micromechanism over a wide range of strain-rates, but modified at low and intermediate $\dot{\epsilon}$ by activities around the grain boundaries and grain corners. At high test temperatures and low stresses, the high vacancy concentration existing near grain boundaries can provide a significant enhancement in creep rate to account for almost all of the applied $\dot{\epsilon}$. Grain boundary sliding and grain rotation effects are a direct result of this mechanism at low $\dot{\epsilon}$ and high temperatures. At higher applied $\dot{\epsilon}$, glide and climb spread into the rest of the grain, leading to increasing contribution of dislocation creep to the overall deformation process.

4 SUMMARY AND CONCLUSIONS

In a superplastic Al-Mg-Mn-Cu alloy, the relationship between $\log \sigma$ and $\log \dot{\epsilon}$ has been investigated for grain sizes in the range of 8 to 30 μm and in the temperature range of 450 to 550°C. The data were acquired using a new step strain-rate test method designed to preserve a nearly isostructural condition during test. Thus, dynamic grain growth and its effect on the measured flow stress were eliminated using this test method. Constants for the constitutive equation, including activation energy, threshold stress, stress exponent, and grain size exponent have been evaluated over the wide strain-rate range with good confidence, using a model proposed by Ghosh [29]. Contrary to the general belief, the activation energies in both grain mantle creep and dislocation creep are found to be the same as that for lattice self-diffusion. A larger $\dot{\epsilon}$ contribution from near grain boundaries and grain corners is due to enhanced diffusivity from the high concentration of vacancies

existing there [5]. The results support the concept that mass transport near grain boundaries occurs due to climb but initiated by dislocation glide at grain boundary sources [29]. The present observations lead to the following conclusions:

1. The sigmoidal nature of the relationship between $\log \sigma$ and $\log \dot{\epsilon}$ in the regime of investigation is the alloy's inherent isostructural response and not due to grain size changes during test. With decreasing test temperature and increasing grain size, the flow stress increases and the shape of $\log \sigma$ - $\log \dot{\epsilon}$ curve gradually changes toward a flatter configuration, still maintaining a consistent pattern as a family of curves.

2. The strain-rate corresponding to peak m is displaced to lower strain-rates as the test temperature decreases, and also as the grain size increases. The magnitude of peak m increases with increasing temperature and decreasing grain size, and its value is found to be 0.6 at 550°C and $\dot{\epsilon} = 4 \times 10^{-3} \text{ s}^{-1}$ for the 8 μm grain size material.

3. The mechanical data fit well with a model equation proposed by Ghosh [29] with high precision over the entire strain-rate range. In that model, the total deformation is viewed as a combination of a dislocation based mantle creep and dislocation creep within the grain core.

4. In the high strain-rate region ($\dot{\epsilon} > 10^{-1} \text{ s}^{-1}$), the activation energy for creep is found to be around 163(kJ/mol), and the stress exponent is approximately 4.55, which relates to a coupled dislocation glide-climb process within grain core. However, there is a small grain size dependence in this region (grain size exponent = 0.37), indicating that stress concentration and shear near the grain boundaries would produce additional contribution to creep.

5. Computed threshold stress (σ_0) obtained by fitting to Eq. (2) shows that threshold stress ranges from 0.25 to 4 MPa, and linearly decreases with increasing temperature. Decreasing grain size also leads to a decrease in σ_0 .

6. The presence of threshold stress makes the determination of activation energy more complex in the low and intermediate strain-rate region ($\dot{\epsilon} < 10^{-3} \text{s}^{-1}$). Based on the relationship between $\log(\sigma - \sigma_0)$ and $\log \dot{\epsilon}$, the activation energy for mantle region creep is found to be around 166(kJ/mol), suggesting that activation of glide and climb in the grain mantle region at low stresses initiates the process of grain boundary sliding [29]. The rate of this creep is however rapid due to a high concentration of vacancies near grain boundaries [5]. The stress exponent is not fixed at 2, but varies between 1.63 and 1.82. Yet the peak m value ranges from 0.4 to 0.65, depending on the mix of the two creep components. The grain size exponent is found to be around 2.3.

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Table 1. Chemical composition of an alloy (wt%)

Mg	Mn	Cu	Cr	Zr	Al
4.75	0.8	0.4	0.2	0.2	Bal.

Table 2. Constants in Constitutive Equation, Eq. (2)

d_{avg} (μm)	Temp. ($^{\circ}\text{C}$)	σ_o (MPa)	B' ($\text{MPa} \cdot \text{s}^{-1}$)	K ($\text{MPa} \cdot \text{s}^{-1}$)	M	N
8.07	450	1.97	1.53×10^{-5}	7.50×10^{-10}	1.80	4.55*
	475	1.53	2.04×10^{-5}	1.70×10^{-9}	1.776	4.77
	500	1.025	4.39×10^{-5}	5.05×10^{-9}	1.738	4.55*
	525	0.662	1.10×10^{-4}	9.50×10^{-9}	1.680	4.55*
	550	0.255	2.50×10^{-4}	1.90×10^{-8}	1.631	4.55*
10.2	450	3.35	3.80×10^{-6}	5.10×10^{-10}	1.828	4.68
	500	1.423	1.54×10^{-5}	3.80×10^{-9}	1.77	4.55*
	550	0.33	1.18×10^{-4}	1.70×10^{-8}	1.635	4.55*
13.5	450	3.68	2.80×10^{-6}	7.50×10^{-10}	1.82	4.55
	500	1.52	1.01×10^{-5}	3.70×10^{-9}	1.76	4.35
	550	0.372	6.85×10^{-5}	1.70×10^{-8}	1.65	4.55*
30.8	450	-	-	4.0×10^{-10}	-	4.36
	500	2.42	1.69×10^{-6}	2.60×10^{-9}	1.785	4.35
	550	0.843	9.48×10^{-6}	1.30×10^{-8}	1.7	4.37

* The reported N value is the average of the following experimental conditions: 8.07 μm (400 $^{\circ}\text{C}$: 4.92, 475 $^{\circ}\text{C}$: 4.77), 10.2 μm (450 $^{\circ}\text{C}$: 4.68), 13.5 μm (450 $^{\circ}\text{C}$: 4.55, 500 $^{\circ}\text{C}$: 4.35), 30.8 μm (450 $^{\circ}\text{C}$: 4.36, 500 $^{\circ}\text{C}$: 4.35, 550 $^{\circ}\text{C}$: 4.37).

Table 3. Calculated Parameters for Dislocation Creep [from Eq. (3)]

(a)		(b)	
Grain Size (μm)	Activation Energy (Q (kJ/mol))	Temp. ($^{\circ}\text{C}$)	Grain Size Exponent (ν)
8.07	162.3	550	0.27
13.5	154.3	500	0.48
30.8	172.2	450	0.36

Table 4. Calculated Parameters for Grain Mantle Creep [from Eq. (7)]

(a)		(b)	
Grain Size (μm)	Activation Energy (Q (kJ/mol))	Temp. ($^{\circ}\text{C}$)	Grain Size Exponent (μ)
8.07	172.5	550	2.4
10.2	169.0	500	2.3
13.5	157.3		

Figure Captions

Fig. 1. Optical micrographs of a recrystallized Al-Mg-Mn-Cu alloy on the L-S plane: (a) $d_{avg} = 8.07\mu\text{m}$, (b) $d_{avg} = 13.5\mu\text{m}$, (c) $d_{avg} = 30.8\mu\text{m}$.

Fig. 2. Linear intercept grain size (averaged over three orthogonal directions) for a recrystallized Al-Mg-Mn-Cu alloy shown as a function of cold rolling reduction.

Fig. 3. Load versus displacement plot during step strain-rate test designed to preserve a nearly isostructural condition. The testing is performed in three stages: low, intermediate, and high strain-rate ranges. In each stage, initial loading is nearly elastic, and then progressively slower strain-rates are applied to the specimen (decremental test). Each step is controlled to achieve a steady stress level within a short time duration (i.e. small increment of strain).

Fig. 4. Temperature dependence of the stress versus strain-rate relationship of an Al-Mg-Mn-Cu alloy for three grain sizes: (a) $8.07\mu\text{m}$, (b) $13.5\mu\text{m}$, and (c) $30.8\mu\text{m}$. As the temperature decreases, the level of flow stress increases and the curve shape changes from sigmoidal toward a flatter configuration.

Fig. 5. Grain size dependence of the stress vs. strain-rate relationship for an Al-Mg-Mn-Cu alloy at a temperature of 550°C . As the grain size increases, the level of flow stress increases and the sigmoidal curve shape becomes flatter.

Fig. 6. The dependence of strain-rate sensitivity (m) on (a) temperature at a grain size of $8.07\mu\text{m}$ and on (b) grain size at a temperature of 550°C . The strain-rate for peak m is displaced to the lower strain-rates with decreasing temperature and with increasing grain size.

Fig. 7. (a) The temperature dependence of (a) K , (b) stress exponent (N) for several different grain sizes in the constitutive equation: $\dot{\epsilon}_C = K\sigma^N$ for an Al-Mg-Mn-Cu alloy. K increases with increasing temperature and with decreasing grain size. However, N is nearly constant over the wide range of temperature and grain size.

Fig. 8. (a) The relationship between grain core strain rate ($\dot{\epsilon}_C$) at a constant stress of 40MPa and the reciprocal of absolute temperature for three different grain sizes. (b) The relationship between grain core strain rate ($\dot{\epsilon}_C$) at a constant stress of 40MPa and grain size for three different temperatures.

Fig. 9. (a) The temperature dependence of (a) B' , (b) stress exponent (M) for several different grain sizes in the constitutive equation: $\dot{\epsilon}_M = B'(\sigma - \sigma_o)^M$ for an Al-Mg-Mn-Cu alloy. B' increases with increasing temperature and with decreasing grain size. However, M slightly decreases with increasing temperature and with decreasing grain size.

Fig. 10. The relationship between threshold stress, σ_o , and temperature in an Al-Mg-Mn-Cu alloy, in which threshold stress is obtained by the curve fitting method (see Appendix 2).

Appendix Figure Captions

Fig. A1. In the analysis of the amount of material flow during a uniaxial tension test, (a) schematics of three regions in a 12.7mm gauge length specimen and a specimen holder. (b) Calculated cross-head speeds based on the model and exponential cross-head speeds. (c) Calculated lengthening rates for three different regions and cross-head speed plotted as a function of strain at $4 \times 10^{-4} \text{s}^{-1}$. (d) Measured area-based strain versus calculated strain at several strain-rates at 550°C .

Fig. A2. (a) An illustration of the curve fitting method to determine the constants: B' , σ_o , and M in the mantle deformation region ($\dot{\epsilon}_M = B'(\sigma - \sigma_o)^M$). The data is for an Al-Mg-Mn-Cu alloy at $d = 8.07\mu\text{m}$, $T = 500^\circ\text{C}$. For strain-rates below that of peak m , various trial values of σ_o are used to compute $\log(\sigma - \sigma_o)$ from the experimental stress data, and then plotted against $\log \dot{\epsilon}_M$. Of the various nonlinear plots, a linear plot is found for $\sigma_o = 1.025\text{MPa}$. For this plot, B' is obtained when $\log(\sigma - \sigma_o)$ is zero, and M = the slope of this straight line. (b) Using a fixed N of 4.55, K is adjusted in the high $\dot{\epsilon}$ region until the combination of high and low $\dot{\epsilon}$ data gives the best fit through data in the intermediate $\dot{\epsilon}$ region.

Fig. A3. (a) The relationship between $(\sigma - \sigma_o)$ and $\dot{\epsilon}_M$ for a grain size of $8.07\mu\text{m}$ in which solid lines result from curve fitting results and the slope ($= d \log \dot{\epsilon}_M / d \log(\sigma - \sigma_o)$) is M (see Table 2), and $\dot{\epsilon}_M$ versus $(1/T)$ curves at fixed values of $(\sigma - \sigma_o) = 0.3$ and 1 MPa . (b) The relationship between $(\sigma - \sigma_o)^M$ and $\dot{\epsilon}_M$ for for a grain size of $8.07\mu\text{m}$ in which the slope ($= d \log \dot{\epsilon}_M / d \log(\sigma - \sigma_o)^M$) is 1, and $\dot{\epsilon}_M$ versus $(1/T)$ curves at fixed values of $(\sigma - \sigma_o)^M = 1 \text{ MPa}^M$ for two different grain sizes.

Fig. A4. (a) $(\sigma - \sigma_o)^M$ vs. $\dot{\epsilon}_M$ plot for four different grain sizes at a temperature of 550°C , in which solid lines result from curve fitting results (Table 2) and the slope ($= d \log \dot{\epsilon}_M / d \log(\sigma - \sigma_o)^M$) is 1. (b) $\dot{\epsilon}_M$ versus d curves at a fixed value of $(\sigma - \sigma_o)^M = 1 \text{ MPa}^M$ for two different temperatures.

(a) $d_{avg} = 8.07\mu\text{m}$

(b) $d_{avg} = 13.5\mu\text{m}$

(c) $d_{avg} = 30.8\mu\text{m}$

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Appendix 1. Constant Strain-rate Tensile Test

It has become increasingly apparent in recent times that maintaining a constant true strain-rate during a superplastic tensile test is difficult [7-9], particularly for the small gauge length test specimens used in such tests. One problem is that a portion of the soft superplastic material from the grip region can flow into the gauge section, thus affecting cross-head displacement even in the so called cross-head controlled tests [9]. Exponential cross-head speed (CHS) cycles developed previously ($\text{CHS} = L_0 \exp(\dot{\epsilon} t) \dot{\epsilon}$, where $\dot{\epsilon}$ = strain-rate, L_0 = initial gauge length, and t = time) have now been found to result in significant departures from constant strain-rate.

To correct for the grip region flow problem, an approximate calculation of grip deformation was carried out in Refs. 7 and 8 for different values of m representing different test conditions. A schematic of the different regions of a specimen contributing to the overall flow is shown in Fig. A1(a). The specimen is divided into three distinct regions for this analysis. Region (a) is a uniform cross-section that experiences uniaxial straining, region (b) is a radius region, and region (c) is a grip region located between two dead zones where no significant straining occurs. The specimen is both edge-loaded from the grips by contacting the specimen shoulders with the specimen holder (Fig. A1(a)), and face-loaded by blocks (not shown) that squeeze the grip region and help minimize straining of the grip region. Although the face loading helps to contain material in the grip region, the benefits from this are limited at elevated testing temperatures. A sharp transition radius between the grip and the gauge is also used to minimize the amount of material flowing into the gauge section. Further, to minimize the end effect, the width of the gauge section is made considerably smaller than that of the grip section.

Details of the deformation analysis of the grip are given in Ref. 7 but a brief summary is given below. In the specimen deformation model, upon imposing force balance along the specimen axis, we obtain:

$$\sigma_a A_a = \sigma_b A_b = \sigma_c A_c \quad (\text{A1.1})$$

where A refers to current cross-sectional area and the subscripts a , b , and c refer to the respective regions shown in Fig. A1(a). The CHS in region (a) is found by computing the extension rates of the three regions, interrelated through Eq. (A1) and given by:

$$\text{CHS} = L_a \exp(\epsilon_a) \dot{\epsilon}_a + L_b \exp(\epsilon_b) \dot{\epsilon}_b + L_c \exp(\epsilon_c) \dot{\epsilon}_c \quad (\text{A1.2})$$

where: L_a, L_b, L_c : initial length of region (a), (b), and (c), respectively

$\epsilon_a, \epsilon_b, \epsilon_c$: axial strain of region(a) = $\int \dot{\epsilon}_a dt$, region (b) = $\int (f_1)^{\frac{1}{m}} \dot{\epsilon}_a dt$, and

region (c) = $\int (f_2)^{\frac{1}{m}} \dot{\epsilon}_a dt$, respectively

w_1 : width of region (a)

w_2 : $w_1 + r$ (r = radius of region (b))

w_3 : $(0.75w_4 + 0.25w_1) \times 0.5$, assumed from empirical observation

w_4 : grip width

f_1 : geometric factor of region (b) = (w_1/w_2)

f_2 : geometric factor of region (c) = (w_1/w_3)

m : strain-rate sensitivity determined by a preliminary step strain-rate test,
which is a function of strain-rate.

Calculated CHS profile and exponential CHS profile are plotted in Fig. A1(b) for two different strain-rates (12.7mm gauge length specimen). The lengthening rates of each region and CHS at a strain-rate of $4 \times 10^{-4} \text{ s}^{-1}$ are shown in Fig. A1(c). The new CHS profile is initially faster than exponential CHS, but as straining continues, the new profile shows slower rates than the exponential one due to increasing differences in the gauge and grip cross-section areas. The new CHS profile has been compared to actual strain measurements from tested specimens and shown in Fig. A1(d). The measured strains from samples are local area-based strains: $\epsilon = \ln(A_0/A)$ where A_0 is the initial cross-section area and A is the current cross-section area. The new CHS cycle is reasonably accurate up to large strains for this alloy.

