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STEADY STATE CREEP IN Pb-Sn EUTECTIC ALLOYS

Dionysios Ioannis Grivas
(M.S. Thesis)

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STEADY STATE CREEP IN Pb-Sn EUTECTIC ALLOYS

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ABSTRACT

Steady state creep rates were measured for the superplastic lead-tin eutectic alloy (Pb-62Sn). Tests were performed on samples of grain size from 2.8 μm to 9.9 μm over a range of temperatures from 0°C to 160°C and strain rates from $1.8 \times 10^{-7}$ to $1.33 \times 10^{-2}$ sec$^{-1}$. The alloy exhibited three different regions of constant value for the stress exponent $n$. The data were consistent with the assumption that the creep behaviors occur simultaneously via independent mechanisms. At high stresses, conventional creep was found to be the dominant mechanism while at intermediate stresses it was superplastic creep. At low stresses against the general belief a non-diffusional mechanism was found to control the creep rates of this alloy. The equation

$$\dot{\gamma} = A \tau^* d^* \exp(-\Delta H^*/RT)$$

proposed by Dorn can represent the results for this superplastic alloy very satisfactorily. Different sets of values for the constants $A$, $n$, $m$ and $\Delta H^*$ were obtained for each region.
I. INTRODUCTION

No theory exists today that can fully explain the creep deformation of superplastic alloys. Until the mechanisms for this deformation are fully understood a precise constitutive equation cannot be written. It has been shown\textsuperscript{1-4} that the semi-empirical equation proposed by Dorn and his associates can represent the steady state creep behavior of a number of important systems. This equation may be written\textsuperscript{3} in the dimensionless form

$$\dot{\gamma}^* = A\tau^* d^{-m} \exp\left(-\frac{\Delta H^*}{RT}\right)$$

(1)

$\dot{\gamma}^*$, $\tau^*$, $d$ are as defined in the nomenclature, the dimensionless shear strain rate, shear stresses and grain size, respectively; $\Delta H^*$ is the activation energy for creep and $n$, $m$ and $A$ represent constants. Most of the suggested constitutive equations for high temperature creep can be drawn from Eq. (1) through proper choice of the constants $n$, $m$ and $A$.

Various authors\textsuperscript{5-9} have suggested that the stress exponent ($n$) is a continuous function of the applied stress $\tau^*$, while others\textsuperscript{3,10,11} have suggested that over a range of stresses, $n$ is constant depending on the applied stress $\tau^*$ only in the transition region. In each region of constant $n$, it has been hypothesized a different mechanism dominates the steady state creep.\textsuperscript{3,10,11}

Bird, Mukherjee and Dorn\textsuperscript{3} support Eq. (1) with an extensive compilation of data on the steady state creep of metals and simple alloys. They further conclude that Eq. (1) will also represent the steady state creep behavior of superplastic materials (see Refs. 11, 13 and 14 for reviews in superplasticity). They specifically suggest that as $\tau^*$ is decreased
a superplastic material will pass through three distinct regions of steady state creep. In each one a particular variant of Eq. (1) will be obeyed. (1) When $\tau^*$ is sufficiently large they predict a "conventional" steady state creep controlled by a dislocation climb mechanism. Hence, they anticipate a stress exponent in the range 4-7, an apparent activation energy $\Delta H^*$ of the order of that for self diffusion and a creep rate independent of grain size ($m = 0$). (2) For smaller $\tau^*$ they suggest a region of well defined "superplastic" creep. On semi-empirical grounds they anticipate $n = 2\Delta H^*$ of the order of that for grain boundary diffusion and an inverse square grain size dependence. (3) At even lower stresses they predict a behavior dominated by Coble creep which involves atom movement through the grain boundaries with an activation energy of the order of grain boundary diffusion, an inverse cube grain size dependence and a stress exponent equal to one.

