COMPRESSION TESTING OF SUPERPLASTIC LEAD-TIN EUTECTIC ALLOY AT ROOM TEMPERATURE

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ABSTRACT

The deformation behavior of fine grained lead-tin eutectic alloy was investigated at room temperature using compression specimens at initial strain rates in the range 10^{-5} to 10^{-1} s^{-1}. The grain size of the tested specimens was in the range of 2.8 to 7.2 µm. The results show that the flow stress- strain rate relation could be divided into two distinct regions. A superplastic region (region II), which prevails at intermediate strain rates, has values of strain rate sensitivity, m and grain size exponent, p of 0.5 and 2.2, respectively. In dislocation creep region (region III) that dominates at high strain rates values of m and p are 0.11 and 1.6, respectively, and the apparent activation energy is 45 kJ/mol. Region I that sometimes appears at low strain rates was not observed because a threshold stress is usually not present in high purity alloys such as the one used in this investigation.

Keywords: *Lead-tin eutectic, Superplasticity, Compression tests, Dislocation creep, Effect of grain size, Strain rate sensitivity.*

الملخص

تمت دراسة سلوك التشوه في سبيكة الرصاص والقصدير الاوتيكتي ذات الحبيبات الدقيقة في درجة حرارة الغرفة باستخدام عينات ضغط تحت معدل انفعال ١٠$^{-٥}$ إلى ١٠$^{-١}$ لكل ثانية . كان قطر حبيبات العينات في المدى من ٢,٨ إلى ٧,٢ ميكرومتر . أظهرت النتائج أن العلاقة بين إجهاد الانسياب ومعدل الانفعال تنقسم إلى منطقتين . منطقة اللدونه الفائقة والتي تظهر عند معدلات الانفعال المتوسطة وفيها معامل معدل الانفعال m، وأس قطر الحبيبات p، تساوي ٠,٥ و ٢,٢ على الترتيب . ومنطقة تزحف الانخلاعات وتسود عند معدلات الانفعال العالية وطاقة التحفيز الظاهرية لها تساوي ٤٥ كيلو جول / مول وتكون قيمة المعاملات p،m لها تساوي ٠,١١ و ١,٦ على الترتيب. عند معدلات الانفعال المنخفضة لم تظهر في هذه الدراسة أي منطقة اخري وذلك لعدم وجود إجهاد حد الانفعال بسبب استخدام سبيكة عالية النقاء .

1. INTRODUCTION

The flow behavior of superplastic materials is usually described by:

$$\sigma = C\dot{\varepsilon}^{m} \tag{1}$$

where σ is the flow stress, $\dot{\varepsilon}$ is the strain rate, m is the strain rate sensitivity and *C* is a constant, which depends on the experimental conditions like temperature and grain size. The value of m for superplastic materials lies in the range of 0.3 to 0.9. This high value of m inhibits the catastrophic necking and leads to large relatively uniform tensile elongation of the order of~ 1000%. The necessary conditions for superplastic deformation (high m value) are: (1) high testing temperatures $\geq 0.5\ T_m$, where T_m is the absolute melting point of the material, and (2) uniform and stable equiaxed grain structure with fine grain size ≤ 10 µm; eutectic alloys satisfy this condition where the two phase structure prevents the excessive grain growth. Superplastic deformation mechanism usually appears at strain rates in the range $10^{-5} < \dot{\varepsilon} < 10^{-3}\ s^{-1}$. However at lower strain rates < $10^{-5}\ s^{-1}$ or higher rates > $10^{-3}\ s^{-1}$, m tends to decrease below a value of 0.2 indicating a change in the rate controlling process.

Mechanics of superplasticity of metals and alloys has been mainly studied by means of a tensile test in which a true constant strain rate or a constant displacement rate is imposed, and the steady-state flow stress is recorded. It was first suggested [Backofen et al., 1964] that in some experiments, the strain rate can be cycled during the test, so that a number of steady state flow stresses are obtained from a single specimen. The optimal superplastic deformation temperature, strain rate, maximum elongation, flow stress as well as strain rate sensitivity of the material are obtained through experiment. However, in practical applications, there are few cases similar to simple tension test but more are compressively formed products. Thus, it is beneficial to find the optimal parameters of superplastic deformation under compression testing and to compare them with those derived from tension tests. In addition, compression tests have the advantage of avoiding the problem of necking particularly at high strain rates. It is worth mentioning that other tests such as stress relaxation [Enikeev and Mazurski, 1995, Ha and Chang, 1999 and 2001] and creep experiments [Mohamed and Langdon 1975, Yan et al. 1994] are used to study the superplastic properties in lead-tin eutectic and other alloy systems.