Appendix 2. Determination of Constitutive Parameters in Eq. (2) by Curve Fitting Method

An illustration of curve fitting method to determine parameters in Eq. (2) is shown in Fig. A2 for $d = 8.07 \mu\text{m}$, $T = 500^\circ\text{C}$. For strain-rates below that of peak m , the primary creep contribution is from: $\dot{\epsilon}_M = B'(\sigma - \sigma_0)^M$. Fitting to this equation starts by selecting various trial values of σ_0 which are used to compute corresponding values of $\log(\sigma - \sigma_0)$ from the experimental stress data, and then plotting against $\log(\dot{\epsilon}_M)$ as shown in Fig. A2(a). Of the various nonlinear fits, the plot with a σ_0 value of 1.025 MPa exhibits essentially linear behavior satisfying the above equation. B' is obtained from the logarithmic plot by noting the strain-rate for $\log(\sigma - \sigma_0) = \text{zero}$. M is obtained as the slope of this straight line. For the test condition shown in Fig. A2(a), the constants for the equation are, thus, $\sigma_0 = 1.025 \text{ MPa}$, $M = 1.74$, and $B' = 4.39 \times 10^{-5} \text{ MPa}^{-M} \text{ s}^{-1}$. It is important to note that in our approach, the determination of threshold stress, σ_0 , does not require the assumption of a specific stress exponent (1, 2, or 3) depending on the creep mechanism or the utilization of any extrapolation method as used by some investigators [31], rather the actual data are sufficient to provide this information.

Now, to determine the constants for grain core creep ($\dot{\epsilon}_C = K\sigma^N$), a commonly used procedure is utilized. Usually N is obtained from the slope in Region III (high $\dot{\epsilon}$ region) in the $\log \sigma - \log \dot{\epsilon}$ plot ($N = d \log \dot{\epsilon}_C / d \log \sigma$). Careful examination of data shown in Fig. 4 and 5 reveals that the $\log \sigma$ vs. $\log \dot{\epsilon}$ data in this region is affected by grain size and

temperature. In this high $\dot{\epsilon}$ region, stress is affected by grain size but the slope of $\log \sigma$ vs. $\log \dot{\epsilon}$ plot does not change with temperature and grain size. Since many data points are available in the lower test temperature and the larger grain size cases, the stress exponent, N , is determined from the data for these conditions. Based on the measurements of the slope in this region (the measured values are shown in Table 2), the average value of N is found to be 4.55. Since N for dislocation creep is generally believed to be unaffected by temperature and grain size, this value for the low temperature and coarse grain conditions is also used for high temperature and fine grain conditions. Using $N = 4.55$, different trial values of K are now used to compute the stress values within the intermediate $\dot{\epsilon}$ regime, where both mantle and core creep mechanisms contribute concurrently. The appropriate value of K is obtained when the overall computed curve best matches the experimental data simultaneously in the intermediate and high strain-rate regions. This is shown in Fig. A2(b), in which the value of K is found to be $5.05 \times 10^{-9} \text{ MPa}^{-N} \text{ s}^{-1}$. By applying the above analytical methods to the experimental results for all test temperatures and grain sizes, the constitutive parameters (B' , σ_o , M , K , and N) were determined over the wide range of temperatures and strain-rates and are given in Table 2.

Appendix 3. Determination of Activation Energy for Grain Mantle Creep

Generally activation energy is obtained from the slope of a logarithmic plot of $\dot{\epsilon}$ versus $(1/T)$ at the fixed values of σ and d . However, this method cannot be applied to the data for grain mantle creep ($\dot{\epsilon}_M = B'(\sigma - \sigma_o)^M$) because the driving stress for grain mantle creep is not the applied stress, σ , but is influenced by the inherent glide resistance of the threshold stress, σ_o . In addition, the stress exponent, M , is also dependent on temperature (see Fig. 9(b)). Recognizing these additional temperature dependencies, we select two separate methods to examine the relevant mechanism: method I is based on the stress function, $f(\sigma) = (\sigma - \sigma_o)$, and method II is based on the stress function, $f(\sigma) = (\sigma - \sigma_o)^M$. The latter incorporates all of the thermally responsive features except for grain size. Activation energy (Q) for grain mantle creep is determined from the slope ($= -Q / 2.303 R$) of a plot of logarithmic $\dot{\epsilon}_M$ versus $(1/T)$ at the fixed values of $f(\sigma)$ and d as expressed by

$$Q = -R \left[\frac{d \ln \dot{\epsilon}_M}{d(1/T)} \right]_{d, f(\sigma)} \quad (\text{A3.1})$$

Method I : Fig. A3(a) shows the relationship between $\log(\sigma - \sigma_o)$ and $\log \dot{\epsilon}_M$ for a grain size of $8.07 \mu\text{m}$ in which the slope ($= d \log \dot{\epsilon}_M / d \log(\sigma - \sigma_o)$) is M (see Table 2), and $\dot{\epsilon}_M$ versus $(1/T)$ curves at fixed values of $(\sigma - \sigma_o) = 0.3$ and 1 MPa , in which Q is found to be

183 and 172 kJ/mol, respectively. This variation is a direct result of how M is affected by temperature.

Method II : Fig. A3(b) shows the relationship between $(\sigma - \sigma_o)^M$ and $\dot{\epsilon}_M$ for a grain size of $8.07\mu\text{m}$ in which the slope $(= d \log \dot{\epsilon}_M / d \log (\sigma - \sigma_o)^M)$ is 1, and $\dot{\epsilon}_M$ versus $(1/T)$ curves at a fixed value of $(\sigma - \sigma_o)^M = 1 \text{ MPa}^M$ for two different grain sizes. The average value of Q is 166(kJ/mol) when three different grain sizes are considered (see Table 4(a)) and is independent of the level of $(\sigma - \sigma_o)^M$.

Appendix 4. Determination of Grain Size Exponent for Grain Mantle Creep

Generally grain size exponent is obtained from the slope in $\log \dot{\epsilon}$ vs. $\log d$ plot at the fixed values of σ and T . However, this method again cannot be applied to the data for grain mantle creep ($\dot{\epsilon}_M = B'(\sigma - \sigma_o)^M$) because σ_o and M are dependent on grain size (see Table 2). Thus, grain size exponent (μ) for grain mantle creep is obtained with the following equation based on the stress function of $f(\sigma) = (\sigma - \sigma_o)^M$ at the fixed values of $f(\sigma)$ and T .

$$\mu = - \left[\frac{d \log \dot{\epsilon}_M}{d \log d} \right]_{T, f(\sigma)} \quad (\text{A4.1})$$

Fig. A4(a) shows the relationships between $(\sigma - \sigma_o)^M$ and $\dot{\epsilon}_M$ for different grain sizes at a temperature of 550°C , in which the slope $(= d \log \dot{\epsilon}_M / d \log (\sigma - \sigma_o)^M)$ is 1. $\dot{\epsilon}_M$ versus d curves are shown in Fig. A4(b) at a fixed value of $(\sigma - \sigma_o)^M = 1 \text{ MPa}^M$ for two different temperatures. The average grain size exponent (μ) is around 2.3 (see Table 4(b)) and its value is independent of the level of $(\sigma - \sigma_o)^M$.

Fig. A1

Fig. A2

Fig. A3

Fig. A4

CAVITY FORMATION AND EARLY GROWTH IN A SUPERPLASTIC Al - Mg ALLOY

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ABSTRACT

The knowledge of the exact physical mechanism of cavity formation and early growth is important for the prediction of the extent of internal damage following superplastic deformation. To this end, the early stages of cavitation in a superplastic Al-Mg-Mn-Cu alloy have been experimentally studied and reported here. Small cavities ($< 0.5\mu\text{m}$) were detected by scanning electron microscopy and the number of cavities per unit volume was monitored by image analysis through optical microscopy. Before deformation, some cavities were seen at the particle-matrix interfaces. However, during tensile deformation in the temperature range of 450°C to 550°C (and strain-rates $\sim 10^{-4}\text{s}^{-1}$ - 10^{-2}s^{-1}), additional cavities emerge and grow. Most cavities are observed at the interface between particles and the matrix from submicron size range, and grow initially along the interface. This suggests that early cavity growth is by matrix/particle decohesion, possibly starting from interfacial defects, and this growth has rapid kinetics. The density of *observable* cavities increases with strain, i.e. "nucleation" is continuous. The number of cavities increases at higher strain-rates and at lower test temperatures. This is due to the higher flow stresses, reduced strain-rate sensitivity and poorer diffusional accommodation process, which assist in the initial growth of the submicron and nanoscale interface defects. But the evidence for diffusional cavity growth in the initial stages was not found.

1 INTRODUCTION

Superplastic deformation leads to large "neck-free" elongation, but most superplastic materials develop internal cavitation during deformation [1-4]. It has been recognized that for such materials, cavitation often precedes failure and excessive cavitation may impose significant limitations on the industrial use of superplastically formed components [1]. During complex part fabrication, strain, strain-rate, stress-state, and temperature can vary widely in different regions of the workpiece. This causes a variable degree of cavitation from one location to another. The level of cavitation is related to the cavity population density and their growth kinetics [3, 5]. To assess the extent of cavitation damage in formed parts, a method for accurate prediction of cavitation is required. The exact mechanisms of cavity formation and early growth have not, however, been precisely identified via supporting microscopy evidences. The population density of cavities, often found to increase with strain [6-8], is largely ignored in the literature, but it does play a critical role under certain conditions. In this study attention has been focused on obtaining reliable evidence regarding the early stage of cavitation in a superplastically deformed Al - Mg alloy.

Various models for the initiation of cavities at grain boundaries have been suggested. To review briefly, for example, cavities are thought to be nucleated by the continuous condensation of vacancies on grain boundaries which experience a normal tensile stress [9] or by vacancy clustering due to the stress concentration on grain boundary inclusions produced by strain incompatibility and grain boundary sliding [10]. The ultimate process of interfacial separation may be by the rupturing atomic bonds due to stress concentrations produced at the head of a dislocation pile-up near the interface of grain boundary obstacles [11, 12], or a combination of the above processes [13]. In the models of Hull and Rimmer [14] as well as Raj and Ashby [10], a stable cavity nucleus is assumed based on thermodynamic stability criterion: $r_c \geq 2\gamma/\sigma$, where r_c is the critical cavity radius, γ is the surface energy, and σ is the applied stress. The concept of critical

radius based on the above equation suffers from a basic difficulty in that cavities, if nucleated in infinitesimal sizes or nanometer sizes (as experimentally seen), require infinitely large stresses to be stable. This is not very satisfactory.

Existing models of cavity nucleation are actually models of growth from small sizes. These models were developed for high temperature creep of coarse grain materials under very slow creep rates and exhibiting low fracture strains. In superplastic metals the grain size is much finer, the strain rates are much higher and the microstructure of cavitation is very different. In many of the creep cavitation papers, a regular distribution of voids is assumed on a single grain boundary normal to the maximum tensile direction. However, in a fine-grained superplastic alloy considered in this study, cavities are seldom found as a regular array on a single grain boundary. In aluminum alloys, for example, cavities are associated with certain non-deformable particles (e.g. Al_6Mn in Al-Mg-Mn alloy), whose spacing is of the order of 1-2 grains or more, although voids are found in one in every 5 grains or more. The phenomenology of superplastic cavitation appears to be much different from that of creep cavitation.

Excluding a few observable pre-existing voids, general cavitation in superplastic metals occurs after an appreciable strain (i.e. ~ 0.2). This emergence of cavities (or nucleation) seems to be related to debonding along certain particle-matrix interfaces. Nucleation by debonding is strain controlled. For materials containing particles or fibers, Needleman [15] and Christman *et al.* [16] calculated the localized hydrostatic stress and effective strain near the surface of a particle when the plastic strain in the matrix was constrained by the non-deformable particle. As plastic deformation continues, a rapid build-up of hydrostatic stresses is predicted and plastic flow spreads rapidly from the corner of the particle, where decohesion begins.

Systematic studies on the nucleation of cavities by progressive straining of superplastic materials are lacking. Even though the population density of *observable* cavities increases with increasing strain (referred to as continuous “nucleation”) [6-8], it

is difficult to ascertain whether they truly nucleated from zero size, or they pre-existed as interface defects of submicron or nanoscale sizes before growing to detectable sizes. For most quasi-single phase superplastic alloys, a combination of warm or cold work and heat treatment is employed to develop recrystallized fine grain sizes of the order of 10 μ m or less [17]. Debonding between particle and matrix can occur during heavy cold rolling to produce sheet. Thus, rolling practice leaves defects (weakly bonded interfaces or broken particles) in the material, and they do not generally self-sinter fully even after an exposure at high homologous temperatures. Prior attempts to heal pre-existing damages before superplastic testing have been documented in Refs. 18 - 20. For example, a fine-grained 7475 Al alloy, hot isostatic pressed before testing, showed improved uniaxial tensile elongation to failure [19]; from 800% tensile elongation to failure for no prior treatment to a failure elongation of ~1400% ($\dot{\epsilon} = 2 \times 10^{-4} \text{s}^{-1}$ and $T = 516^\circ\text{C}$) after treatment for 8hrs at 516 $^\circ\text{C}$. This case also exhibited significantly lower cavitation. For the alloy considered in this study, annealing at 550 $^\circ\text{C}$ for 3hrs seems to heal particle-matrix interface damage as evidenced by a drastic reduction in the number of voids generated during superplastic deformation (see Appendix 1). Defect-free interfaces may be obtained by long annealing times, but the possibility of loss of certain alloying elements due to evaporation and/or grain coarsening are practical problems.

It is thus highly likely that under normal alloy processing conditions, a partially healed interface in a test material would have nanometer size defects (< 100nm in size). According to the criterion for a thermodynamically stable cavity nucleus mentioned before, the applied stress necessary to enlarge such a void should be about 40MPa. However, voids actually open up along the interface at deformation stresses much lower than this stress and do so progressively with strain. This observation raises serious doubt about how the initial void size assumed in the existing creep cavitation model (e.g. diffusional growth process) became so large at the outset (see Appendix 2 for details). Recent studies with particle-containing alloys have suggested an improved approach for

the understanding of the early stage of cavity growth based on an interface-constrained growth model, according to which much smaller defects are shown to debond rapidly via local plasticity [8]. Much of the detailed observations reported in this study provides the basis for such a model. However, cavitation is influenced not only by external factors, such as strain, strain-rate, temperature, and stress-state, but also by microstructural features, such as grain size, the nature of interfacial defects, and the energetics of the interfaces. The complex interaction of all these parameters makes it difficult to clearly distinguish the dependence of cavitation on each parameter [3, 4]. Detailed examination of small cavities formed during superplastic deformation by using Scanning Electron Microscopy (SEM) have, however, revealed some key features of early cavity growth and the variation of cavity population density as functions of strain, strain-rate, and temperature.

2 MATERIAL AND EXPERIMENTAL

The material used in this study was an aluminum alloy containing 4.7%Mg, 0.8%Mn, and 0.4%Cu by weight. The alloy was received in the DC cast condition from Kaiser Al Company. It was homogenized at 500°C for 12 hours in an air-circulating furnace and subsequently hot forged (from 30mm thickness to 13mm thickness at 250°C). To obtain a fine-recrystallized microstructure prior to testing, it was cold rolled to 1.7mm final thickness (cold reduction $\approx 87\%$), and then annealed at 500°C for 0.5 hour in a salt bath. The resulting grain structure showed linear intercept grain sizes of 9.68, 9.37, 5.40 (μm) in the longitudinal (L), long transverse (T) and short transverse (S) directions, respectively (average grain size = $\sqrt[3]{9.68 \times 9.37 \times 5.4} = 7.88\mu\text{m}$).

Uniaxial tensile tests were carried out on dog-bone specimens of this material (specimen gauge length = 12.7mm) under a constant strain-rate condition. The strain-rate range for the tests was 10^{-4}s^{-1} - 10^{-2}s^{-1} and the temperature range was 450°C - 550°C. Due

to flow of soft superplastic material from the grip region into the gauge region during the elevated temperature test, maintaining a constant strain-rate in the gauge region is complicated [21]. To correct for this flow problem, an improved cross-head speed schedule for a constant strain-rate [21, 22] was used. Cross-head speed was controlled by computer through a digital interface board on an Instron machine. The tests were performed by placing the specimen and load train within a clamshell furnace having three heating zones independently controlled to maintain temperature within $\pm 1^\circ\text{C}$ of the test temperature over a 130mm length. Interruptions were made to these tests to produce samples with a variety of strain levels in the range of 0.15 to 1.2. To preserve the microstructure of these deformed samples, test specimens were water quenched immediately after the pre-determined strain level was reached. Specimen strain level was checked by actual area measurements from the samples: $\epsilon = \ln(A_0/A)$ where A_0 was the initial cross-section area and A was the current cross-section area. The deviation from desired strain level was within $\pm 5\%$ at large strains.

All tested specimens were sectioned along three orthogonal planes: L-S, L-T, and T-S planes, taken from the uniform part of the gauge region. The sections were mechanically polished. Preparation of a metallographically polished surface in particle-containing alloys without creating additional damage is difficult. Debonded and fragmented particles tend to fall off during mechanical polishing. To prevent alteration of the appearance of cavities which could occur due to rounding of cavity edges by the polishing pressure, an OP-Chem cloth supplied by Struers Company was used for polishing, along with a colloidal silica suspension ($< 0.04\mu\text{m}$). That this method was reasonably effective was supported by the fact that the decrease in the population density of initial voids after a high temperature (550°C) diffusion annealing treatment could be detected by this special polishing technique [23].