This low stress creep region is controversial. Many authors expect a diffusional process to be the dominating mechanism. This diffusional mechanism can be either Coble creep as discussed earlier, or Nabarro creep which involves atom movement through the grains with an activation energy of the order of that for self diffusion, a stress exponent ($n$) equal to one and an inverse square grain size dependence. While evidence has been reported of the Coble creep in Zn-Al eutectoid others have disputed altogether the idea of diffusional creep on experimental grounds.
Bird et al. have analyzed Avery and Backofen's Pb-Sn eutectic and Ball and Hutchison's creep data on Zn-22Al eutectoid by different variants of Eq. (1). They argue that both alloys show clear regions of conventional and superplastic creep. They also found evidence of Coble creep in the Zn-22Al but were unable to establish its existence for the Pb-Sn eutectic alloy due to unavailability of data. Recently both Vaidya, Murty and Dorn and Misro and Mukherjee have reinvestigated the steady state creep of Zn-22Al. Both sets of investigators claim verification of the Bird, Mukherjee-Dorn representations. There is, however, no comparable data for the Pb-Sn eutectic alloy. While several investigators have studied the steady state creep of this alloy, no single investigation contains enough information to evaluate all the parameters in Eq. (1). A superposition of data would be of dubious value since the results of these investigations are not consistent with each other. Also limited data in the low stress creep region on Pb-Sn eutectic reveals a stress exponent in this region much higher than 1, more in the order of 3. Such a value for \( n \) would exclude any of the above mentioned diffusional mechanisms from being the controlling mechanism in this region.

This study was undertaken to observe if Eq. (1) can represent the data for the superplastic Pb-Sn eutectic alloy and to establish if the three regions of constant \( (n) \), suggested by Bird et al., do exist for the above alloy. To fulfill this objective, the constants \( n \), \( m \) and \( A \) and \( \Delta H^* \) must be computed for each region. I will try to point out any relation of these values to already existing theoretical models to explain the creep behavior for each region.
II. APPROACH

Because there is no single machine in this laboratory that can precisely record all the creep rates required for this investigation, two different sets of experiments were performed. In one set the transition from the superplastic creep region to the conventional creep region was studied and another, the transition from the low stress region to the superplastic region was studied. The first set was performed on an Instron testing machine, while the second set was performed on a creep test machine. We will present the two experimental sets and discuss their results separately. In the discussion section I will bring them together and argue my objective.
III. STUDY OF THE TRANSITION FROM THE SUPERPLASTIC CREEP REGION TO THE CONVENTIONAL CREEP REGION

A. Experimental Procedure

Samples of the Pb-Sn eutectic alloys were cast from pure lead and pure tin (both 99.999) into an ingot of diameter 1 in. The ingot was then rolled into 7/8 in. diameter rods at room temperature. Specimens of the "double shear" type were machined from these rods. The specimens were annealed at 175°C to obtain different mean grain sizes, which were measured from optical or scanning electron micrographs using the mean intercept method. Specimens exhibited grain sizes of 7.8±0.4 µm after 1 day anneal, 9.9±0.2 µm after 10 days anneal, and 5.5±1 µm after an anneal of 21 hours.

The creep rates of these specimens were then determined at strain rates from 2.66·10^{-5} sec^{-1} to 1.33·10^{-2} sec^{-1} and temperature from 0°-160°C in an Instron testing machine. The low temperature tests were conducted in ice water, the room temperature tests in air, and the high temperature tests in an electrically heated oil bath. The bath temperatures were monitored with chromel-alumel thermocouples and were maintained constant to ±1°C.

B. Results

Apparent steady state creep was easily obtained at all temperatures and strain rates used in this series of tests. A logarithmic plot of the measured steady state strain rate as a function of the applied stress appears as in Fig. 1, which shows the data for the specimens of mean grain size ~9.9 µm. The curve divides naturally into two distinct regions: a high stress region with slope ~7 and a lower stress region
slope ~2. These regions of creep behavior seem associated with different dominant creep mechanisms. Samples tested in the high stress region show significant primary creep, while samples tested in the lower stress region do not; samples tested in the lower stress region show creep rates which depend markedly on grain size, while samples tested in the high stress region do not. Hence, assuming that Eq. (1) applies, we must anticipate that the two distinct regions of creep behavior will be governed by different variants of the equation. In the following we refer to the two types of creep behavior as "conventional" and "superplastic" creep.

1. The Conventional Creep Region

The steady state creep data in the high stress region showed no evidence of dependence on grain size. Hence, the exponent \( m \) must be set equal to zero in Eq. (1), leaving three independent parameters: The activation energy, \( \Delta H^* \), the stress exponent, \( n \), and the coefficient \( A \). These parameters should be determined independently. However, given the large value of the stress exponent (\( n \sim 7 \)) in conventional creep, we were unable to obtain enough data points at fixed \( \tau^* \) to permit an independent determination of \( \Delta H^* \). We, therefore, found values of \( n \) and \( \Delta H^* \) through a consistency procedure. Approximating \( n = 7 \), we approximated \( \Delta H^* \) using data taken at fixed strain rate, and then assumed this value of the activation energy and adjusted \( n \) to achieve the best representation of the data with an equation of the form of Eq. (1).