In the present investigation, the superplastic characteristics of a fine-grained lead-tin eutectic alloy are examined by conducting compression tests at constant displacement rates at room temperature. The results will be compared with previous ones carried out under similar experimental conditions using tensile tests [Soliman, 1994 and 1995a].

2. EXPERIMENTAL WORK

The Pb-62% Sn eutectic alloy was prepared by melting in air high purity lead (Pb) and tin (Sn) using graphite crucible. Chill-cast ingots of 32 mm diameter were produced and then hot extruded at room temperature (~0.65 T_m) in a single reduction into 10 mm diameter rods. Compression cylindrical specimens of length to diameter ratio (L/D)=2 to 3, in accordance with ASTM standards E9 and E209, were machined from the extruded bars.

The grain size, d, of the as-extruded specimens as represented by the mean linear intercept of grain boundaries on optical micrographs, was 2.8 µm. In order to study the effect of grain size on flow properties, some specimens were annealed at 423 K for different times (1, 6 and 24 hours), which produced grain sizes of 3.9, 5.0 and 7.2 µm, respectively. The accuracy of grain size measurements was within ±5%.

Compression tests were performed at room temperature, using an Instron machine model 1197 operating at a constant rate of crosshead displacement of 0.02-200 mm/min (corresponds to initial strain rates of ~ 10^{-5}-10^{-1} s^{-1}). In compression testing, bearing surface friction causes barreling and hence a higher measured applied force. In order to get homogenous deformation, friction was minimized by lubricating bearing surfaces using PTFE (Teflon) thin sheets. The force-displacement curves were monitored on a strip-chart recorder and tests were run for sufficiently large strains > 1 to establish unequivocally the steady state behavior. In addition to constant velocity tests, velocity jump tests were performed by increasing or decreasing the crosshead speed on a single specimen. For each velocity change, enough strain was allowed to ensure that the steady state had been reached.

3. RESULTS

3.1. True stress-strain curves

The force displacements data were used to construct the true stress-strain curves at various initial strain rates. Figure 1 exhibits the true stress-strain curves for specimens of grain size of 2.8 µm (top) and 5.0 µm (bottom) as a function of initial strain rates. Inspection of these curves along with those for other grain sizes, which are essentially similar to those presented in Figure 1, reveals the following points. First, the true stress-strain curve does not exhibit an immediate steady state flow, but there is a region of significant strain hardening before reaching steady state. In the strain-hardening region, stress increases to reach a steady state value at strain of about 0.2 for low strain rates (low stresses) and small grain size. Second, at high strain rates (high stresses) and large grain size, flow stress increases and reaches a maximum (peak) value and then decreases to the steady state value at strain values > 0.2. Third, the steady state value of stress tends to slightly increase at high strains > 1 due to the increase in strain rate with decreasing specimen length.

The mechanical property data (maximum stress σ_m (MPa), steady-state stress σ_s (MPa) and strain rate $\dot{\varepsilon}$ (s^{-1})) for various grain sizes are listed in Table 1.

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Table 1: Mechanical Properties for Various Grain Sizes

Grain size, μm	2.8		3.9		5.0		7.2	
$\dot{\varepsilon}$, s^{-1}	σ_m, MPa	σ_s, MPa	σ_m, MPa	σ_s, MPa	σ_m, MPa	σ_s, MPa	σ_m, MPa	σ_s, MPa
$1.1x10^{-5}$	3.7	3.7	5.4	4.8	8.0	5.9	10.3	7.5
$2.8x10^{-5}$	5.5	5.5	8.0	7.8	12.1	9.8	16.5	12.5
$5.3x10^{-5}$	--	--	--	--	16.1	14.4	--	--
$1.1x10^{-4}$	9.6	9.6	17.9	13.4	19.1	15.4	23.6	18.9
$2.8x10^{-4}$	18.2	17.2	25.4	20.8	26.7	21.2	30.2	24.3
$1.1x10^{-3}$	29. 0	25.6	35.5	29.0	36.2	30.2	38.8	37.0
$2.8x10^{-3}$	36.6	31.8	40.0	36.8	41.5	37.4	44.3	40.5
$1.3x10^{-2}$	45.7	40.8	49.9	46.2	47.3	45.6	54.6	52.4
$3.3x10^{-2}$	49.1	44.1	54.7	54.7	--	--	55.7	54.7
$5.6x10^{-2}$	--	--	59.3	55.0	55.0	52.2	59.1	58.7
$1.3x10^{-1}$	49.6	47.5	67.1	61.0	67.8	61.4	69.3	62.7