To observe dispersoid particles and voids under the microscope, sections were taken 0.5mm apart (3 times) for each of three different planes. These sections were then

examined under optical microscope and SEM. From SEM micrographs, the particle-matrix interfaces and small cavities ($< 0.5\mu\text{m}$) were investigated. From digitized images taken with a CCD camera (640×480 pixels) through an optical microscope, measurements of cavity number and size were made; two-dimensional metallographic measurements of cavities on the L-S, L-T, and T-S planes were made in the computer using NIH-Image software. The dimension of each image was $273.5 \times 205.13\mu\text{m}$, with a resolution of $0.427\mu\text{m}$ per pixel. Fig. 1 is an example of a digitized image, in which the optical gray level of pixels varies from 0 to 255. The optical gray level of the background (uncavitated region) was in the range of 35 - 50 and that of particles was in the range of 65 - 90 and that of cavities shown as black area was higher than 180. By comparing this digital image to the original image via an optical microscopy, the threshold optical gray level that best represented the cavities was determined to be the optical gray level which was 20 higher than the maximum gray level in the particles as shown in Fig. 1(c).

3 RESULTS AND DISCUSSION

3.1 Particle Size and Distribution

Optical and SEM photographs of the Al alloy, after static annealing at 500°C for 0.5 hour, are shown in Fig. 2(a) and (b), respectively. Particles are seen at several locations and exhibit a distribution in size. Pre-existing voids are also seen in these photographs. A detailed view of the particles also can be seen from a SEM photograph shown in Fig. 3. The larger particles ($0.5 \sim 2.5\mu\text{m}$ in radius) located near the grain boundaries are intermetallic dispersoids such as Al_6Mn containing Cu, and the smaller particles ($< 0.1\mu\text{m}$ in radius) seen in Fig. 2(b), randomly distributed throughout the grain and grain boundary regions, are Mg-Al-Cu aging precipitates (β -phase) [24]. These latter precipitates which are found in samples cooled after test, are in solid solution at the test

temperatures ($> 450^{\circ}\text{C}$) and not expected to generate cavities during superplastic deformation.

To determine the population density of particles, polished microstructures prepared for optical microscopy were slightly etched with Graf Sergent etchant (15ml HNO_3 , 0.5ml HF , 3g CrO_3 , and 84ml water). Because of the small difference in optical contrast between particle and matrix in the unetched condition (Fig. 1), the light etching treatment was useful. Micrographic image analysis method was used to quantify data on the number and size of particles and voids, and the area-based measurement was converted to a 3-dimensional representation using the Schwartz-Saltykov method [24]. The size distribution of second phase particles determined this way is shown in Fig. 2(c) for the static annealing condition, in which only particles larger than $0.5\mu\text{m}$ radius were counted. Based on the fit in Fig. 2(c), the size distribution of particles can be approximately given by [6]

$$n(r) = n_0 e^{-ar} \quad (1)$$

where $n(r)$ is the number of particles of a certain radius, r , per unit volume, and n_0 and a are constants. The values of n_0 and a in Fig. 2(c) are found to be $4.26 \times 10^7/\text{mm}^3$ and 3.23, respectively. It should be noted that for large particles ($0.5 \sim 2.5\mu\text{m}$ in radius), which may be considered to be potential sites for the formation of cavities, the spacing is typically of the order of 1-2 grains.

3.2 Cavity Formation Site

Although some pre-existing cavities are present in the test alloy, cavities are continuously formed during deformation [6-8]. To identify the cavity formation sites, deformed specimens with strain levels of 0.45 and lower were carefully examined using SEM. Fig. 3 shows the unetched micrograph of a sample tested at $T = 550^{\circ}\text{C}$ and $\dot{\epsilon} = 10^{-4}\text{s}^{-1}$. Although a few cavities $> 10\mu\text{m}$ diameter have been observed in specimens at this strain level [5], this figure shows only small cavities ($< 0.5\mu\text{m}$) situated on particles

located at grain boundaries. Many similar micrographs were examined from different deformation conditions, and in the overwhelming number of cases at the lower strain levels, cavities are found to be associated with grain boundary particles. Thus, the detailed study reveals that cavities are formed at the particle-matrix interfaces, which are situated on grain boundaries, and that the cavities reach observable size due to deformation.

Did the voids grow from pre-existing defects or did they truly nucleate? While both possibilities exist, it does not seem feasible to nucleate a very small (nanometer scale) void at high temperature by overcoming of surface tension in a superplastic alloy in which diffusional accommodation is appreciable. This was also discussed in the Introduction of this paper. A preferred viewpoint is that pre-existing defects or weakly bonded interfaces exist in conventionally processed alloys. As explained before, these interface defects are generated during heavy cold rolling (thickness reduction $\approx 87\%$). These defects do not completely sinter even after long exposure to high homologous temperatures [23], although existing sintering theory estimates that any pre-existing microvoids smaller than $0.5\mu\text{m}$ radius should have been eliminated* [26]. The failure to meet an expected sintering criterion may be due to unusual surface tension conditions, non-equilibrium void shape, etc. that do not match theoretical condition. Partial healing of defects at the particle-matrix interfaces by diffusion anneal does tend to minimize the size of voids and improve bond strength, but complete healing of the particle-matrix interfaces by diffusion anneal is not likely. The existence of small voids $< 0.5\mu\text{m}$ in the

* The stability of microvoids in the absence of external stress may be estimated as the time t , required to remove a void of radius r by sintering [26]: $t = (\Phi k T r^4 / \Omega \gamma \delta D_{gb})$, where Φ is a constant having a value of ~ 0.6 , γ is the surface energy, Ω the atomic volume, D_{gb} the grain boundary diffusion coefficient, δ the grain boundary width, k Boltzmann's constant and T the absolute temperature. Assuming a pre-existing cavity of $r = 0.5\mu\text{m}$ undetected under an optical microscope and $\gamma \approx 1.1\text{Jm}^{-2}$ in an aluminum alloy [25], the calculated time to eliminate the cavity by sintering is 7.8, 2.4, and 1.6min at the temperatures of 450, 500, and 550°C, respectively. Considering the 0.5hr time allowed at 500°C in recrystallization step and an additional holding time ($\sim 10\text{min.}$) at the testing temperature to stabilize the isothermal test condition, this theory estimates that any pre-existing microvoids smaller than $0.5\mu\text{m}$ would have been eliminated.

alloy (before tensile test) verified by numerous SEM investigations bears testament to such a conclusion.

3.3 Initial Cavity Growth

Small cavities ($< 0.5\mu\text{m}$) in specimens deformed to a strain level of 0.45 were examined under higher magnifications using the SEM. The results are shown in Fig. 4. Sub-micron size cavities are found at the particle-matrix interfaces and they are generally elongated along the interfaces. The crack-like cavity shape suggests that early cavity growth is by matrix/particle interface decohesion.

When a particle is well-bonded to matrix, high levels of hydrostatic tension stresses develop near the interface under stress, leading to lower rates of local strain [26]. Under elastic conditions, the level of peak hydrostatic stress within the matrix may be as high as 6 times the matrix flow stress [16]. However, when the interface contains defect (debonded region), stresses become deviatoric at the free surface of the defect, i.e. dislocation slip impingement can occur to relieve the local displacement incompatibility. Initially this condition of plasticity is constrained by the particle interface, i.e. decohesion is by a tearing process. Grain boundary sliding in the adjacent grains can assist in the interface debonding process, as schematically shown in Fig. 5(a). Fig. 5 describes two possible scenarios, in which a pre-existing defect (an embryonic cavity) located at (a) the particle-grain boundary interface or at (b) the particle-matrix interface on sliding grain boundaries is subjected to normal and shear stresses during deformation. The effects of combined normal and shear stress near the defect create strain concentration at the periphery of the cavity that is dominated by lateral (circumferential) tensile strain. As shown in Ref. 8, the magnitude of the local tensile strain increases with particle size because the constrained zone of plasticity at the interface becomes larger. Growth of an interface defect by plasticity is a preferred concept for cavity formation because (i) it is

energetically more favorable than breaking atomic bonds under high hydrostatic tension, and (ii) debonding occurs gradually with increasing strain, while the development of hydrostatic tension is expected to affect all particle interfaces.

Growth of cavities after the start of decohesion process was studied next. Cavities in the range of $2 \sim 5\mu\text{m}$ in diameter were examined in detail using the SEM, and the results are shown in Fig. 6. A small cavity that is partially (Fig. 6(a)) or completely (Fig. 6(b)) detached from the particle is shown. A cavity grown to be larger than the particle is observed in Fig. 6(d), in which an apparently detached particle is still found inside. In most situations, particles, which create voids larger than the size of particles with debonded interfaces, fall off during specimen polishing. The fact that partially debonded interfaces are seen at lower strains, but completely debonded interfaces are seen at large strains indicates that debonding process is strain assisted. The growth of a pre-existing void created between broken particle fragments can also be seen in Fig. 6(c). On the other hand, the size and shape of cavities at the same level of strain are found to be different as seen in Fig. 4 and 6. This suggests that all "nucleated" cavities are not present at the same time or do not have the same size. The embryonic cavities may have a large distribution of sizes and different cavity growth kinetics depending on the particle size and strain-rate sensitivity [8]. Based on these phenomenological observations, it is likely that decohesion at the particle-matrix interface progressively evolves with strain, and after debonding is complete, the size of the cavity suddenly rises to the size of the particle ($1 \sim 4\mu\text{m}$). After this, cavities may grow with continued plastic deformation of the matrix, in which interface constraint is removed. Statistically, an opportunity for the retention of the debonded particle in the cavity is extremely low to permit its metallographic observation. This fact may lead to erroneous interpretations of cavitation if careful and progressive analysis at small strains is not carried out.

Interfacial defect evolution due to constrained plasticity growth has rapid but variable kinetics [8]. While diffusion-based creep cavitation models also predict the rapid

cavity growth at the early stage, the choice of the stable cavity size dominates the predicted cavitation level for diffusional growth. As pointed out, the selected stable size does not agree with observation [23]. Furthermore, at higher test temperature, cavitation tendency is reduced* rather than increased as diffusional growth concept might suggest. Void growth due to diffusion along the grain boundaries is still predicted to be too low for the fine-grained superplastic materials due to the low flow stress.

3.4 Population Density and Nucleation of Cavities

Since the number of *observable* cavities increases continually with increasing strain, the cavity population density has been measured in detail as a function strain, strain-rate, and temperature. Digital images in the L-S, L-T, and T-S planes based on optical metallography were analyzed for all tested specimens by counting the number of cavities. Even though the minimum diameter detectable by this method was $0.44\mu\text{m}$, only cavities larger than $0.5\mu\text{m}$ were counted to reduce measurement errors. Fig. 7 shows the total number of cavities (density = number per unit volume) for three different strain-rates as a function of strain at the three different temperatures: (a) 450°C , (b) 500°C , and (c) 550°C . The cavity population density at zero strain was obtained after a thermal exposure for 10min at the test temperature to simulate the microstructural state immediately before deformation. At 550°C , this level is the lowest among the three temperatures resulting from a higher annealing temperature and healing of pre-existing defects [23].

While some pre-existing cavities are present, newly emerging cavities begin to appear after some initial strain (i.e. $\epsilon \sim 0.2$). Above this strain, the number of cavities steadily increases with strain for all test conditions. As mentioned in Section 3.3, this

* Reduced cavitation at higher test temperatures is due to higher diffusional rates providing accommodation. Opposing observation, if any, may simply be due to tests carried out with poorer control of strain-rate during tension test, or with very different material processing conditions.

initial strain stems from achieving decohesion at the particle-matrix interface in the early stage. On the other hand, the number of emerging cavities depends on the size of the constrained zone of plastic deformation near the particle interface, and is a function of particle size [8]. Generally, larger particles and larger interface defects induce more rapid decohesion with increasing strain, while smaller particles have a smaller constrained zone and require more strain to produce complete debonding. Because the population of small particles in the alloy (Eq. (1)) is substantial, continuous nucleation of small cavities can continue till very large strain.

Second, the frequency of cavity formation events increases with increasing strain-rate. This strain-rate effect on continuous nucleation is directly related to a higher strain-rate for constrained plasticity near the particle interface, which is believed to be related to higher deformation stresses in the matrix. This condition also reduces the strain-rate sensitivity and diffusional accommodation process at the particle-matrix interfaces, thereby assisting interface debonding.

The linear increase of cavity population density with strain observed for $\epsilon > 0.2$ in Fig. 7 can be expressed by

$$\frac{dN_c}{d\epsilon} = \chi \quad (2)$$

where N_c is the total population density of cavities and χ is a constant. This cavity nucleation rate, χ , is plotted as a function of applied strain-rate in Fig. 8. This figure shows that χ generally increases with strain-rate, but it does show a higher value at an intermediate temperature of 500°C (at intermediate and high strain-rates). At the high test temperature due to the lower flow stress and greater diffusional accommodation near grain boundaries, it may be expected that χ would be lower, but it appears unclear as to why χ would peak at an intermediate temperature. A possible reason may be related to shear or mantle strain near the grain boundaries. Compared to lower test temperatures where grain boundary sliding effects are less, more strain (shear) occurs near the grain

boundaries at higher test temperatures, thereby nucleating more voids. This damaging tendency could be alleviated at yet higher temperatures where diffusional accommodation becomes appreciable. This hypothesis may be expressed as follows. At elevated temperature, deformation is inhomogeneously distributed through the solid and macroscopic strain is a result of multiple mechanisms [28]. The relative contributions of the mechanisms vary from a larger fraction of grain core creep ($\dot{\epsilon}_C$) at low temperature to a larger fraction of grain mantle creep ($\dot{\epsilon}_M$) at high temperature. The grain mantle deformation has been considered as a localized dislocation climb process operating near the grain mantle region and is believed to be responsible for grain boundary sliding displacements and grain rotation. Its contribution to total deformation has been shown to be significant and dependent on the microstructural and deformation condition [22, 28]. Since particles located at grain boundaries lead to cavitation, the responsible local strain-rate for this process is believed as $\dot{\epsilon}_M$ rather than $\dot{\epsilon}_{total}$ (total strain-rate).

To develop a more quantitative understanding of the above thesis, the contribution of grain mantle deformation, expressed by the ratio $\beta = \dot{\epsilon}_M / \dot{\epsilon}_{total}$, can be approximately calculated by mechanically separating $\dot{\epsilon}_M$ from $\dot{\epsilon}_{total}$ (for the detailed procedure, see Ref. 28). The contribution of grain mantle deformation is a function of concurrent grain growth [28] and dynamic recrystallization, and can vary during the test. Fig. 9(a) shows the value of β as a function of test temperature and strain-rate. The bar on each test point indicates the range of β for $0 \leq \epsilon \leq 0.85$. This ratio increases with decreasing strain-rate and increasing temperature. When $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$ and $T = 550^\circ\text{C}$, grain mantle creep accounts for nearly the entire total strain. This ratio is again plotted in Fig. 9(b) as a function of flow stress, σ . The range of stress shown corresponds to the above strain range. Experimentally the relationship between β and σ may be empirically described by $\beta = M_o - M\sigma$ (valid for $\sigma \leq (M_o / M)$), where $M_o = 1.02$ and $M = 2.12 \times 10^{-2} [\text{MPa}^{-1}]$ for this alloy. This upper limit of σ is 48 MPa, above which mantle creep contribution is

expected to be non-existent. Below $\sigma = 1$ MPa, β value is not appropriately computed possibly due to the effect of threshold stress, σ_o , a glide-based resistance necessary to initiate the grain mantle deformation [22, 28].

To apply this analysis to the continuous "nucleation" phenomenon, the nucleation rate, χ , is first normalized by β , and then plotted in Fig. 9(c) as a function of effective stress, $\sigma - \sigma_o$. The value of χ / β increases with increasing effective stress, and all data fall within a narrow band. Plotted in this manner, no separate effects of temperature and strain-rate are noticeable. Based on this plot, χ may be roughly expressed by:

$$\chi = c \beta (\sigma - \sigma_o)^H \quad (3)$$

where c is a constant, and H is ~ 1.85 (see Table 1). Thus, cavity nucleation rate may be empirically predicted in terms of applied stress and β over a wide range of strain-rate and temperature. Since β decreases and σ increases with decreasing temperature, a peak in the value of χ is expected at an intermediate temperature (Fig. 8).

4 SUMMARY AND CONCLUSIONS

Early stages of cavitation in a superplastic Al-Mg alloy have been examined by scanning electron microscopy. Starting from very few initial cavities, cavities within a wide size range have been observed to emerge with increasing strain, including those $< 0.5 \mu m$ that nucleate at grain boundary particles. Cavity population density (i.e. the number of cavities/volume) has also been monitored by image analysis method on unetched specimens. In this study, the results of uniaxial deformation over a temperature range of $450^\circ C$ to $550^\circ C$ and a strain-rate range of $10^{-4} s^{-1}$ to $10^{-2} s^{-1}$ have been presented. New cavities are continuously formed at the particle-matrix interface located near grain boundaries during deformation, and their rapid early growth is constrained along the interface. This crack-like decohesion is identified as one due to slip impingement near the

tips of a pre-existing damage on the particle interface. The specific observations made in this work are listed as follows:

1. The observed correlation between the presence of hard second phase particles and cavitation evinces that cavities are formed at the particle-matrix interfaces located at grain boundaries.