Specifically, if we assume \( n = 7 \), fix the strain rate, \( \dot{\gamma} \), and measure the required shear stress \( \tau \) as a function of absolute temperature \( T \), then it follows from Eq. (1) and the definitions of \( \dot{\gamma}^* \) and \( \tau^* \) given
in the Nomenclature that $\Delta H^*$ is simply related to the slope of a plot of $\ln \tau^7/(G^6 T)$ vs $1/T$. Such a plot is shown in Fig. 2. The data obtained with samples of all three grain sizes essentially coalesce. The slope of a least-squares line faired through this data gives $\Delta H^* = 19.4 \pm 0.4$ kcal/mole as an estimate of the activation energy for creep. This activation energy is approximately 3 kcal/mole less than that for creep in pure tin and pure lead (both have $\Delta H^* \sim 23$ kcal/mole),\(^1\) and is less than the activation energy for self-diffusion by about 5 kcal/mole for pure lead and 4 kcal/mole for pure tin.\(^1\)

Given the activation energy $\Delta H^*$, if Eq. (1) is valid the data will coalesce onto a straight line in a plot of $\ln \gamma^* \exp(\Delta H^*/kT)$ against $\ln(\tau^*)$. The line will have slope $n$ and intercept $A$. Such a plot is shown in Fig. 3. The data do essentially coalesce. A least squares line then yields $n = 7.1 \pm 0.3$ and $A = 1.3 \times 10^{15}$. These values are comparable to those obtained by Mohamed, et al.\(^1\) for creep of tin over roughly the same range of $\tau^*$: $n = 6.5$ and $A = 3.6 \times 10^{15}$.

Hence, as demonstrated in Fig. 3, conventional creep of the lead-tin eutectic alloy may be represented by an equation of the form (1):

$$\gamma^* = 1.3 \times 10^{15} (\tau^*)^{7.1} \exp(-19,400/RT)$$

(2)

2. The Superplastic Region

At lower stress $n = 2$, and hence the Pb-Sn eutectic satisfies the conditions for superplastic creep.\(^{14}\) Creep behavior in the superplastic region shows evident grain size dependence. Assuming a constitutive equation of the form of Eq. (1), the coefficient $A$ and the exponents $n$, $m$ and $\Delta H^*$ must be determined.
Assuming Eq. (1), an independent determination of the activation energy $\Delta H^*$ may be made from the slope of a plot of $\ln \gamma T$ against $1/T$ at constant applied stress and grain size. Plots for each of the grain sizes used in this research are shown in Fig. 4. Least-squares lines through these points yield the data appearing in Table I, and gives an average value of $\Delta H^*$ of 11.5 kcal/mole. This value is identical to that reported by Bandelet and Suery,\textsuperscript{9} by Cline and Alden.\textsuperscript{6} The activation energy for creep is about 0.5 of that for self-diffusion in Sn,\textsuperscript{1} and hence, is of the right order of magnitude to be interpreted as an activation energy for grain boundary diffusion, as suggested by Bird, Mukherjee, and Dorn.\textsuperscript{3}

One should, however, note that the data plotted in Fig. 4 show an apparently consistent curvature, which indicates either that $\Delta H^*$ is a slightly increasing function of temperature, or that it depends on some other experimental variable which is a function of temperature. The latter point of view is more consistent with available data. The values of $\Delta H^*$ obtained by Bandelet and Suery\textsuperscript{9} and by Cline and Alden\textsuperscript{6} are identical to those we found, but were taken over different temperature ranges: -44°C to 19°C in the work of Bandelet and Suery,\textsuperscript{9} and 0°C to 80°C in the work of Cline and Alden.\textsuperscript{6} While previous work on the Pb-Sn eutectic has not been reported in sufficient detail to compare this observation, a similar increase of the activation energy with temperature is apparent in the data of Ball and Hutchison\textsuperscript{18} and of Vaidya, Murty and Dorn\textsuperscript{4} on the superplastic creep of the Zn-Al eutectoid. It was not possible to study the temperature dependence of $\Delta H^*$ in detail in the present work. The reported value should be regarded as an average over
a range of experimental temperatures.

Given a value for the activation energy, $\Delta H^*$, Eq. (1) predicts that the data for superplastic creep of samples of given grain size will coalesce into a straight line of slope $n$ in a plot of $(\gamma^* \exp(\Delta H^*/RT))$ against $(t^*)$. This coalescence is illustrated in Figs. 5 and 6. In Fig. 5, we have plotted the steady-state creep data obtained with samples of $d = 5.5\mu m$ and $d = 7.8\mu m$ at various test temperatures. The plot shows that, in the superplastic region, the data nearly superimpose on a line of slope close to 2.0 for each grain size. The transition to the conventional creep mechanism is also apparent. The separation of the creep curves at higher stress is due to the approximate doubling of the apparent activation energy for creep which occurs when superplastic creep is superceded by conventional creep. In Fig. 6 we compare the superplastic data obtained with samples of $d = 9.9\mu m$ with the curves shown in Fig. 5. The data for the larger grain size may also be represented by a straight line of slope $n-2$ in the superplastic region. A least squares analysis of the data for each grain size in the superplastic region revealed the results shown in Table I. From these values an average slope $n = 1.95\pm0.23$ was obtained, in essential agreement with the estimate $n = 2$. From Table I it can be noticed that there is no apparent dependence of $n$ and $\Delta H^*$ on the grain size.

The grain size dependence of superplastic creep is apparent in Fig. 6. Given Eq. (1) the value of the exponent $m$ can be computed in either of two ways: (1) a plot of the logarithm of the temperature-compensated strain rate $(\gamma^* \exp(\Delta H^*/RT))$ against the logarithm of the mean grain diameter $(d^*)$ at constant stress should yield a straight line
of slope \((m)\), while (2) a plot of \(\ln(\tau^*/G)\) vs \(\ln(d^*)\) at a constant value of the temperature compensated strain rate should yield a straight line of slope \((m/n)\). The plots are shown in Fig. 7. Both yield \(m = 1.8\), in rough agreement with the suggested value \(m = 2\).

Finally, given Eq. (1), a logarithmic plot of the strain rate compensated for temperature and grain size, \(\dot{\gamma}^*(d^*)^m \exp(\Delta H^*/RT)\), against \(\tau^*\) should yield a straight line of slope \(n\) and intercept \(A\). The plot is shown in Fig. 8. The value obtained for \(A\) is 900, and for \(n\) is 1.97±0.03.

We, hence, concluded that the superplastic creep of these samples of Pb-Sn eutectic may be represented by a constitutive equation of the form (1):

\[
(\dot{\gamma})^* = 900(\tau^*)^2 (d^*)^{-1.8} \exp(-11.5/RT)
\]
IV. STUDY OF THE TRANSITION FROM THE LOW STRESS CREEP REGION TO THE SUPERPLASTIC CREEP REGION

A. Experimental Procedure

Pb-62Sn eutectic alloy was prepared from 9.99% pure tin and 9.99% pure lead. The alloy was cast in a graphite crucible to 2.5 in diameter, and reduced to 7/8 diameter rods by a sequence of reductions at -44°C. From these rods double shear specimens were machined. Annealing the specimens at 170°C for 1.25, 7, 12 and 17 hours resulted in 2.8 μm, 4.3 μm, 5.8 μm, and 6.7 μm grain size respectively. These grain sizes were determined from scanning electron micrographs by the mean intercept method. The specimens were kept at -40°C at all times prior to testing to prevent extensive grain growth at room temperature. Creep tests were performed on a constant load creep machine. A silicon oil bath was used to control the temperature, which was maintained constant to ±1°C. One specimen was used to obtain all the data for every temperature in a particular grain size, except the 6.7 μm where 2 specimens were tested at the same temperature.

B. Results

Tests were performed over a temperature range of 98°C to 168°C and a stress range from 2.2 psi to 468 psi.

Figure 9 is a plot of the measured strain rate \( \dot{\gamma} \) against the applied stress for the 5.7 mm grain size at various temperature. For each temperature the data naturally break into two distinct regions of different slope. The higher stress creep region has a slope of \( \sim 2 \); the low stress creep region has a slope of \( \sim 3 \). Indications of this behavior have already been reported elsewhere. Given \( n \sim 2 \), the higher
stress creep region fulfills the condition for superplastic behavior, and will be referred to as the superplastic region. Fig. 10 is a typical plot for the computation of the steady state strain rate. As can be seen from this plot primary creep is present in the low stress region tests but was not found in the superplastic region. These regions, hence, seem associated with different controlling mechanisms. Assuming that Eq. (1) applies to each one of these two regions, by choosing the proper value for m, n, A and \( \Delta H^* \) we can obtain constitutive equations that describe the creep behavior of this alloy.