2. Due to the heavy cold rolling prior to superplastic deformation, some weakly bonded particle-matrix interfaces and broken particles pre-exist. Annealing treatment can partly heal these defects, but some submicroscopic defects do remain at the interface.

3. Small pre-existing defects on the particle-matrix interface can initiate crack-like growth in the early stage of superplastic deformation. This decohesion progressively evolves with strain, leading to complete debonding of the cavity from the particle. The kinetics of evolution of such interfacial defects is generally rapid but variable depending on particle size and type.

4. While some pre-existing cavities are present, some initial "incubation" strain is required to permit new cavities to begin to emerge. This initial strain is a function of particle size and type. Generally, larger particles and larger interface defects induce more rapid decohesion with increasing strain, while smaller particles have a smaller constrained plasticity zone and require more overall strain to produce complete debonding [8]. This causes cavity nucleation to be continuous.

5. The frequency of cavity formation events increases with increasing strain-rate. However, the dependence of this nucleation rate, χ , on temperature is not monotonic; it reaches a peak at an intermediate test temperature, as it is affected by both stress and mantle-to-core strain ratio. This combined effect is expressed by $\chi = c \beta (\sigma - \sigma_o)^H$, where c is a constant, H is ~ 1.85 , β is the ratio of the grain mantle strain-rate to the applied strain-rate, and σ_o is the threshold stress.

5 ACKNOWLEDGEMENTS

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Table 1. Experimentally determined constants for cavity nucleation rate in Eq. (3)

Temp. (°C)	c [mm ³ MPa ^{-H}]	H
450	3.25×10^3	1.88
500	6.97×10^3	1.90
550	7.64×10^3	1.81

(a)

Optical Micrograph

Gray Level

Background = 35 - 50

Particles = 65 - 90

(b)

Threshold Level = 90

(c)

Threshold Level = 110

Fig. 1. The determination of threshold optical gray level to distinguish cavities in the digitized image shown in (a). The optical gray level of pixels in the digitized image varies from 0 to 255. The level of the background (uncavitated region) is in the range of 35 - 50 and that of particles is in the range of 65 - 90. The threshold gray level that best represents the cavities is determined to be the level that is 20 higher than the maximum gray level in the particles as shown in (c).

Fig. 3. Small size cavities in a tested sample of Al-Mg-Mn-Cu alloy observed using SEM on the L-S plane. The test condition was $T = 550^{\circ}\text{C}$, $\dot{\epsilon} = 1 \times 10^{-4} \text{s}^{-1}$, and $\epsilon = 0.44$. All small cavities marked by arrow are observed on particles located at grain boundaries.

Fig. 4. SEM pictures showing decohesion between particle and matrix in an Al-Mg -Mn- Cu alloy at the condition of (a) 500°C, 10^{-3}s^{-1} , $\epsilon = 0.42$, (b) 550°C, 10^{-2}s^{-1} , $\epsilon = 0.42$, (c) 550°C, 10^{-4}s^{-1} , $\epsilon = 0.44$ and (d) 550°C, 10^{-3}s^{-1} , $\epsilon = 0.44$ in the L - S plane.

Fig. 5. Schematic drawings of interface-constrained cavity growth at a pre-existing defect (an embryonic cavity) located at (a) the particle-grain boundary interface or at (b) the particle-matrix interface on sliding grain boundaries subjected to normal (σ_n) and shear (σ_s) stresses during deformation. Tensile strain normal to the interface at the free surface of a void leads to an outward movement of the surrounding matrix material as well as rapid spreading along this interface.

Fig. 6. SEM pictures of (a) partially and (b) fully detached cavities from particles and (c) a cavity located between particles and (d) a cavity grown after decohesion in an Al-Mg-Mn-Cu alloy.

Fig. 7. Cavity population (number/mm³) for three different strain-rates as a function of strain at the temperature of (a) 450°C, (b) 500°C and (c) 550°C in the superplastic Al-Mg-Mn-Cu alloy deformed under uniaxial tension. The cavity density at zero strain is obtained from the specimen just before the start of deformation at the corresponding temperature.

Fig. 8. The rate of increase of cavity density with strain, $\chi = dN_c / d\epsilon$. As the strain-rate increases, the χ - value increases. The maximum value of χ is found at the intermediate temperature of 500°C at $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$ and $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$.

Fig. 9. (a) The ratio of mantle strain-rate to the applied strain-rate, $\beta = \dot{\epsilon}_M / \dot{\epsilon}_{total}$, determined by using the method described in Appendix 3. This ratio is plotted as a function of average stress (σ) in (b). β is empirically described by $\beta = M_0 - M\sigma$ where M_0 and M are found to be 1.02 and $2.12 \times 10^{-2} [\text{MPa}^{-1}]$, respectively. (c) Cavity nucleation rate (χ) normalized by β is plotted as a function of effective stress $(\sigma - \sigma_0)$. The increasing trend is generally unaffected by test temperature and strain-rate.

Appendix 1. Healing of damage at the particle-matrix interface

Thermomechanical processing to produce fine-grained microstructure in an Al-Mg-Mn-Cu alloy requires heavy cold rolling reduction (e.g. from a thickness of 13mm down to 1.7mm - reduction = 87%), followed by recrystallization heat treatment. Coarse intermetallic particles in the alloy are fractured during rolling and some interfacial voids are created, which do not fully close up during heat treatment. Optical micrograph of as-rolled sample is shown in Fig. A1.1(a), in which small pre-existing voids are observed at several locations. However, after annealing at 550°C for 3hrs, almost no voids are observed in the micrograph (Fig. A1.1(b)). To examine this effect of annealing on the healing of pre-existing microvoids, cold-rolled samples were thermally soaked under several annealing conditions. The size distribution of initial voids ($> 0.5\mu\text{m}$ in diameter) detected by optical microscope is shown in Fig. A1.1(c). The initial void density is found to decrease with increasing annealing temperature and time. Fig. A1.1(d) shows the initial void density as a function of annealing time at two different temperatures. Void density decreases rapidly in a few minutes, and slowly afterward depending on the annealing temperature. These results indirectly confirm that depending on the annealing temperature and time, pre-existing voids and/or weak interfaces between particle and matrix are always present, although some of them can be reasonably healed (at least partially) by annealing under conventional alloy processing conditions.

To examine the influence of healing on the degree of cavitation, cold-rolled test specimens were heat treated in an air-circulating furnace at 500°C for 0.5hr, and at 550°C for 3hrs. Several interrupted uniaxial tension tests were performed at $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$ and $T = 500^\circ\text{C}$ for these specimens. Optical micrograph of a specimen annealed at 500°C for 0.5hr and then deformed to a strain of 0.83 is shown in A1.2(a) and that of a specimen annealed at 550°C for 3hrs and then deformed to a strain of 0.88 is shown in Fig. A1.2(b). In the latter case only a few cavities are observed and they are relatively small. Quantitatively, Fig. A1.3 shows (a) the number density of voids and (b) the volume fraction of voids as a

function of strain for tested specimens. Both the void number density and the volume fraction of voids are much lower for the specimens annealed at 550°C for 3hrs. In addition, the rate of increase of void density with strain is relatively low for the specimen annealed at the higher temperature and longer time. This confirms that true sintering times are much larger than what is generally assumed, and if more complete healing of damage occurs, fewer defects remain and fewer voids are formed during the subsequent forming step. The observed voids in Fig. A1.2(a) may have grown from larger pre-existing defects which did not heal completely. These experimental results for the effect of healing of pre-existing damages to the overall cavitation damage are consistent with the data seen in Refs. 18-20.

Fig. A1.1. Optical micrographs of (a) an as-rolled specimen and (b) a specimen annealed at 550°C for 3hrs in an Al-Mg-Mn-Cu alloy. The number density of initial voids is plotted as a function of void diameter at the various annealing conditions in (c) and the total number density of initial voids ($> 0.5\mu\text{m}$ in diameter) is plotted as a function of time at two annealing temperatures in (d).

Fig. A1.3. The variation of (a) the void number density and (b) the volume fraction of voids with strain for the uniaxially deformed specimens annealed before testing for two annealing conditions; one was at 500°C for 0.5hr and the other was at 550°C for 3hrs. Tests were conducted at $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$ and $T = 500^\circ\text{C}$ and interruptions were made in the strain range of 0.15 to 0.9. Where more complete healing of defects occurs, fewer voids are formed.

Appendix 2. Creep cavitation models vs. observed superplastic cavitation

Many predictive models were developed for cavity growth during high temperature creep. A model based on diffusional growth of a grain boundary cavity was developed by Hull and Rimmer [14]. Based on this approach, Speight and Beere [29] derived the following cavity growth rate with respect to strain:

$$\frac{dr}{d\varepsilon} = \alpha \frac{\Omega \delta D_{gb}}{2 r^2 k T} \left[\frac{\sigma - (2\gamma / r)}{\dot{\varepsilon}} \right] \quad (\text{A2.1})$$

$$\text{where } \alpha = \left[\frac{1}{\ln(a/r) - (1 - r^2/a^2)(3 - r^2/a^2)/4} \right] \quad (\text{A2.2})$$

and r is the radius of each cavity located on a grain boundary with a spacing a , ε is the strain, $\dot{\varepsilon}$ is the strain-rate, Ω is the atomic volume, D_{gb} is the grain boundary diffusion coefficient, δ is the grain boundary width, σ is the applied stress, γ is the surface energy, k is Boltzmann's constant, and T is the absolute temperature. Another concept represented by plasticity-controlled cavity growth has been described by the following equation [30]:

$$\frac{dr}{d\varepsilon} = \frac{\eta}{3} \left(r - \frac{3\gamma}{2\sigma} \right) \quad (\text{A2.3})$$

where the cavity growth rate factor, η , is dependent on the applied stress-state, temperature, and the geometry of deformation [31-35].

To investigate the applicability of these models to superplastic cavitation, tests were performed under an optimum condition of superplasticity (i.e. $m = 0.55$ at $T = 550^\circ\text{C}$ and $\dot{\varepsilon} = 10^{-3}\text{s}^{-1}$). Substitution of the values of $\delta D_{gb} = 5 \times 10^{-14} \exp(-84 \times 10^3/RT)$ [36], $\gamma = 1.1\text{Jm}^{-2}$ [25], $\eta = 3$ [30] into Eq. (A2.1) and Eq. (A2.3), leads to the relationships between cavity growth rate (with respect to strain) and cavity radius shown in Fig. A2.1(a). Corresponding relationships between cavity radius and strain are shown in Fig. A2.1(b). For this calculation, it is assumed that a void pre-exists and it has the minimum size consistent with thermodynamic stability, which is $0.47 \mu\text{m}$ ($r_c = 2\gamma/\sigma$ where $\sigma = 4.7\text{MPa}$ [5]). From these calculations, it is seen that diffusional growth process dominates

the initial stage of void growth, and for void radii $> \sim 2\mu\text{m}$, growth is mainly controlled by plasticity. However, the expected void radius based on the combined growth by diffusion and plasticity processes is significantly lower than that observed experimentally (Fig. A2.1(b))^{*}. This disagreement between theory and experiment was also demonstrated by Pilling and Ridley [17] in SUPRAL 220. While a possible reason for this discrepancy was suggested as cavity coalescence at low strain, there is no strong evidence for this. Certainly for the present test material, there is no observed particle stringer formation, and no evidence of early coalescence of cavities to justify the large discrepancy observed. The predominance of single voids attached to the sides of particles further suggests that cavity coalescence at the early stage is not of any consequence for this alloy. In reality, however, the discrepancy between theory and experiment is considerably larger than what appears from Fig. A2.1(b). This is because the majority of the voids which emerge in the alloy are well below $r = 0.1\mu\text{m}$ in size when they become observable, and which occurs after considerable amounts of strain. This indicates that the criterion of $r_c = 2\gamma/\sigma$ is not a valid condition for cavity growth in this alloy, and secondly if Eqs. (A2.1) and (A2.3) are applied to these nanoscale voids, the discrepancy between theory and experimental graphs will be much greater (at least 3 orders of magnitude).

Other problems exist in applying the creep cavitation models. For example, in diffusional growth model, a regular distribution of small voids was assumed to exist on a single grain boundary. However, in most superplastic aluminum alloys voids are associated with non-deformable particles, and particle spacing is of the order of 1-2 grains or more. Voids are seldom found as a regular array on a single grain boundary, and the average cavity spacing is found to be of the order of 3 ~ 10 grains and also shown in the plot of Fig. A2.2. The average spacing in Fig. A2.2 is calculated from Fig. 7(b) with the dynamic variation of grain size vs. strain data [37]. For this calculation, the number of

^{*} In Fig. A2.1(b), the 25 or 50% area fraction is the radius of the cavity corresponding to a cumulative frequency of cavity section of area of 25% or 50% [5].

grains per volume is $(d_L \times d_T \times d_S)^{-1}$, where d_L , d_T , and d_S are the mean linear intercept grain sizes along L, T, and S directions, respectively, and voids are assumed to be homogeneously distributed in the specimen.

Thus, although Eq. (A2.1) has been used in prior work to predict the early growth of voids by grain boundary diffusion in coarse grain alloys under creep condition, critical examination of data for the fine-grained alloy suggests that these models may not be applicable here. Further insight into this issue of creep in coarse grain alloys vs. fine-grained superplastic metals may be gained by comparing the stresses to maintain a strain-rate of $1 \times 10^{-4} \text{ s}^{-1}$ in Al alloys with different grain sizes as shown in Fig. A2.3(a). Both the stress and cavity growth rate are plotted as a function of temperature in this figure. The stress data are experimental values found in the literature for the same (Al-Mg-Mn-Cu) and similar aluminum alloys (i.e. 5083 Al, 6061 Al, 7475 Al, and Al-3at%Mg). More of the data for fine grain alloys is shown at the higher temperature. In addition, the thermodynamically stable void sizes based on the equation, $r_c = 2\gamma/\sigma$, are marked on the right side for $r_c = 0.01$ and $0.1 \mu\text{m}$, a range of void sizes often quoted in the literature [38]. First, considering the low flow stress of the fine grain alloys (g.s. $\sim 8 \mu\text{m}$), the nucleation of cavities in this size range is not explainable, specially for temperatures $> 400^\circ\text{C}$. For coarse grain alloys, the higher flow stress in the $250 - 450^\circ\text{C}$ temperature range would permit the satisfaction of the critical stress. Many voids finer than this range have been observed in this and other work, which point to the inapplicability of the thermodynamic stability criterion for the present situation.

Flow stress is strongly dependent on grain size at the higher temperature. The stress level for a grain size of $\sim 200 \mu\text{m}$ (dotted line in Fig. A2.3(a)) is 7 ~ 8 times higher than that of the grain size of $8 \mu\text{m}$ in the temperature range of $450 - 550^\circ\text{C}$. The volumetric void growth rate ($\delta V/\delta t$) due to diffusion along the grain boundary [14, 29] is calculated by using Eq. (A2.1) and shown in Fig. A2.3(b). Because the flow stresses in Fig. A2.3(a) are well below the critical stress for the observed values of r_c in the material, Eq. (A2.1)

predicts no diffusional growth unless an unusually large initial value of r_c of $1\mu\text{m}$ is selected to obtain the results in Fig. A2.3(b). Because much early growth occurs prior to void radius reaching $1\mu\text{m}$, it is clear that superplastic void growth via diffusion cannot explain the actual observation in this alloy. Hence, although vacancy transport along the grain boundary may be a contributor for the initial void growth in large-grained materials, it cannot be a major contributor to initial void growth in fine-grained materials at superplastic strain-rates. Rather diffusional transport tends to sinter voids in fine grain materials. The early stage of cavitation is believed to be dominated by interface-constrained plasticity growth [8].

Fig. A2.1. (a) The calculated variation of void growth rate per strain vs. cavity radius and (b) the cavity radius vs. strain relationship at a uniaxially deformed condition of $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$ and $T = 550^\circ\text{C}$. There is a large difference between the experimental data and the predictions from existing models.

Fig. A2.2. The calculated number of grains per void ($> 0.5 \mu\text{m}$ in diameter) with strain for three strain-rates at $T = 500^\circ\text{C}$. The calculation is based on the data from Fig. 7(b) and the variation of grain size with strain [37], and assuming that voids are homogeneously distributed in the specimen.

Fig. A2.3. (a) Flow stress at a strain-rate of 10^{-4} s^{-1} plotted as a function of temperature for aluminum alloys with different grain sizes. Theoretical stress for stabilizing a spherical void, $\sigma = (2\gamma/r_c)$, for $r_c = 0.01$ and $0.1 \mu\text{m}$ are also indicated. (b) Volumetric void growth rate by diffusion calculated from Eq. (A2.1) is plotted as a function of temperature for two different grain sizes.

Fig. A1.2. Optical micrographs of tested Al-Mg-Mn-Cu alloy: (a) annealed at 500°C for 0.5hr and then deformed to $\epsilon = 0.83$ at $T = 500^{\circ}\text{C}$ and $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$ and (b) annealed at 550°C for 3hrs and then deformed to $\epsilon = 0.88$ at $T = 500^{\circ}\text{C}$ and $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$.