In the next paragraphs the parameters for each of the two regions will be computed.

1. **Low Stress Region**

   Examining Eq. (1), it can be seen that the activation energy for creep deformation in this region at constant stress and grain size may be found from the slope of the plot of \( \ln YT \) against \( 1/T \). A plot of this kind for the 5.7 \( \mu \)m specimen shown in Fig. 11. A slight curvature may be noticed. Previously, a similar curvature was found in a plot of the same kind for the superplastic region of Pb-Sn eutectic. We suggested then that \( \Delta H^* \) might depend on some experimental parameter which is a function of temperature. While in the previous study the \( \frac{\partial (\ln YT)}{\partial (1/T)} \) was found positive in the present case it is negative. Previous work on Pb-Sn eutectic has not been reported for this region in sufficient detail to compare this observation. A least squares analysis through the experimental points revealed an average activation energy equal to 18.9±0.5 kcal/mole. This value is equal within experimental error to the value 19.4±0.4 kcal/mole we obtained for the conventional creep
at higher stresses. This value is of the order of the activation energy for self diffusion.

Once the activation energy has been computed, one can make a composite plot of $\ln \gamma^* e^{\Delta H^*/RT}$ against $\ln \tau^*$, compensating for the temperature dependence of the strain rate $\gamma^*$ to the applied stress $\tau^*$. Such a plot is shown in Fig. 12 for the 5.7 μm grain size. The points of all temperatures essentially coalesced to give a straight line of slope equal to $3.0 \pm 0.1$. Figure 13 is the same kind of plot for the other 3 grain sizes studied in this investigation. Three parallel lines can be seen, each giving the behavior for a specific grain size.

Judging from Fig. 13, there seems to be a grain size dependence of the creep rate at constant load. The power of this dependence can be obtained from a plot of $\ln \gamma^*$ against $\ln d$ at constant $\tau^*$. This plot is shown in Fig. 6. A value of $2.1 \pm 0.1$ is obtained, indicating a grain size dependence $\sim d^{-2}$. To complete the determination of the constitutive equation we require values for $A$ and $n$. These can be obtained from a plot of strain rate against stress, compensating for both the temperature and grain size dependence. Such a plot is shown in Fig. 7 where $\ln \gamma^* d^n e^{\Delta H^*/RT}$ against $\ln \tau^*$ has been plotted. The fit seems good; datum points for all temperatures and grain sizes essentially coalesced to give a straight line. A least squares analysis through these points revealed a value for $n = 3.0 \pm 0.1$ and for $A$ an average value of $3.2 \times 10^{12}$.

A constitutive equation for the low stress creep region may now be written in the form of Eq. (1):

$$\gamma^* = 3.2 \times 10^{12} \tau_d^{3.1-2.1} e^{18900/RT}$$

(4)
2. Superplastic Region

Data gathered in the superplastic region yielded essentially the same activation energy and stress exponent as reported in the previous section, where a more detailed discussion may be found.

Figure 16 is a plot of ln T vs 1/T for the computation of the activation energy for the creep deformation in this region. A value of 12.2±0.3 kcal/mole was obtained. This value compares with the value of 11.5 kcal/mole obtained in. A temperature compensating plot for the 5.7 µm grain size of \( \ln \gamma^* \exp(\Delta H^*/RT) \) against \( \ln T^* \) is shown in Fig. 4. Data at all four temperatures come together to give a straight line of slope 2.0±0.1. Figure 10 is a similar plot for the three other grain sizes. Again, three parallel lines can be observed, each one corresponding to a particular grain size.

The specimen of this alloy exhibited rapid grain growth at the test temperatures used. In this investigation the applied loads were increased during testing to obtain strain rates in the superplastic region at higher values of the applied stress. In the previous investigation the initial loads applied were in the superplastic region. Since grain growth occurred during testing, the actual sample grain size for tests in the superplastic region is uncertain. Hence, we could not obtain a valid independent measure of the grain size dependence. Assuming \( m = 2 \) and \( \Delta H^* = 11.5 \), a least squares analysis through the points given by plotting \( \ln \gamma^* d^m \exp(\Delta H^*/RT) \) against \( \ln T^* \) revealed a value for A = 5000 and for n=2.1±0.1. This value for A is much higher than the value 209 reported previously against our expectations that an increase in grain size would decrease the strain rate. This shift of the A value may be attributable
to any one of three factors: (1) A difference in purity between the specimen in the two investigation, (2) the difference in testing methods, described above, and (3) the difference in the preparation procedure to obtain the desired grain sizes. While in this study the specimens were reduced at -44°C from 2.5 in. in diameter to 7/8 in. in diameter in the work reported previously, the specimens were reduced at room temperature from 1 in. to 7/8 in. This severe reduction at low temperature gave smaller initial grain size. Annealing these specimens to obtain the mentioned grain sizes could result in more equiaxed grains yielding a higher creep rate at constant load. Microscopic observations tend to support the expectation of more equiaxed grains.
V. DISCUSSION

From Figs. 3, 8 and 15 it can be seen that Eq. (1) can represent the creep data for the superplastic Pb-Sn eutectic alloy very satisfactorily. Different values of the exponents \( n, m, \Delta H^* \) and \( A \) are required for each region. It has been shown that the high temperature steady state creep behavior of the Pb-Sn eutectic alloy at relative high stresses may be presented by Eq. (2) while at lower stress obeys Eq. (3). The values of the parameters appearing in these equations are roughly in agreement with those empirically suggested by Bird, Mukherjee and Dorn.\(^3\) In their viewpoint these equations represent simultaneous, independent creep mechanisms. A complete constitutive equation for the steady state creep of the lead-tin eutectic should then be obtained by summing the Eqs. (2) and (3):

\[
\dot{\gamma} = 900(\tau^*)^2 (d^*)^{-1.8} \exp(-11500/RT) + 1.3 \times 10^{15} (\tau^*)^{7.1} \exp(-19400/RT)
\]

The results of several independent creep experiments are compared with the prediction of Eq. (4) in Fig. 9. Although the fit of the 5.5 \( \mu \)m and 9.9 \( \mu \)m seems to be quite good, the 7.8 \( \mu \)m grain size deviates from the above equation in the superplastic region by a factor of 1.8. This is most likely due to an error in the grain size determination. The fit is very good in the conventional creep region where there is no grain size dependence. If \( m \) is taken as 2, then \( A \) becomes 209, and an equally good fit can be observed. The shape of the computed curves illustrates that the value of the right-hand side of Eq. (4) is completely
dominated by the larger of its two terms except over a narrow region of \((\tau^*)\) where the two terms have nearly the same value. Due to the change of the A value in the two studies, and the grain growth during testing, it is not possible to add Eq. (5), which represents the low stress creep behavior, to Eq. (4) and hence, complete the equation for the creep deformation of lead-tin eutectic for the stress range tested. It is of importance to investigate how A depends on the history of the specimen. If in fact the A value is a function of the specimen preparation technique the inconsistency of the results of the various investigators, as discussed in the introduction, may be explained. All the investigators were using different methods to obtain the desired microstructure.

Table II summarizes the results found for each of the three regions. The rate controlling step for the conventional creep region is generally believed to be the stress assisted climb of dislocations in the bulk metal, though the specific details of the mechanism remain in doubt.\(^3\) The present results seem to satisfy the conditions suggested for this mechanism to be the dominant at high stresses.

The theoretical basis for Eq. (3), the rate controlling step in superplastic creep, remains cloudy. Reasoning from data on superplasticity in aluminum-zinc eutectoid alloy, similar to the data reported here, Ball and Huchison\(^{18}\) suggested a model on the dissolution of dislocation pile-ups through dislocation climb in grain boundaries. While this model yields a constitutive equation similar to Eq. (3), it has been questioned on empirical grounds by Nicholson.\(^{22}\) Langdon\(^{23}\)
has proposed a model for grain boundary sliding in which sliding occurs by the movement of dislocations along, or adjacent to, the boundary by a combination of climb and glide. He suggested that the constants in Eq. (1) should take the values \( n = 2, \ m = 1 \) and \( \Delta H^* \) of the order of that for self-diffusion. The present results do not seem to agree with this model in the activation energy and grain size dependence.

As mentioned in the introduction, many investigators believe that the controlling mechanism at lower stresses than those in this superplastic region is a diffusional mechanism. The findings of (1) an activation energy of 18.9 kcal/mole, (2) a grain size dependence \( d^{-2} \), (3) primary creep and (4) an \( n \) value equal to 3, lead me to suggest that another mechanism must be involved.