STRESS - STATE DEPENDENCE OF CAVITATION AND FLOW BEHAVIOR IN SUPERPLASTIC ALUMINUM ALLOYS

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ABSTRACT

A detailed and quantitative investigation of the stress-state dependence of superplastic cavitation has been conducted on fine-grained aluminum alloys. Several stress-states, such as uniaxial tension, plane-strain tension, plane-strain compression, shear, and equibiaxial tension have been examined. Tests were carried out in an interrupted manner under constant effective strain-rate ($\dot{\epsilon}_e$) in the range of 10^{-4}s^{-1} to 10^{-2}s^{-1} . Measurements of volume fraction, population density, and size distribution of cavities, made by image analysis via optical microscopy, show continuous emergence of new cavities as well as growth of cavities during superplastic straining. The total cavity volume fraction (V) increases exponentially with strain. Cavity growth rate represented by η ($= d \ln V / d \epsilon_e$) as well as cavity population change with strain ($dN_c / d \epsilon_e$, where N_c = cavity number/unit area) increase with the level of mean hydrostatic tension (σ_m / σ_e). For a fixed cavitation volume fraction, V , the principal surface strains, ϵ_1 and ϵ_2 , for the various stress-states can be empirically given by: $\epsilon_1 = a V^b - \alpha \epsilon_2$, where a and b are constants determined from ϵ_1 values for plane-strain ($\epsilon_2 = 0$). The value of b is found to be 0.2 ~ 0.3, and α is 0.4 - 1.0.

1 INTRODUCTION

During superplastic forming of fine grain metals, internal damage (cavities) often develops, the extent of which depends on the processing history of the alloy and forming conditions [1-6]. Superplastic cavitation is affected by strain, strain-rate, temperature, stress-state, as well as alloy chemistry, presence of second phase particles, microstructure, etc. [2, 6]. While cavitation damage beyond a critical level is unacceptable in load bearing structures due to its adverse effect on mechanical properties [7, 8], the complex interaction of the many responsible parameters makes it difficult to accurately predict the internal damage in formed components.

A critically important parameter affecting the degree of cavitation is the imposed stress-state. Various aspects of stress-state dependence of cavitation during hot working and superplastic forming processes have been studied by several investigators [2, 9-13]. Cavitation in formed parts has been reduced by the superposition of a hydrostatic pressure during the forming process, which can in some cases also increase the uniformity of strain in the part [9-13]. Superplastic cavitation can also be reduced by hot isostatic pressing following the forming process to sinter any voids [5]. These observations underscore the importance of mean tensile stress on cavitation.

Since the application of hydrostatic pressure is time-consuming and difficult for rapid manufacturing operations, this practice is not desirable in many industrial applications. Consequently, precise knowledge of cavitation including cavity sizes under various stress-states is necessary when no hydrostatic pressure is used. The extent of cavitation is generally characterized by total volume fraction of cavities, and it increases with increasing strain not only due to their increasing size but also due to continuous emergence of new cavities [14-16]. Thus while some cavities are found to pre-exist in conventionally processed alloys (processed by rolling and recrystallization), the population of cavities increases as they grow during deformation. The size of cavities as

related to their growth kinetics has a major contribution to the overall cavity volume^{*}. It has been suggested that cavity growth during high temperature deformation is controlled by diffusion of vacancies along the grain boundaries [17] or by plastic deformation of the matrix surrounding the cavity [18]. A coupled diffusion and plasticity process has also been suggested [19, 20]. The diffusional void growth is believed to be controlled by the maximum tensile stress normal to grain boundaries, and the plasticity-controlled void growth is controlled by the mean stress. However, much of the experimental data on superplastic alloys show that the growth of cavities is governed primarily by the mean stress, rather than the maximum principal stress [2].

A variety of surface strain and stress-state combinations is found in the formed parts, but detailed and quantitative information on the stress-state dependence of cavitation during superplastic deformation is limited. In this experimental study, five different stress-states have been considered: uniaxial tension, shear, equibiaxial tension, and plane-strain tension and compression. Through microstructural examination of the evolution of population density, size distribution, and volume of cavities with strain, the effect of stress-state on cavitation has been evaluated. In addition, iso-cavity volume contours under a variety of stress-states have been constructed by relating in-plane principal strains, ϵ_1 and ϵ_2 , which describe these contours, to serve as a manufacturing limit of superplastic formability under a variety of strain paths.

2 MATERIALS FOR STUDY

Two aluminum alloys based on Al-4.7%Mg composition were used, which are identified as Alloy I and Alloy II. Alloy I is a modified 5083 Al alloy containing Al-Mg-Mn-Cu and Alloy II is a fine-grained 5083 Al alloy containing Al-Mg-Mn. The chemical

^{*} Cavity interlinkage also has a major effect on cavity volume at large strains. This effect is excluded in this study as focus is placed on low levels of plastic strain and an alloy with low tendency for cavitation.

compositions of the alloys are given in Table 1. In Alloy I, the dispersed intermetallic particles are Al-Mn base compounds containing Cu; in Alloy II they are Al_6Mn particles [21]. Alloy I was obtained in DC cast condition from Kaiser Aluminum Company. This alloy was homogenized at 500°C for 12 hours and subsequently hot forged (from 30mm thickness down to 13mm at 250°C). To develop a fine grain microstructure, the alloy was cold rolled to 1.7mm final thickness (cold reduction ~ 90%), and then annealed at 500°C for 0.5 hour in a salt bath. Alloy II was purchased from Sky Aluminum Company in the form of a thermomechanically processed sheet of 2mm thickness. The linear intercept grain sizes before testing in the longitudinal (L), long transverse (T), and short transverse (S) directions are given in Table 2. Average grain sizes before testing for Alloy I and Alloy II are 7.9 μ m and 8.7 μ m, respectively. The uniaxial formability of both alloys was similar (see Appendix 1). Equibiaxial tension tests and comparable uniaxial tension tests were performed on Alloy II. All other stress-states were studied for Alloy I.

3 EXPERIMENTAL APPROACH

A variety of test specimens and test set-ups was used to obtain the different stress-states imposed on the superplastic alloys. The von Mises criterion was used for calculation of effective stress (σ_e) and effective strain (ϵ_e) for all tests. The geometries of the specimens and test methods for uniaxial tension, plane-strain compression, equibiaxial tension, and simple shear are schematically shown in Fig. 1. For the uniaxial tension test, an undeformed test specimen (specimen gage length = 12.7mm) and a specimen deformed to a strain of 0.7 (at $\dot{\epsilon} = 10^{-3}s^{-1}$ and $T = 500^\circ C$) are shown in Fig. 1(a). Crosshead speed was varied in order to produce a constant strain-rate in the sample, as described in Ref. 22. For the plane-strain compression test, the test method is schematically shown in Fig. 1(b). Several sheets stacked together and placed tightly in a channel die to constrain two parallel sides as the stack was compressed, which allowed the material to flow outward in a direction parallel to the channel illustrated in Fig. 1(b).

Again these tests were also conducted by controlling the crosshead speed during compression in order to maintain a constant strain-rate, using the assumptions that (i) the specimen deforms uniformly and (ii) volume remains constant.

For the equibiaxial tension test (bulge test), gas pressure forming was performed in a die with circular opening. The method of gas pressure control to maintain a constant $\dot{\epsilon}$ has been discussed in Ref. 23. Fig. 1(c) shows a schematic illustration of a bulge test and a sectional view of a deformed test piece with an effective strain of 0.7 (at $\dot{\epsilon}_e = 10^{-3} \text{s}^{-1}$ and $T = 500^\circ\text{C}$). For the simple shear test, Fig. 1(d) shows a planar simple shear device and test specimens in an undeformed condition as well as in a deformed condition. The shear strain for this sample is defined as $\Delta\ell / w = 1.45$, where $\Delta\ell$ = crosshead displacement and w = width of the specimen gage. The shear test device consists of two rigid specimen grip holders subjected to a parallel displacement during tensile pulling. The grip blocks are maintained parallel to the pulling direction by sliding blocks, which prevents rotation of the sample. Tests are carried out at constant crosshead speed which produces a constant shear strain-rate in the specimen. (The experimental method and deformation behavior in such a test are discussed in Ref. 24). Because the details of the above tests appear in Refs. 22 - 24, only the details of the plane-strain tension test are given here.

The common method to achieve the plane-strain tension condition is to pull wide specimens with narrow gage lengths ($w/l > 10$ where w is the gage width and l is the gage length) [9]. It is rather difficult to perform such a test with a wide tensile specimen at elevated temperature due to the difficulty associated with obtaining a suitable wide furnace, and maintaining uniformity of temperature and strain in the specimen. In this study, a 21mm wide test strip with a transverse groove (reduced section) was used and pulled as indicated in Fig. 2a. The small gage length (l) is the length of this groove which is of uniform initial thickness (t), being one-third of the grip thickness of the test specimen. The gage dimensions of the groove region before testing are: $w = 21\text{mm}$, $l =$

1.75mm, and $t = 0.6\text{mm}$. This produces a w/l ratio of 12. With an average grain size of $8\mu\text{m}$, the number of grains through the thickness is ~ 75 , which also satisfies the requirements of a valid mechanical test.

Two problems exist in such a test. One is that some strain occurs in the region of the specimen outside of the groove, which contributes to the crosshead displacement, thereby adding errors in strain estimation in the gage length region. The other is that transverse stress component becomes zero at the ends of the width of specimen gage, where the stress-state is uniaxial tension. To perform tests at constant strain-rate, an analysis of displacement rate in the different portions of the specimen is needed (see Fig. 2a). It is assumed that strain contribution comes from only the ungripped regions of the specimen (Region (b) in Fig. 2a). In an analysis of the displacement rate in the specimen, the specimen is divided into two regions: Region (a) [gage region] of an initial cross section area of A_{a0} and Region (b) of an initial cross section area of $A_{b0} (= 3 A_{a0})$. Based on force balance between these two regions and material constitutive equation: $\sigma = C\dot{\epsilon}^m$ (where $C = \text{constant}$, and $m = \text{strain-rate sensitivity}$, $\sigma = \text{flow stress}$ and $\dot{\epsilon} = \text{strain-rate}$), the relationship between ϵ_a and ϵ_b (where ϵ_a and ϵ_b are the strains in Regions (a) and (b), respectively) can be expressed as follows [25]:

$$\exp(-\epsilon_b / m) - 1 = (f)^{1/m} (\exp(-\epsilon_a / m) - 1) \quad (1)$$

where
$$f = \frac{A_a}{A_b} \quad (2)$$

A_a and A_b are the current cross section areas of Regions (a) and (b), respectively. Therefore, the crosshead speed (CHS) to maintain a constant strain-rate in Region (a) during the test is approximately

$$\text{CHS} = l_a \exp(\epsilon_a) \dot{\epsilon}_a + 2l_b \exp(\epsilon_b) \dot{\epsilon}_b \quad (3)$$

where l_a is the initial length of Region (a) ($= 1.75\text{mm}$) and l_b is the initial length of Region (b) ($= 6.2\text{mm}$).

For the problem involving the departure in the strain-state near the edge of the plane-strain tensile specimen, the extent of this departure was measured. A test specimen with grid lines printed over the gage region was prepared. The grid line spacing was 1mm. From deformed grid lines, the length and width strains along the specimen width are plotted in Fig. 2(b). This case represents a test conducted at $\dot{\epsilon}_e = 10^{-3}\text{s}^{-1}$ and $T = 550^\circ\text{C}$ for $\epsilon_L = 0.5$. The middle 75% of the width is uniformly deformed to $\epsilon_L \cong 0.5$ and $\epsilon_T \cong -0.045$ where T refers to the width direction (i.e. $\epsilon_T / \epsilon_L \cong -0.09$). Toward the edges of the width, the absolute value of the ratio, ϵ_T / ϵ_L , is high, as in uniaxial tension. The axial stress (σ_L) along the specimen width is thus assumed to be distributed as shown in Fig. 2(c). That is, the stress-state is uniaxial tension at the ends and is plane-strain tension in the middle portion. The axial stress (σ_L) in the middle portion of the gage width is then calculated from the measured load as discussed in section 5.1.

4 EXPERIMENTAL PROCEDURES

All mechanical tests for the various stress-states were performed under constant effective strain-rate. For uniaxial tension, plane-strain tension and compression, and simple shear tests, the tests were conducted at a test temperature of 550°C for Alloy I in the effective strain-rate range of 10^{-4}s^{-1} to 10^{-2}s^{-1} . Equibiaxial tests were carried out only on Alloy II at a test temperature of 500°C and effective strain-rates of 10^{-3}s^{-1} and 10^{-2}s^{-1} . Corresponding uniaxial tension tests were also conducted on Alloy II for comparison.

The tests other than for equibiaxial tension were performed by placing the specimen and load train within a clamshell furnace having three heating zones independently controlled to maintain temperature within $\pm 1^\circ\text{C}$ of the test temperature over a 130mm length. All test were performed at constant $\dot{\epsilon}$. To do this, crosshead speed was controlled by computer through a digital interface board on an Instron machine. Test interruptions were made to produce samples with a variety of strain levels in the effective

strain range of 0.4 to 1.2. To preserve the microstructure of strained samples, they were water quenched immediately after the desired strain level was reached. Bulge tests were performed for the equibiaxial tension state in a preheated die with the capability for gas pressurization. To maintain a constant strain-rate at the pole, a pre-calculated pressure - time profile was used to vary gas pressure continuously during the test [23].

Tested samples were sectioned along three orthogonal planes: L-S, L-T, and T-S planes, taken from the uniform part of the gage region or the pole area of the bulge samples, and then mechanically polished. Care was needed during polishing to prevent alteration of the appearance of cavities which could occur due to rounding of cavity edges by the polishing pressure. This was avoided by using an OP-Chem cloth supplied by Struers for polishing along with a colloidal silica suspension ($< 0.04\mu\text{m}$) in an automated polisher. To measure statistically meaningful values of the number and size of cavities with precision, metallographic sections were taken 0.5mm apart on three different planes. The measurement of cavity size was made on unetched specimens using digitized images taken with a CCD camera (640 x 480 pixels) through an optical microscope. More than 20 images (1400x) at different locations were taken for each of three different planes in each tested specimen. The dimension of each image was $273.5 \times 205.13\mu\text{m}$ with a pixel size of $0.427\mu\text{m}$. Two-dimensional metallographic measurements of cavities on the L-S, L-T, and T-S planes were conducted using NIH-Image software. The images of cavities were computer-fitted to (best-fit) ellipses of equivalent area. The resulting data consisted of the dimensions of the major and minor axes of the ellipse, and the orientation of the major axis, and an equivalent diameter for each cavity. The cavitation has been evaluated in this study by assessing the volume fraction, total population density, and size distribution of cavities. The volume fraction of cavities is equivalent to the area fraction of cavities [26] obtained by dividing the area of all captured cavities by the total image area. Also the number of all captured cavities divided by the total image area was defined as the population density of cavities. The cavity diameter data was later grouped into

various size groups to assist in plotting histograms of cavity population density vs. size (see Appendix 2).

5 RESULTS

5.1 Mechanical Behavior

For the plane-strain tension test, the distribution of axial stress (σ_L) along the gage width shown in Fig. 2(c) is used to determine the axial stress for the plane strain region:

$$\sigma_L = \left(\frac{2}{1+q} \right) \left(\frac{F}{A} - \left(\frac{1-q}{2} \right) \sigma_{\text{uniaxial}} \right) \quad (4)$$

where F is the force on the specimen, and A is the current cross section area, and q is the fraction of the gage width that is under plane-strain tension during deformation (e.g. $q = 0.75$). σ_{uniaxial} is the stress for uniaxial tension at the same longitudinal strain-rate. The level of uniaxial stress was estimated based on the data shown in Ref. 27. The axial stress (σ_L) vs. axial strain (ϵ_L) curves in the middle portion of gage width are shown in Fig. 3(a) at a test temperature of 550°C for three different effective strain-rates in Alloy I. The corresponding effective stress (σ_e) vs. effective strain (ϵ_e) curves are given in Fig. 3(b), together with the uniaxial tension data. These were calculated by using the von Mises criterion (see Table 3). The difference in effective flow stress between plane-strain tension and uniaxial tension is small. In addition, the flow behavior under simple shear for Alloy I was also investigated [24]. It should be mentioned that the von Mises effective stress vs. effective strain curves for shear were not always the same as those for uniaxial tension at 550°C (see Ref. 24 for the difference in the magnitude of effective stress between uniaxial tension and shear).

As shown in Fig. 3, the flow stress is found to be a strong function of strain-rate at 550°C for Alloy I, with a weak dependence on strain. The observed strain hardening behavior is believed to be a result of concurrent grain growth during deformation (see

Appendix 1 and Ref. 28). The mechanical behavior of Alloy I and Alloy II was found to be quite similar (see Appendix 1). For larger strains ($\epsilon > 0.4$), the flow stress of Alloy II was only slightly higher. This is possibly due to a slightly higher rate of concurrent grain growth in Alloy II (see Fig. A1.2). Also for Alloy II, a slightly higher initial strain-rate sensitivity (m) was observed in the strain-rate range of 10^{-4}s^{-1} to 10^{-2}s^{-1} (see Fig. A1.4).