In Fig. 19 I have plotted \( \ln T^* \) against \( \ln \gamma^* \) for the low stress creep region for the two extreme grain size. I have also estimated the rate of hypothetical Coble creep using equations given by Bird et al.\(^3\)

With the 2.8 \( \mu \)m grain size specimen, we tried to observe Coble creep but was unable to go to stresses low enough due to limitations of the apparatus. Although two of the points fall below the Coble creep the uncertainty in the precise form of the equation governing Cobble creep in this material preclude a conclusion that it does not occur. It is, however, conclusive that some mechanism other than those introduced by Coble and Nabarro controls the creep rate at stresses below those at which superplastic creep is observed.

Weertman\(^24\) has suggested a model for low stress creep deformation involving dislocation movement from subgrain boundaries. This mechanism leads to an \( n \) value equal to 3 and an activation energy of the order of
that for self diffusion, but involves no grain size dependence. The present results are in good agreement with this model except for the grain size dependence. However, the grain size dependence we observed may not indicate an explicit dependence on grain size, but rather an implicit dependence through some other parameter which varied with the grain size in our tests. Such a hidden parameter may be the dislocation density, subgrain boundary morphology, or other variables of microstructural state dependent on the annealing history of the specimen.

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**NOMENCLATURE**

The quantities appearing in Eq. (1) are defined as follows:

\[ \dot{\gamma}^* = \frac{kT}{D_0} Gb \] (a dimensionless strain rate).

\[ \tau^* = \frac{\tau}{G} \] (a dimensionless shear stress).

\[ d^* = \frac{d}{b} \] (a dimensionless grain size).

The symbols have the following meanings:

\( \dot{\gamma} \) = strain rate

\( k \) = Boltzmann's constant

\( T \) = absolute temperature

\( D_0 \) = a characteristic diffusivity, chosen equal to the pre-exponential

\[ D_0 \] in the diffusion equation for pure Sn, (0.08 cm\(^2\)/sec).\(^{15}\)

\( G \) = a characteristic stress, taken equal to the shear modulus of pure

Sn, (2×10\(^{11}\) (d/cm)).\(^{15}\)

\( b \) = a characteristic length, taken equal to the Burger's vector of pure

Sn (3.18 (A)).\(^{15}\)

\( \tau \) = resolved shear stress

\( d \) = mean grain diameter
REFERENCES


7. S. W. Zehr and W. A. Backofen, Trans ASM 61, 300 (1968).


11. O. D. Sherby and P. M. Burke, Prog. Mat. Sci. 13, 325 (1967).


<table>
<thead>
<tr>
<th>Grain Size</th>
<th>n Superplastic</th>
<th>$\Delta H^*_{\text{superplastic}}$</th>
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<tr>
<td>5.5 $\mu$m</td>
<td>2.06 ± 0.1</td>
<td>11.45 ± 0.11 kcal/mole</td>
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<tr>
<td>7.8</td>
<td>1.76 ± 0.12</td>
<td>11.83 ± 0.46</td>
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<tr>
<td>9.9</td>
<td>2.05 ± 0.06</td>
<td>11.15 ± 0.25</td>
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<tr>
<td></td>
<td>Low Stress</td>
<td>Superplastic</td>
</tr>
<tr>
<td>----------------</td>
<td>------------</td>
<td>--------------</td>
</tr>
<tr>
<td>$\Delta H^*$ (kcal/mole)</td>
<td>18.9</td>
<td>11.5</td>
</tr>
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<td>$n$</td>
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<td>2.0</td>
</tr>
<tr>
<td>$m$</td>
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<td>Primary Creep</td>
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TABLE II
FIGURE CAPTIONS

Fig. 1. Stress vs steady state creep rate of Pb-Sn eutectic at various temperatures for the 9.9 µm grain size, indicating the two distinct regions at different stress levels.

Fig. 2. Arrhenius plot of ln(τ^7/c^6T) vs 1000/T for the computation of the activation energy in the conventional creep region.

Fig. 3. Plot of Υ^* exp(ΔH^*/RT) vs τ^* for the conventional creep region revealing no grain size dependence and a slope n = 7.1.

Fig. 4. Arrhenius plot of lnY^T for the computation of the activation energy for superplastic creep vs 1000/T (Υ^T is related to Υ^* by a constant as can be seen from the definitions in the appendix).

Fig. 5. The logarithmic plot of the temperature compensated strain rate vs τ^* for the 5.5 µm and 7.8 µm grain size.

Fig. 6. The logarithmic plot of the temperature compensated strain rate vs τ^* for the 9.9 µm including the superplastic region of the 5.5 and 7.8 µm grain size.

Fig. 7. The effect of grain size on the strain rate and stress in the superplastic region.

Fig. 8. Plot of the strain rate compensated for temperature are grain size vs τ^* for all superplastic data.

Fig. 9. Stress vs steady state strain rate for 5.7 µm grain size diameter at various temperatures. Two distinct regions of steady state creep are revealed.

Fig. 10. A typical plot for the determination of the steady state creep rate. Notice the primary creep for the low stress creep in contrast to the absence of primary creep at higher stresses.
Fig. 11. Arrhenius plot of \( \ln \gamma T \) vs \( 1000/T \) for the computation of the activation energy for the low stress creep region (\( \gamma T \) is related to \( \gamma^* \) by a constant).

Fig. 12. Temperature compensated plots of \( \ln \gamma^* \exp(\Delta H^*/RT) \) against \( \ln T^* \) for the 5.7 \( \mu \)m specimen in the superplastic and low stress region.

Fig. 13. A logarithmic plot of the temperature compensated strain rate against \( \ln T^* \) for the 2.8 \( \mu \)m, 4.3 \( \mu \)m and 6.7 \( \mu \)m grain sizes.

Fig. 14. A plot of \( \ln \gamma^* \exp(\Delta H^*/RT) \) vs \( \ln T^* \) to determine the grain size dependence in the low creep region.

Fig. 15. A logarithmic plot of the strain rate compensated for temperature and grain size against \( T^* \).

Fig. 16. Arrhenius plot of the \( \ln \gamma T \) against \( 1000/T \) from which we compute the activation energy for the superplastic region.

Fig. 17. The logarithmic plot of the temperature compensated strain rate \( \gamma^* \exp(\Delta H^*/RT) \) against \( \tau^* \) for the 2.8 \( \mu \)m, 4.3 \( \mu \)m and 6.7 \( \mu \)m grain size.

Fig. 18. A plot of \( T^* \) vs \( \gamma^* \) as obtained experimentally and from Eq. (4).

Fig. 19. The low stress region data for the 2.8 \( \mu \)m and 6.7 \( \mu \)m grain size including the Coble creep region as introduced by Bird et al. \[ \gamma^* = 48(T^*)^{1/2} (d^*)^{-3} \exp(11500/RT) \].
Fig. 1
Creep Region

\[ Q = 19,400 \pm 417 \text{ cal/mole} \]
Fig. 3

$\gamma^* \exp \frac{\Delta H^*}{RT}$

$\tau^*$

$d, \mu m$
- $5.5$
- $7.8$
- $9.9$

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Fig. 4
Fig. 5
Fig. 6
Fig. 7
Fig. 8
Fig. 9
Fig. 10

\[ \gamma \times 1 \times 10^{-6} \text{ (in./in.)} \]

- \( T = 17.5 \text{ psi} \)
- \( T = 115 \degree \text{C} \)
- \( d = 5.7 \mu\text{m} \)
- \( T = 72 \text{ psi} \)
- \( T = 115 \degree \text{C} \)
- \( d = 5.7 \mu\text{m} \)
\[ Q_{\text{low stress}} = 18.9 \pm 0.5 \text{ kcal/mole} \]
\[ d = 5.7 \mu \text{m} \]
Low stress region

Superplastic region

grain size 5.7 μm

\( d = 5.7 \, \mu m \)

○ 168 °C

□ 138 °C

△ 98 °C

\( \gamma \) exp

\( \Delta H^* \)

\( \tau^* \)

Fig. 12
Fig. 13
Fig. 14
Fig. 15
$Q_{\text{superplastic}} = 12.2 \pm 0.3 \text{ kcal/mole}$
$d = 5.7 \mu\text{m}$
Fig. 17
Fig. 18
### Experimental Data

<table>
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<tr>
<th>d, μm</th>
<th>T, °C</th>
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<td>9.9</td>
<td>83</td>
</tr>
<tr>
<td>7.8</td>
<td>27</td>
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<tr>
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Calculated by Eq. 4

![Graph](image)

Fig. 19
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