5.2 Cavitation Characteristics

5.2.1 Optical Micrographs of Cavities

Micrographs of Alloy I specimens deformed at $T = 550^{\circ}\text{C}$, $\dot{\epsilon}_e = 10^{-2}\text{s}^{-1}$, and $\epsilon_e = 0.84$, are shown in Fig. 4 in the L-S and L-T planes for (a) uniaxial tension and (b) plane-strain tension. A significant difference in void size is observed between the different stress-states. For plane-strain tension, cavities larger than $50\mu\text{m}$ in diameter are found at several locations, but for uniaxial tension, voids are less than $25\mu\text{m}$. For the shear test, cavities less than the size of particles ($< 5\mu\text{m}$) were observed only at a few locations at $\epsilon_e = 0.84$. Both the size and population density of cavities were extremely small in shear. For plane-strain compression, cavities could not be found, implying that cavity growth is negligible or nonexistent. This is expected for the tests with negative mean stress. The micrographs revealed a broad distribution of cavity sizes. Cavities in the L-S plane are found to be generally elongated along the applied load direction (L) for uniaxial tension as well as plane-strain tension. In the L-T plane of the plane-strain tension test specimen, coalesced cavities were seen, which were aligned along the transverse direction (T) in several locations, possibly due to the presence of transverse stresses.

For Alloy II, optical micrographs of cavities for the test condition of $T = 500^{\circ}\text{C}$, $\dot{\epsilon}_e = 10^{-3}\text{s}^{-1}$ and $\epsilon_e = 1.12$ are shown in Figs. 5(a) and (b) for the case of (a) uniaxial

tension and (b) equibiaxial tension. These photos from the L-S and L-T planes also show a significant difference in cavity size between equibiaxial tension and uniaxial tension for this test condition. For equibiaxial tension, cavities larger than 50 μm in diameter are found at several locations, but for uniaxial tension, much smaller voids ($< 25\mu\text{m}$) are observed. There is also a broad range of cavity sizes in each micrograph. In the L-T plane of equibiaxial tension, large cavities are essentially equiaxed aligned along both L and T directions. By comparing Figs. 4 and 5, it is also seen that Alloy II has less propensity for cavitation possibly due to the lower population density of particles* or less damaged interfaces [16] in Alloy II.

5.2.2 Evolution of Cavitation Damage

The change in volume fraction, total population density, and size distribution of cavities with increasing strain has been quantitatively examined for several stress-states and compared with similar data for uniaxial tension.

(a) *Plane-Strain Tension*

The volume fraction and population density of cavities are plotted as a function of effective strain in Fig. 6(a) and (b), respectively, at 550°C in Alloy I, together with similar data for uniaxial tension. The data is averaged over three orthogonal sections: L-S, L-T and T-S planes. The linear curves in the semi-logarithmic plot in Fig. 6(a) indicate that the volume fraction of cavities increases exponentially with strain. Such data suggest that overall void growth is controlled by plasticity [18]. In addition, the cavity population increases with increasing strain (Fig. 6(b)), indicating that new cavities continuously emerge due to either nucleation or growth from submicron-scale damage or both. (Diffusional cavity growth is found to be too slow and not important [16]). Fig. 6 shows that the level of cavitation in plane-strain tension is much higher than that in uniaxial

* The population density of particles larger than 1 μm in diameter for the static annealing condition was 4086.4/mm² and 377.9/mm² for Alloy I and Alloy II, respectively. These data were obtained from optical microscopy images for particles ($> 1\mu\text{m}$).

tension irrespective of strain-rate¹. The rate of increase of cavity population is also higher for plane-strain tension. However, increasing strain-rate increases both the overall cavitation and the rate of cavity emergence. A detailed evaluation of the continuous formation and growth of cavities with strain is given in Appendix 2.

(b) *Simple Shear*

The volume fraction and population density of cavities in Alloy I from planar shear tests are plotted as a function of effective strain-rate in Figs. 7(a) and (b), respectively, together with the similar data for uniaxial tension (for $T = 550^{\circ}\text{C}$ and $\epsilon_e = 0.84$). The level of cavitation in shear is less than that in uniaxial tension but not zero near the specimen edges where some additional tensile components are present. As shown in Fig. 7, an increase in strain-rate produces a greater cavity population density and an increased level of overall cavitation. In addition, for strain-rates below 10^{-4}s^{-1} the level of cavitation is similar or lower than that for pre-existing defects in the undeformed alloy [16]. This suggests that due to the compressive component during the shear test, cavity nucleation cannot occur until the applied stress becomes large.

(c) *Equibiaxial Tension*

The volume fraction and population density of cavities in Alloy II are plotted as a function of effective strain in Figs. 8(a) and (b), respectively, for $T = 500^{\circ}\text{C}$, together with the similar data for uniaxial tension. Overall, the level of cavitation in equibiaxial tension is higher than that in uniaxial tension irrespective of strain-rate, and equibiaxial tension produces the greater population density of cavities. Again cavities are found to be continuously nucleated with strain, and total volume of cavities increases exponentially with strain. However, the population density of cavities is relatively unaffected by strain-rate for Alloy II. The dependence of cavitation on the experimental parameters in

¹ The population density of cavity in plane-strain tension is only slightly greater than that in uniaxial tension for a strain-rate of 10^{-2}s^{-1} .

superplastic aluminum alloys cannot be simply described because many processes are involved e.g. interface-constrained debonding in the early stages of deformation, the continuous nucleation of new cavities and plasticity-based cavity growth. Furthermore, each of these processes has a different dependency on the experimental parameters as discussed in Ref. 29. The size distribution of cavities in the above conditions is also given in Appendix 2.

5.2.3 Monitoring Cavity Growth

Cavities grow with increasing superplastic strain, but monitoring cavity growth kinetics for individual cavities is not feasible due to the broad distribution of cavity sizes involved. Furthermore, in addition to the pre-existing cavities, new cavities continuously emerge and grow during deformation. Quantitative estimate of void growth rate would be meaningful if a representative void size is selected to monitor its growth. Monitoring of growth should be done for cavities which have definitely passed the stage of nucleation, and also exclude the largest cavities whose size can be influenced by the coalescence of cavities. The representative cavity size at a fixed level of strain is thus selected as the cavity diameter which corresponds to 50% cumulative frequency of cavity area, d_{50} , from the cavity size distribution plot as described in Ref. 29. The assumption here is that even though new cavities continuously emerge, the 50% point (d_{50}) is not influenced by them and that the cavity coalescence effects are also small as long as cavitation is measured at small strain levels. The median value of d_{50} obtained from the L-S, L-T and T-S planes is plotted on a semi-logarithmic scale in Fig. 9. This median size increases exponentially with increasing strain, indicating that void growth is generally plastically controlled [18]. Again, the cavity growth kinetics indicated by the slope of these plots increases with increasing degree of biaxiality (i.e. greater for plane-strain tension and equibiaxial tension than for uniaxial tension). Thus, if σ_m is defined as the mean stress

and σ_e the von Mises effective stress, the void growth rate ($d\ln r / d\epsilon$) increases with increasing σ_m / σ_e .

6 DISCUSSION

As discussed in Ref. 30, in superplastic aluminum alloys containing particles on grain boundary, cavities initiate at particle interfaces, possibly from pre-existing defects of submicron size. The formation and initial growth of the cavities have been shown to be due to decohesion at particle/matrix interface and their subsequent enlargement, which have rapid kinetics [30]. More new cavities continuously emerge during deformation and the slower process of diffusional growth does not explain the observed magnitude of cavity volume [16]. After debonding of the entire particle interface, growth of cavities is not constrained to the interface and is controlled primarily by the plastic deformation of the surrounding matrix, which is a somewhat slower process. Complex dependencies of cavitation on experimental parameters such as strain, strain-rate, and temperature in uniaxial tension have been reported in Ref. 29.

Plasticity-controlled cavity growth has been described in the literature by the following equation [18, 19]:

$$\frac{dr}{d\epsilon} = \frac{\eta}{3} \left(r - \frac{3\Gamma}{2\sigma_e} \right) \quad (5)$$

where r is the cavity radius, ϵ is the strain, σ_e is the effective stress, Γ is the surface energy of the cavity and η is the cavity growth rate factor ($d\ln V / d\epsilon$). Stress-state dependency of cavity growth is sometimes described by considering the dependence of η in Eq. (5) on stress-state. Following the analysis of Cocks and Ashby [31], Stowell *et al.* [32] and Pilling and Ridley [2] expressed η in terms of strain-rate sensitivity of flow stress (m) and the ratio, σ_m^L / σ_e , where σ_m^L is the local mean stress as

$$\eta = \frac{3}{2} \left(\frac{m+1}{m} \right) \sinh \left[2 \left(\frac{2-m}{2+m} \right) \left(\frac{k_s}{3} - \frac{P}{\sigma_e} \right) \right] \quad (6)$$

where

$$\left(\frac{k_s}{3} - \frac{P}{\sigma_e} \right) = \frac{\sigma_m^L}{\sigma_e} \quad (7)$$

P is the superimposed pressure and k_s is a constant depending on the geometry of deformation and the extent of grain boundary sliding (GBS) [2, 31, 33]. For the case of fully rigid grains (no GBS), the local mean stress, σ_m^L , is equal to the remote mean stress, σ_m , while the extent of mean stress on sliding grain boundaries would be higher (see Ref. 2), leading to the higher rate of cavity growth. Under the optimum superplastic condition, it is believed that approximately 50% of the accumulated strain is accommodated by GBS [2]. The values of σ_m / σ_e for several stress-states are summarized in Table 3.

To evaluate how stress-state affects η parameter for Al alloys, η -values were calculated from experimental cavitation data. The slopes ($= d \ln V / d \epsilon$) from Figs. 6(a) and 8(a) for Alloy I and Alloy II are plotted in Fig. 10, along with η data for similar aluminum alloys [2] (at 460°C) as functions of σ_m / σ_e . Also shown on this plot are model-based theoretical estimates, by using Eqs. (6) and (7) as indicated by dotted lines for $m = 0.48$ as a comparison plotted as functions of σ_m / σ_e . The values of k_s used in Eqs. (6) and (7) are obtained from Ref. 2. These values are respectively, $k_s = 1 - 1.5$ for uniaxial tension, 1.73 - 2.16 for plane-strain tension, and 2 - 2.25 for equibiaxial tension. The lower values of above sets represent a case for no GBS while the higher values are stated to be valid when 50% of the accumulated strain is accommodated by GBS. Cavity growth rate factor, η , is seen to be strongly dependent on the applied stress-state, based on both experimental data and theoretical estimates. This effect is a result of rapid lateral enlargement of cavities under biaxial stress conditions. For a fixed σ_m / σ_e , however, the variation in the value of η for similar alloys is also significant. This depends largely on the microstructure, inclusion, dispersoid particle size, and particle's volume fraction in

the alloy, and also on experimental parameters, such as strain-rate and temperature. The effect of these factors can be larger than the stress-state effect. These experimental dependencies could not be simply described by the existing theoretical models. From Fig. 10, it is clear that the values of η and the slope, $d\eta/d(\sigma_m/\sigma_e)$, are not described well by the estimates of Eqs. (6) and (7). It is also clear that the rate of increase of η with σ_m/σ_e is generally overestimated by the existing theory [2, 31]. A critical observation is that fine grain alloys generally have a lower degree of cavitation, such as Supral 220. This opposite of cavitation wisdom which suggests that more GBS would be expected in a fine grain alloy. Thus the theoretical assertion of GBS producing higher cavity growth rate may not be true if diffusional accommodation is sufficiently high.

An estimation of the overall effect of stress-state on cavity nucleation rate has also been made. Fig. 11 shows the rate of increase of cavity population density with effective strain ($dN_c/d\epsilon_e$ where N_c = total cavity number / mm²) as a function of the mean stress ratio, σ_m/σ_e . The cavity nucleation rates for Alloy I (at 550°C) and Alloy II (at 500°C) were determined from the slope of the plots in Figs. 6(b) and 8(b). In Fig. 11, $dN_c/d\epsilon_e$ is found to increase with increasing σ_m/σ_e . The effect of hydrostatic tension on cavity nucleation rate is believed to stem from the stress-state effect on particle interface debonding and early growth. For a pre-existing defect located at the particle-matrix interface, hydrostatic tension causes debonding and/or rapid enlargement of submicroscopic defects [34]. The early growth of these defects (as in hole growth models) can appear to be a "nucleation" effect. This hydrostatic stress effect is akin to the effect of high flow stress resulting from lower test temperatures or higher applied strain-rates. However, as Fig. 11 shows the stress (or strain-rate) effect on cavity nucleation is much stronger. Thus the overall dependence of cavitation on σ_m/σ_e is due to both the higher rate of increase of cavity population density and the higher cavity growth rate.

Superplastic forming processes represent a wide range of deformation states. While most of these states lie between plane-strain tension and equibiaxial tension, the

strain limit of forming can be described by forming limit diagrams (FLD) in the range between uniaxial tension and equibiaxial tension (as in conventional sheet metal forming). These diagrams cover a range of principal surface strains from tension-compression states to tension-tension states. Due to the existence of cavitation damage during superplastic forming [1-6], the definition for limiting deformation in superplastic forming is generally related to a fixed level of cavitation within the material rather than necking or fracture [8]. Thus over a range of strain-states, contours representing iso-cavity volume may be drawn to designate the limiting strains for forming operations. Two different representations of iso-cavitation limit diagram are presented here: one is effective strain for selected iso-cavity volume vs. σ_m/σ_e , and the other is the relationship between principal surface strains, ϵ_1 and ϵ_2 , for iso-cavity volume under a variety of strain-states.

Based on cavity volume data given in Figs. 6(a) and 8(a), cross plotting of the effective strains required to reach fixed cavity volume, V , of 0.5% and 5% allows us to construct one such diagram shown in Fig. 12(a) as a function of the ratio, σ_m/σ_e , for an effective strain-rate of 10^{-3}s^{-1} . This limiting effective strain decreases as the ratio, σ_m/σ_e , increases. This is consistent with trends of the higher values of η (Fig. 10) and $dN_c/d\epsilon_e$ (Fig. 11) at the higher ratio, σ_m/σ_e . The region below a selected limiting curve may be designated as the "recommended processing zone". In this case the cavitation level of 0.5% has been shown as the limiting criterion since some deterioration of mechanical properties may be expected above this level based on prior studies [8]. However, selection of this level may be application-dependent. For example, a higher level of cavitation may be tolerable in certain structural components which are either non-critical or less critical during service.

The other representation of iso-cavitation limit diagram is the locus of principal surface strains, ϵ_1 and ϵ_2 , required to produce a fixed cavitation level. Based on the

cavity volume data for plane-strain tension (Fig. 6(a)), shear (Fig. 7(a))⁺, and equibiaxial tension (Fig. 8(a)), again the effective strains for a fixed cavity volume under a variety of stress-states are determined. These effective strains are then converted to the surface strains, ϵ_1 and ϵ_2 , as follows:

$$\epsilon_1 = \frac{\sqrt{3}}{2} \frac{\epsilon_e}{\sqrt{(1 + \rho + \rho^2)}} \quad (8)$$

$$\text{where } \rho = \epsilon_2 / \epsilon_1 \quad (9)$$

The values of ρ for different stress-states are summarized in Table 3. Figs. 12(b) and 12(c) show the surface strains based on contours for cavity volume fraction, V , of 0.5, 2, and 5% at an effective strain-rate of 10^{-3}s^{-1} . The observed linear contours can be described by the equation:

$$\epsilon_1 = a V^b - \alpha \epsilon_2 \quad (10)$$

where a , b , and α are constants. These contours help in deciding whether forming to a desired level of part severity would be possible while staying below the critical level of cavitation. The values of a and b are obtained from ϵ_1 values for plane-strain tension ($\epsilon_2 = 0$), and listed in Table 4, in which b is found to be approximately 0.2 ~ 0.3, and α from Eq. (10) is found to be in the range of 0.4 to 1, increasing with decreasing strain-rate for Alloy I (at 550°C). These findings suggest that only a few tests would be required to evaluate a , b , and α for a material and develop iso-cavitation limit diagram as a function of strain path. It might be noted that the slope of the limiting curves in Fig. 12(b) and (c) is approximately - 0.7 (based on internal cavitation). This value compares favorably with

⁺ For the shear test, theoretical value of η is zero. However, slight cavitation has been observed in the planar shear test, because the local stress-state may be different from the remote stress-state. Since the achievement of the high level of cavity volume by shear deformation is difficult, cavity volumes at an effective strain-rate of 10^{-3}s^{-1} in Fig. 12(b) were obtained by exponentially extrapolating the following data: ($V = 0.017\%$ at $\epsilon_e = 0$, $V = 0.095\%$ at $\epsilon_e = 0.84$).

values of about - 0.5 to - 0.75 for forging limit of steels and aluminum alloys reported by Kuhn [35] and with values of - 0.4 to - 0.5 for forging limit of an aluminum composite material reported by Syu and Ghosh [36]. These forging limit diagrams were determined based on surface cracks, and it is interesting that the values of the slopes of the limiting curves match reasonably well with that obtained based on internal cavitation.

7 SUMMARY AND CONCLUSIONS

Cavitation and flow behavior in superplastic aluminum alloys have been investigated under a variety of stress-states: uniaxial tension, plane-strain tension, plane-strain compression, shear, and equibiaxial tension, to pinpoint the role of stress-state on both superplastic cavity nucleation and growth. Constant effective strain-rate tests were carried out to several preselected strain levels within a strain-rate range of 10^{-4}s^{-1} to 10^{-2}s^{-1} and temperatures of 500°C and 550°C . The change in volume fraction, total population density, and size distribution of cavities with increasing strain has been quantitatively examined. The level of cavitation increased with increasing level of mean hydrostatic tension (σ_m / σ_e). The observations and primary conclusions of this study are listed below:

1. A broad distribution in cavity size is seen at different strain levels. Compared to the narrow range of sizes of the pre-existing cavities, new cavities continuously emerge and grow during deformation.

2. Increase in cavity population with increasing strain is aided by hydrostatic tension. Mean tensile stress enhances continuous cavity "nucleation".

3. Median cavity size exponentially increases with increasing strain, indicating that cavity growth is controlled by plasticity. The median size also increases with increasing hydrostatic tension, i.e. increasing value of σ_m / σ_e .

4. The total volume fraction of cavities increases exponentially with strain due to the combination of the two effects mentioned above, i.e. due to an increase in the cavity population density and the growth of individual cavities with strain. In addition, a higher level of cavitation is observed at higher values of σ_m/σ_e due to enhanced cavity nucleation rate ($dN_c/d\epsilon_e$) and a higher cavity growth rate (η) at higher σ_m/σ_e .

5. Contours of iso-cavity volume fraction, V , constructed by relating principal strains, ϵ_1 and ϵ_2 , in the sheet provide a forming limit for superplastic forming of the alloys examined. For the various strain-states, this limiting curve can be described empirically by: $\epsilon_1 = a V^b - \alpha \epsilon_2$, where a and b are constants obtained from ϵ_1 values for plane-strain ($\epsilon_2 = 0$) in which b is found to be of the order of 0.2 ~ 0.3, and α is found to range between 0.4 and 1. When iso-cavitation limit is described by a locus of effective strain, its level decreases as the ratio, σ_m/σ_e , increases.

6. Effective stress vs. effective strain curves for plane-strain tension are found to be essentially the same as those for uniaxial tension at 550°C.

8 ACKNOWLEDGEMENTS

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Table 1. Chemical Compositions of Alloy I and Alloy II (wt%)

	Mg	Mn	Cr	Cu	Al
Alloy I (Modified 5083 Al alloy)	4.75	0.8	0.2	0.4	rest
Alloy II (5083 Al alloy)	4.7	0.71	0.14	-	rest

Table 2. Initial Grain Sizes (μm) of Alloy I and Alloy II

	d_L	d_T	d_S	d_{avg}
Alloy I (Modified 5083 Al alloy)	9.68	9.37	5.4	7.94
Alloy II (5083 Al alloy)	9.84	9.80	6.75	8.67

where d_L , d_T , d_S are the grain dimensions in the longitudinal (L), long transverse (T), short transverse (S) directions, respectively, and $d_{\text{avg}} = \sqrt[3]{d_L \times d_T \times d_S}$.

Table 3. A Summary of the Expressions for von Mises Effective Stress (σ_e), Hydrostatic Stress (σ_m), Maximum Principal Stress (σ_1) and the ratio, ρ ($= \epsilon_1/\epsilon_2$), for Different Stress-states.

	Equivalent State	σ_e	σ_m/σ_e	ρ	σ_1/σ_e
Uniaxial Tension	$\sigma_1 = \sigma_U, \sigma_2 = 0, \sigma_3 = 0$	σ_U	1/3	-1/2	1
Plane-strain Tension	$\sigma_1 = \sigma_{PS}, \sigma_3 = 0,$ $\sigma_2 = (1/2)\sigma_{PS}$	$\sqrt{3}/2 \sigma_{PS}$	$\sqrt{3}/3$	0	$2\sqrt{3}/3$
Equibiaxial Tension	$\sigma_1 = \sigma_B, \sigma_2 = \sigma_B, \sigma_3 = 0$	σ_B	2/3	1	1
Plane-strain Compression	$\sigma_1 = -\sigma_{PS}, \sigma_3 = 0,$ $\sigma_2 = -(1/2)\sigma_{PS}$	$\sqrt{3}/2 \sigma_{PS}$	$-\sqrt{3}/3$	0	$-2\sqrt{3}/3$
Shear	$\sigma_1 = \tau, \sigma_2 = -\tau, \sigma_3 = 0$	$\sqrt{3}\tau$	0	-1	$\sqrt{3}/3$

Table 4. Constant a , b , and α in Eq. (10) for the Relationship Between Two Principal Strains, ϵ_1 and ϵ_2 , at Iso-cavity Volume Fraction, V .

Material	Temperature	Effective Strain-rate	a	b	α
Alloy I	550°C	10^{-2} s^{-1}	1.249	0.217	0.42
		10^{-3} s^{-1}	1.397	0.23	0.70
		10^{-4} s^{-1}	1.829	0.222	0.9*
Alloy II	500°C	10^{-2} s^{-1}	2.515	0.288	0.75
		10^{-3} s^{-1}	2.275	0.311	0.75

* The value of α at $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$ and $T = 550^\circ\text{C}$ for Alloy I was obtained from the data of uniaxial tension and plane-strain tension only.

Fig. 1. The geometries of test specimens and testing methods for (a) uniaxial tension, (b) plane-strain compression, (c) equibiaxial tension, and (d) simple shear.

Fig. 2. (a) Diagram of plane-strain tension specimen grip holder and test specimen as well as photos of test specimens. (b) Distribution of length and width strains along the width of specimen gauge at an overall length strain of 0.5 (at $\dot{\epsilon}_e = 10^{-3} \text{s}^{-1}$ and $T = 550^\circ\text{C}$). The middle portion of gauge width is uniformly deformed with $\epsilon_T / \epsilon_L \approx -0.09$, but departure from the plane-strain condition is observed near the edges of the specimen gauge. Based on this flow pattern, the distribution of axial stress (σ_L) along the width of specimen gauge is assumed as shown in (c).

Fig. 3. For the plane-strain tension test, (a) axial stress (σ_L) vs. axial strain (ϵ_L) curves in the middle portion of gauge width ($0.75w$) at a test temperature of 550°C in Alloy I, calculated by using Eq. (4). (b) The corresponding effective stress vs. effective strain curves (using the von Mises criterion), together with the data of uniaxial tension.

Fig. 3. For the plane-strain tension test, (a) axial stress (σ_L) vs. axial strain (ϵ_L) curves in the middle portion of gauge width ($0.75w$) at a test temperature of 550°C in Alloy I, calculated by using Eq. (4). (b) The corresponding effective stress vs. effective strain curves (using the von Mises criterion), together with the data of uniaxial tension.

(a) Uniaxial Tension

(b) Plane Strain Tension

Fig. 4. Optical micrographs of cavities at an effective strain of 0.84 in the L-S and L-T planes in Alloy I. Tests were conducted at an effective strain rate = $1 \times 10^{-2} \text{s}^{-1}$ and $T = 550^\circ\text{C}$ under (a) uniaxial tension and (b) plane-strain tension.

(a) Uniaxial Tension

(b) Equibiaxial Tension

Fig. 5. Optical micrographs of cavities at an effective strain of 1.12 in the L-S and L-T planes in Alloy II. Tests were conducted at an effective strain-rate = $1 \times 10^{-3} \text{s}^{-1}$ and $T = 500^\circ\text{C}$ under (a) uniaxial tension and (b) equibiaxial tension.

Fig. 6. The variations of (a) volume fraction and (b) population density of cavities with effective strain for plane-strain tension and uniaxial tension at a temperature of 550°C in Alloy I (All cavities having average diameter > 0.5μm are counted).

Fig. 7. The variations of (a) volume fraction and (b) population density of cavities with effective strain-rate for simple shear and uniaxial tension at an effective strain of 0.84 at 550°C in Alloy I. "Initial Condition" indicates the level of pre-existing defects in the undeformed alloy.

Fig. 8. The variations of (a) volume fraction and (b) population density of cavities with effective strain for equibiaxial tension and uniaxial tension at a temperature of 500°C in Alloy II.

Fig. 9. The variation of cavity diameter corresponding to 50% area fraction with effective strain in several stress-states for Alloy I shown in (a) and (b) and for Alloy II shown in (c). Generally, the larger cavity size is found at the higher ratio, σ_m / σ_e , at a given effective strain.

Fig. 10. Experimentally determined cavity growth rate factors (η) plotted as a function of the ratio, σ_m / σ_e , for various aluminum alloys (solid lines), compared with predictions based on Eqs. (6) and (7) (dotted lines) for $m = 0.48$ ($k_s = 1 - 1.5$ for uniaxial tension, 1.73 - 2.16 for plane-strain tension, and 2 - 2.25 for equibiaxial tension). The lower values represent the case for no grain boundary sliding while the higher values would be valid when 50% of the accumulated strain is accommodated by grain boundary sliding).

Fig. 11. The rate of increase of cavity density with effective strain, ($dN_c / d\epsilon_e =$ nucleation rate), plotted as a function of the ratio, σ_m / σ_e , in Alloy I at 550°C and Alloy II at 500°C.

Fig. 12. (a) The effective strain vs. σ_m / σ_e for $V = 0.5\%$ and $V = 5\%$ at $\dot{\epsilon}_e = 10^{-3} \text{ s}^{-1}$ in Alloy I at 550°C and Alloy II at 500°C . The "recommended processing zone" would be defined below a critical cavitation level of choice, such as 0.5% , as suggested in Ref. 8. (b) Principal surface strains, ϵ_1 and ϵ_2 , at several iso-cavity volume levels at $\dot{\epsilon}_e = 10^{-3} \text{ s}^{-1}$ and $T = 550^\circ\text{C}$ for Alloy I and (c) for Alloy II at $\dot{\epsilon}_e = 10^{-3} \text{ s}^{-1}$ and $T = 500^\circ\text{C}$.

Appendix 1. Mechanical behavior of the two test alloys

The need to use the two different alloys in this research was a result of unavailability of large enough sheets of Alloy I to permit bulge testing experiment. Thus a similar alloy was obtained for this part of the study, Alloy II, which needed to be examined to verify if its properties are similar. Furthermore, the differences between the two alloys were intended to add an extra dimension to the investigation of the property variations between the different superplastic alloys. Uniaxial tension tests were carried out at constant strain-rates of 10^{-3}s^{-1} and 10^{-2}s^{-1} at 500°C . Stress vs. strain curves from interrupted tests are shown in Fig. A1.1. The two alloys are found to behave virtually the same except at large strains. The departure at large strains might indicate either our inability to maintain true constant strain-rate till large strains or a slight difference in the strain hardening behavior between the two alloys, indicative of their slightly different grain growth kinetics. This latter possibility was verified by metallography of specimens examined after interrupted tests. The grain size measurements taken from these samples are shown in Fig. A1.2 as a function of strain.

To obtain a more detailed characterization of the mechanical properties of the two alloys, the strain-rate dependence of flow stress is characterized. The stress vs. strain-rate relationship has been obtained from step strain-rate test. These are obtained by special tests described in Refs. 22 and 27 in which the effects of concurrent grain growth are avoided. Sigmoidal relationships between the logarithms of the stress and strain-rate are shown in Fig. A1.3(a). The behavior for the two alloys is very similar. In the low strain-rate range (10^{-5}s^{-1} - 10^{-3}s^{-1}), Alloy I shows slightly higher flow stress than those of Alloy II, although the grain size of Alloy I is slightly smaller. This may be due to the higher Cu content in Alloy I. Strain-rate sensitivity ($m = d \log \sigma / d \log \dot{\epsilon}$) vs. strain-rate relationships are plotted in Fig. A1.3(b). This derivative plot accentuates small differences between the alloys, and so it is found that the maximum m is 0.47 for Alloy I at a strain-rate of $1.4 \times 10^{-3}\text{s}^{-1}$, as compared with 0.55 for Alloy II at a strain-rate of $1.1 \times 10^{-3}\text{s}^{-1}$.

Fig. A1.1. Stress vs. strain curves from uniaxial tension tests under constant strain-rates of 10^{-3} s^{-1} and 10^{-2} s^{-1} at 500°C in Alloy I and Alloy II, in which interruption was made at a strain of 0.8.

Fig. A1.2. The average grain size ($d_{\text{avg}} = \sqrt[3]{d_L d_T d_S}$) against strain at 500°C in uniaxially deformed Alloy I and Alloy II, in which d_L , d_T , and d_S are the linear intercepted grain sizes in L, T, and S directions, respectively.

Fig. A1.3. (a) Stress versus strain-rate curves and (b) m values in an isostructural condition ($0 < \epsilon < 0.1$) at 500°C in Alloy I and Alloy II.

Appendix 2. The size distribution of cavities

Micrographs of superplastically deformed specimens have shown cavities with a broad distribution in size. A quantitative evaluation of this distribution was conducted by analyzing the cavity population density as a function of cavity size. Since the shape of cavities observed in the micrographs of tested specimens was irregular and simultaneous correlation among the three orthogonal dimensions for each cavity is impossible via area-based measurements, a previously developed procedure for estimating individual cavity volume and size distribution (per Ref. 29) was used.

For plane-strain tension, the size distribution of cavities at different levels of effective strain is plotted in Fig. A2.1 at effective strain-rates of (a) 10^{-2}s^{-1} and (b) 10^{-4}s^{-1} at 550°C in Alloy I, together with the similar data of uniaxial tension. Two important trends are observed from these plots: (i) there is a distribution of size associated with the cavities, which is present from the start (i.e. cavities are not mono-sized) and gets larger with increasing strain, and (ii) while a large number of bigger cavities are seen with increasing strain, the number of small size cavities also tends to increase. The shape of these distribution curves clearly indicate that continuous nucleation of cavities from yet finer particles continues to occur, as the larger cavities grow. Without this feature the distribution curves would develop a truncated shape in the small size range. The level of cavitation in plane-strain tension is found to be much higher than that for uniaxial tension in terms of both the population density and the size of cavities.

For equibiaxial tension, the size distribution of cavities at different levels of effective strain is plotted as a function of cavity size in Fig. A2.2 at effective strain-rates of (a) 10^{-2}s^{-1} and (b) 10^{-3}s^{-1} at 500°C in Alloy II, together with the similar data of uniaxial tension. Again the same trends seen in plane-strain tension are observed. Overall, the level of cavitation in equibiaxial tension is higher than that in uniaxial tension.

Fig. A2.1. Size distribution of cavities under plane-strain tension at effective strain-rates of (a) 10^{-2} s^{-1} and (b) 10^{-4} s^{-1} at 550°C in Alloy I, together with the similar data of uniaxial tension.

Fig. A2.2. Size distribution of cavities under equibiaxial tension at effective strain-rates of (a) 10^{-2} s^{-1} and (b) 10^{-3} s^{-1} at 500°C in Alloy II, together with the similar data of uniaxial tension.

A PLANAR SIMPLE SHEAR TEST AND FLOW BEHAVIOR IN A SUPERPLASTIC Al - Mg ALLOY

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ABSTRACT

Superplasticity is generally studied by performing tensile and gas-pressure bulge tests. In formed parts, however, a variety of strain-states including in-plane shear are encountered. The understanding of the mechanical response in shear is helpful in the study of superplastic metal forming. In this study, a device for a planar simple shear test was designed and used to perform tests on a superplastic Al-Mg alloy sheet at elevated temperatures of 500°C and 550°C. In such a test, the incremental rotation of the principal strain axes and specimen-end effects during deformation can complicate the determination of true mechanical response. The possible approximations regarding the strain-state in the specimen gage have been investigated. $\sigma_e - \epsilon_e$ curves obtained based on simple shear assumption show a lower flow stress than that under uniaxial tension and strain hardening is related to dynamic grain growth. The rate of strain hardening at a fixed $\dot{\epsilon}_e$ is essentially the same for both uniaxial tension and shear, but the difference in the effective stress between uniaxial tension and shear depends upon strain-rate and temperature. This study marks the first known characterization of simple shear response under superplastic conditions.

1 INTRODUCTION

A deformation mode of shear is an important stress-state in many sheet metal forming operations. In order to study the mechanical response and microstructural evolution characteristics such as dynamic grain growth and cavitation during superplastic deformation, shear test on a superplastic sheet specimen is worth investigating. Typical shear test methods for metallic materials are summarized in Ref. 1. Except for the torsion test method, which imposes a pure shear-state in tube or bar specimens, most other test methods provide a mixed stress-state in the plane of the sheet. The torsion test has certain limitations. For solid bars in torsion, stress and strain are non-uniformly distributed in the specimen. For hollow tubes in torsion, plastic instability occurs at low strain, thus limiting its usefulness for large plastic strains. Furthermore, these methods are unsatisfactory for sheet materials since producing a tube from a sheet would change its properties. A planar simple shear test method has been used here to learn about the mechanical behavior of sheet specimens. This test has shown some promise for determining the flow stress at large strains [2-5], which is not always possible in the uniaxial tension test due to the effects of strain localization, failure, and cavitation occurred.

The simple shear deformation of a parallelepiped solid can be defined, as illustrated in Fig. 1, by the relative displacement of parallel T planes along the shear direction (L axis). The engineering shear strain is expressed by the ratio $\gamma = \Delta\ell / w$ where $\Delta\ell$ is the relative displacement of the parallel facing T planes of the parallelepiped and w is the width of the sample. The shear stress is defined by $\tau = F/(\ell t)$ where F is the shearing force applied to the T planes and ℓ is the length of the parallelepiped and t is its thickness. Several requirements for the test specimen geometry for the planar simple shear test are recognized [4, 5]. To achieve a homogeneous stress-state, the length (ℓ) to width (w) ratio is made as large as possible ($\ell/w > 15$), which minimizes the

contribution of the end effect (free edge deviates from a state of shear) on the gage section. To avoid buckling during the simple shear test, the ratio of the thickness (t) to the width needs to be as large as possible ($t/w > 0.44$).

To evaluate the mechanical behavior in shear, the measured load vs. displacement data must be analyzed to determine the effective stress and effective strain in order to compare with different deformation mode data such as uniaxial tension data. For the case where the material obeys Mises yield behavior, the effective stress - effective strain curves (according to Mises equation) should coincide for the various deformation modes. However, the Mises effective stresses based on τ in the stress - strain curves obtained from such simple shear tests and torsion tests have been shown to be lower than those under uniaxial tension [4-6]. In addition, the rate of strain hardening is sometimes reported to be lower in shear than that in uniaxial tension at room temperature [6]. There are two possible physical explanations for these differences. First, the material is no longer isotropic and Hill's proposal [7] is not applicable, or the evolving dislocation structure and its associated deformation resistance depend on deformation modes and not truly equivalent*. Another possibility for high temperature deformation is that the deformation behavior in uniaxial tension is not the same as that in shear [10].

Tome *et al.* [6] had proposed that at least at room temperature the number of active slip systems for different deformation modes could be different, e.g. the number of slip systems in uniaxial tension is higher than that in shear. They claimed that this results in different texture evolution under in the different deformation modes, although texture change due to small deformation is not expected to be large. For fine grain superplastic materials at $0.95 T_m$, texture evolution does not occur (unless the starting alloy is recrystallized). In fact, in most cases the recrystallized texture after thermomechanical processing becomes less intense after superplastic deformation [11-13] due to grain

* It has been shown that isotropic hardening does not prevail for multiaxial strain-states [8, 9].

rotation and grain boundary sliding (GBS). This leads to only a minor dependence of mechanical response on the deformation mode.

Grain movements associated with rotational and translational displacements of grains have been observed in fine grain superplastic materials deformed under uniaxial tension [14]. Due to these processes of grain rotation and GBS, shearing occurs through the specimen thickness as well as along the specimen face, and a new surface continuously emerges. On the other hand, in planar shear test, the process of shearing through the thickness direction cannot occur as observed during torsion test by Mayo and Nix [10]. That is, grain rotation and GBS of individual grains do not occur through the specimen thickness in shear. New surface also does not emerge at least on an overall scale during shear deformation. This different deformation behavior between uniaxial tension and shear may lead to different levels of stress.

The planar simple shear displacement is composed of a pure shear displacement plus a rotation as illustrated in Fig. 2. This creates continuous rotation of the principal strain axes 45° to the direction of displacement during deformation. This rotation creates continuous changes of strain path in each material element and it is well known that strain path changes affect the stress-strain curves [8, 9]. The comparison of stress-strain curves for shear and tension thus becomes complicated.

In this study, the planar simple shear tests were conducted at elevated temperatures under controlled strain-rate. Test temperatures were 500°C ($\sim 0.9 T_m$ where T_m is the material's melting temperature in absolute scale) and 550°C ($\sim 0.96 T_m$). Test specimens having a ℓ/w ratio of 7.6 and a t/w ratio of 0.22 were utilized. This is the first known characterization of shearing response under superplastic conditions. Attempts to convert the measured load vs. displacement curves from such tests into the shear stress vs. shear strain relationships (and then into the effective stress vs. effective strain relationships) were carried out with possible approximations regarding the prevailing strain-state in the specimen gage which will be discussed later. Comparison with the

effective stress vs. effective strain curves determined by uniaxial tension tests has also been made.

2 DESCRIPTION OF TEST METHOD AND EXPERIMENTAL PROCEDURE

A planar simple shear device and test specimen geometry used in this investigation are shown in Fig. 3. The shear displacement was imposed on the gage section through the axial displacement of the Instron testing machine. As shown in Fig. 3(a), a narrow groove machined into the sheet, parallel to the pulling direction, serves as the gage section. The grip sections, located on the opposite sides of the gage are pulled in directions opposite to one another. The gage section has a uniform reduced thickness of $t = 0.62\text{mm}$, which is approximately one-third of the grip thickness. The length ($\ell = 20.9\text{mm}$) to width ($w = 2.75\text{mm}$) ratio (ℓ/w) was 7.6, and the thickness to width ratio (t/w) was 0.22. The grip blocks are designed with serrated faces and through holes for pin loading, and aligned along the center line of the gage section. The sample's grip sections are firmly clamped by tightening bolts on the serrated grip blocks to prevent slippage during test. To prevent rotation of the sample, grip blocks had side restraint bars having frictionless sliding segments which ensure relative displacements of the specimen edges only in the pulling direction. The lateral forces on the bars are mutually self-compensating. The shearing process causes lengthening of horizontal lines scribed on the specimen gage along their displaced direction but resists shrinkage of lines at 90° to the displaced direction, i.e. it produces compression (Fig. 3(b)).

The material used in this study was a fine grain aluminum alloy containing 4.7%Mg, 0.8%Mn, and 0.4%Cu by weight. The evaluation of its mechanical behavior under uniaxial tension has been reported elsewhere [15-18] and used here for comparison. The initial linear intercept grain size (averaged over three orthogonal directions) of this alloy was $8\mu\text{m}$.

Tests were performed under a constant crosshead speed (CHS) in the range of 0.286 to 2.86 (mm/min) at a temperature of 550°C (which is the near optimum temperature for superplasticity in this alloy [15, 16]). This produces a constant rate of shear in the gage section. During the test, the specimen and load train were surrounded by a clamshell furnace having three heating zones independently controlled to maintain temperature within $\pm 1^\circ\text{C}$ of the test temperature over a 130mm length.

The test was interrupted after a crosshead displacement ($\Delta\ell$) of 4mm which corresponds to $\Delta\ell/w = 1.45$. Fig. 3(c) shows the specimen deformed to this level at a CHS = 2.86 (mm/min) and $T = 550^\circ\text{C}$. Overall, the specimen gage was found to be uniformly deformed and no buckling is observed. However, the ends of the specimen gage become curved with a convex (bulging) curvature. The thickness at the edges is also found to be slightly reduced after deformation. The free edges cannot maintain a compressive constraint and thus the compressive push from the interior bulges out the edge as the strain-state changes from shear toward tension in this region. Micrographs of the specimen in the L-T plane taken from the center of the shear region and the grip region are also shown in Fig. 3(d). Since the imposition of a state of shear is associated with lengthening along a diagonal direction defined by displacement, $\Delta\ell$, the morphology of grains closely follows this macroscopic shape change. The grain size in the gage region is found to be slightly larger than that in the grip region.

3 RESULTS AND DISCUSSION

Load vs. displacement curves for three different displacement rates at 550°C are shown in Fig. 4. Load continuously increases with increasing displacement unlike the behavior in uniaxial tension where cross section continuously decreases. Thus shear test provides a more direct information on the material's strain hardening response without many assumptions about necking or volume constancy. Deformation load also shows

strong dependence on deformation rate. The load vs. displacement data were analyzed to determine the effective stress and effective strain in order to compare with the uniaxial tension data. If the material obeys Mises yield behavior, the effective stress - effective strain curves (per the Mises relation) should be the same for the two types of test. Approximations and simplifications for the prevailing stress-state in the shear test specimen were made, as illustrated in Fig. 5. The first approximation was to consider a simple-shear state over the entire gage region (Fig. 5(a)), with no special consideration for the rotation of the principal stress axes. The second approximation also ignored the rotation effect but made a correction for the bulged convex curvature at the free edges of the gage section, where uniaxial tension state prevailed (Fig. 5(b)). Another possible approximation based on a restoring moment might be considered. It might consist of a couple of shear forces (F) applied on the two end faces of the gage section (distance ℓ apart) creating a rotational moment $F\ell$, which must be counterbalanced by the forces exerted on the specimen grips [4]. The reaction forces might produce additional stresses perpendicular to the shearing direction (Fig. 5(c)).

During our tests, the specimen showed no tendency for rotation, nor any sign of intense plasticity near the edges where counteracting moment forces may be supposed to act. Rather the bulging effect was the only detectable effect which was observed. Thus the case of Fig. 5(c) is not analyzed further. The approximations of Fig. 5(a) and (b) are analyzed below, with labels I and II, respectively.

Approximation I: Simple-shear state in the entire gage region

Assuming a homogeneous simple-shear state in the entire gage region, the shear strain (γ) is $\Delta\ell/w$, and the shear stress (τ) is (F/A) . Using Mises criterion, the effective stress (σ_e) is $\sqrt{3}\tau$ and the effective strain (ϵ_e) is $\gamma/\sqrt{3}$.

Approximation II: Simple-shear state in the center region with approximate edge effects

The bulging of the ends of specimen gage arises due to loss of the compressive component of shear near the free edge. Without this constraint, the compression from the interior of the gage section can push material outward in the form of bent elements under tension. The shear resistance near the ends of the gage is therefore reduced considerably. The exact determination of the stress near the edge is difficult due to a varying stress-state near this region, leading to uniaxial tension along the free edge.

In stead of undertaking a finite element analysis of the whole specimen, an upper bound for the shear stress in the center portion of the sample may be obtained by ignoring the stresses supported by the ends. It is assumed that the edge effects which minimize the shear stress extend to a distance of $w/2$ from the edge following St. Venant's principle. G'sell *et al.* [4] showed deformed grid lines from the FEM analysis. The region of distortion near the edge spread over a distance of $w/2$ from the free edge. Thus the portion of the gage length carrying shear load can be assumed to be approximately over a length of $(\ell - w)$ rather than ℓ . This simplistic assumption can provide an upper bound estimate of the shear stress,

$$\tau = \frac{F}{(\ell - w) \cdot t} = \frac{F}{\ell t} \left(\frac{1}{1 - (w/\ell)} \right) \quad (1)$$

and the shear strain in the center region is simply $\Delta\ell/w$.

$\sigma_e - \epsilon_e$ curves calculated based on approximations I and II at three different effective strain-rates are given in Fig. 6 at test temperatures of (a) 500°C and (b) 550°C, together with the uniaxial tension data. The upper bound approximation of effective stress (approximation II) calculated from Eq. (1) with $w/\ell \cong 0.13$ is about 15% higher than that for the simpler approximation I. However, if the specimen geometry is

improved (i.e. a ratio w/ℓ is made much smaller), errors from the edge effect would be minimized.

It should be noted in Fig. 6 that the peak in stress shown for uniaxial tension is artificial. The loss of cross sectional area, gradual necking, and internal cavitation cause measured stress to decrease during the tensile test. These problems are almost nonexistent in a shear test. Thus the tensile curve is extrapolated beyond the peak to represent a situation closer to reality. Overall the stress vs. strain curve (based on Mises effective stress vs. effective strain) for the planar simple shear test lies below that for the uniaxial tension test. At a low effective strain rate of $1 \times 10^{-4} \text{ s}^{-1}$, the effective stress in shear is reasonably close to that for uniaxial tension. The difference between these curves increases with increasing strain-rate. The slope of the uniaxial stress-strain curve extrapolated from its early part does match that from the shear test thereby suggesting that the rate of strain hardening is not influenced much by the change in stress state.

Strain hardening in fine grain superplastic materials mainly stems from dynamic grain growth [17]. Fig. 7 shows the average grain size plotted as a function of effective strain in uniaxial tension and in shear at 550°C . Average grain size was determined by the cube root of the linear intercepted grain size measured along each of the three orthogonal directions. The three directions were L, T, and S directions in uniaxial tension. In shear (Fig. 2), two orthogonal directions in the L-T plane were the directions of elongation (e.g. the direction of arrow) and contraction. The average grain size is found to increase linearly with increasing strain. Lower strain-rates produce the larger grain sizes at a fixed effective strain. Average grain sizes are found to be approximately the same at a fixed effective strain for both uniaxial tension and shear.

It is postulated that the continuous rotation of stress axes may be beneficial to the ease of grain boundary sliding process. If grain boundary sliding is retarded by blocking grains, the rotation of stress axes can initiate sliding on additional "free" boundaries or initiate slip on additional planes as well as enhanced climb process. At high strain-rates

this softening response may provide a more marked drop in the stress level. But the effect may be little at the lower strain-rates where grain boundary sliding is already substantial in magnitude.

In addition to the study of mechanical response, microstructural evolution characteristic such as cavitation in shear is worth investigating. Fig. 8 shows micrographs of cavities for an Al-Mg-Mn-Cu alloy deformed (a) under uniaxial tension and (b) shear to an effective strain of 0.84 at $\dot{\epsilon}_e = 1 \times 10^{-2} \text{s}^{-1}$ and $T = 550^\circ\text{C}$ in the L-T plane. A significant difference in void size is observed between uniaxial tension and shear. For uniaxial tension, cavities larger than $20 \mu\text{m}$ in diameter are found at several locations, but for shear, voids are smaller (less than $5 \mu\text{m}$). For the shear test, the number of cavities is also found to be smaller. That is, the level of cavitation in shear is less than that in uniaxial tension. The degree of cavitation is affected by the imposed stress-state [18-21]. The lower level of cavitation in shear is due to the lower value of the mean stress (σ_m / σ_e) under shear where σ_m is the mean stress (see Ref. 18 for the effect of the stress-state on cavitation).

4 SUMMARY AND CONCLUSIONS

In the present study, planar simple shear tests were conducted on superplastic Al-Mg alloy sheets at test temperatures of $0.9 T_m$ and $0.96 T_m$. The samples were designed with a ℓ/w ratio of 7.6 and a t/w ratio of 0.22, which gave some departure from simple shear state near edges. Nevertheless the results provide interesting insight into the level of flow stress and strain hardening behavior under different deformation modes. Effective stress vs. effective strain relationships were developed from the measured load - displacement data utilizing the simple shear approximations, and compared with the uniaxial tension data. The primary conclusions of this study are listed below:

1. The stress vs. strain curve (based on Mises effective stress vs. effective strain) for planar simple shear test lies below that for the uniaxial tension test. This difference cannot be fully resolved by correcting for the end effects.

2. The incremental rotation of the principal strain axes and specimen-end effects arising during deformation in planar simple shear tests complicate the determination of true mechanical response. It is possible that continuous rotation of the principal stretching direction during shear test assists in the grain boundary sliding process and helps to keep the stress level lower than in tension.

3. Strain hardening in such an alloy mainly stems from dynamic grain growth. The rate of strain hardening at a fixed effective strain-rate is essentially the same for both uniaxial tension and shear, thereby suggesting that the rate of strain hardening is not influenced much by the change in stress-state under superplastic conditions.

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Fig. 1. Definition of the reference axes and dimensions of a parallelepiped solid deformed under planar simple shear.

Fig. 2. Illustration showing that (a) simple shear is related to pure shear by a rotation, leading to the rotation of principal strain axes, 1 and 2, as shown in (b).

Fig. 3. (a) The geometry of a planar simple shear test specimen (undeformed). (b) Schematic drawing of the shearing device, which consists of two rigid holders subjected to a parallel displacement and clamping holders to compress the lateral parts of the sample. (c) A specimen deformed to $(\Delta l / w) = 1.45$ at $\text{CHS} = 2.86 \text{ mm/min}$ and $T = 550^\circ\text{C}$. (d) Micrographs of deformed specimen gauge region and undeformed grip region in the L-T plane for an Al-Mg-Mn-Cu alloy. Grains are generally aligned along the direction of arrow in the deformed specimen gage.

Fig. 4. Load vs. displacement ($\Delta\ell$) curves obtained from the planar simple shear tests (interrupted at $\Delta\ell = 4$ mm) for three different displacement rates at 550°C in an Al-Mg-Mn-Cu alloy. Load shows continuous increase with increasing displacement.

Fig. 5. Diagrams illustrating the three different approximations used to analyze the deformation in a planar simple shear specimen gage: (a) a simple shear-state in the entire gage, (b) a simple shear-state in the center region with the end effect (convex bulging curvature), and (c) a simple shear-state with normal stresses acting on the shear plane to prevent rotational moment.

Fig. 6. The effective stress - effective strain curves (according to Mises equation) obtained based on approximations I and II, together with the uniaxial tension data, at test temperatures of (a) 500°C and (b) 550°C for an Al-Mg-Mn-Cu alloy.

Fig. 7. Average grain size plotted as a function of effective strain in uniaxial tension and in shear at 550°C. Average grain size was determined by the cube root of the linear intercepted grain size measured along each of the three orthogonal directions. The three directions were L, T, and S directions in uniaxial tension. In shear (Fig. 2), two orthogonal directions in the L-T plane were the directions of elongation (e.g. the direction of arrow) and contraction.

(a) Uniaxial Tension

(b) Shear

Fig. 8. Optical micrographs of cavities at an effective strain of 0.84 in the L-T plane for a superplastic Al-Mg-Mn-Cu alloy. Tests were conducted at an effective strain rate = 10^{-2}s^{-1} and $T = 550^{\circ}\text{C}$ under (a) uniaxial tension and (b) shear.

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