3.2 Strain rate dependence of flow stress

The dependence of flow stress, σ_m on the applied strain rate is investigated by plotting σ_m versus the initial strain rate $\dot{\varepsilon}$, using double logarithmic scale as shown in Figure 2. The data points for each grain size fall on two segment lines suggesting that the deformation behavior under the present conditions can be divided into two regions. The value of the strain rate sensitivity, m as represented by the slope of each segment line $(\partial \ln \sigma / \partial \ln \dot{\varepsilon})_{T,d}$, changes from m~ 0.5 at low strain rates to m~ 0.11 at high strain rates. This change in m value suggests transition from superplastic region (m~ 0.5) to dislocation deformation region (m ~ 0.11) with increasing strain rate. The corresponding change in stress exponent n (=1/m) is from a value of 2 at low strain rates to a value of 8.9 at high strain rates. In addition, inspection of Figure 2 suggests that values of the flow stress and transition strain rate between the two deformation regions depend on the grain size. This dependence on the grain size will be presented in the next section. When σ_s is plotted vs. $\dot{\varepsilon}$, curves similar to those presented in Figure 2 are obtained. The σ_s- $\dot{\varepsilon}$ curves are not given in the present paper.

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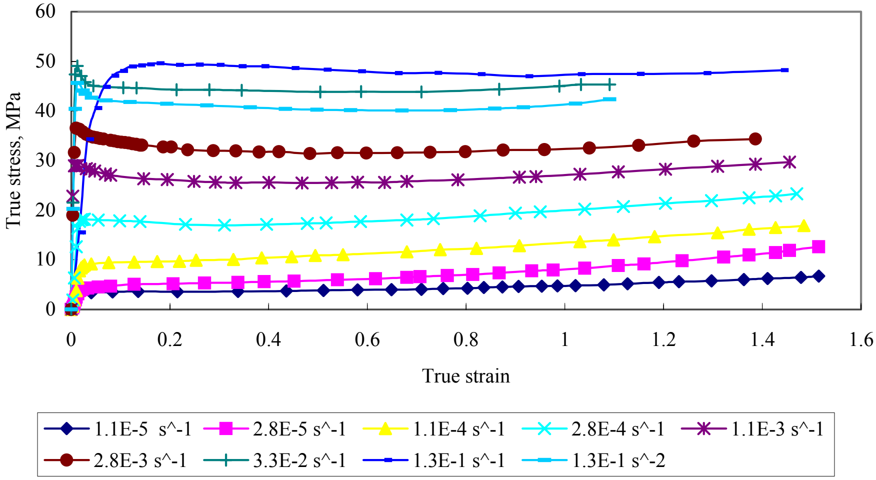

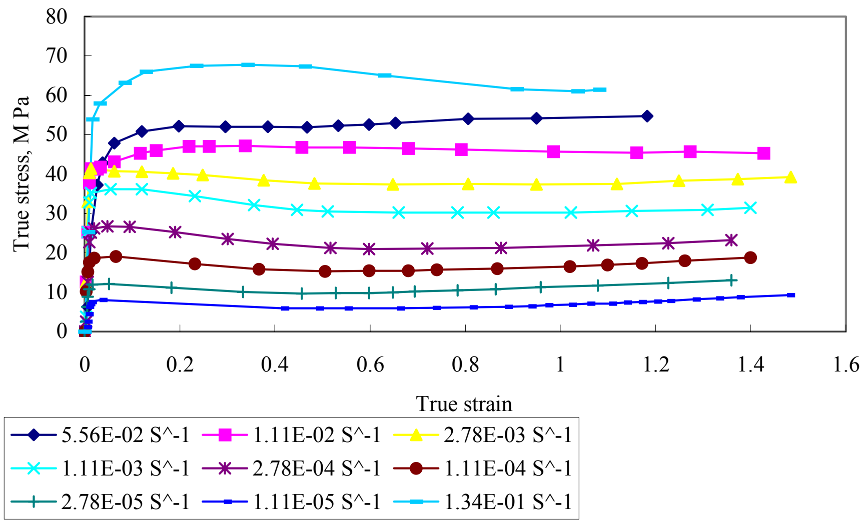

Figure 1. True stress-strain curves for d= 2.8 µm (top) and d=5.0 µm (bottom).

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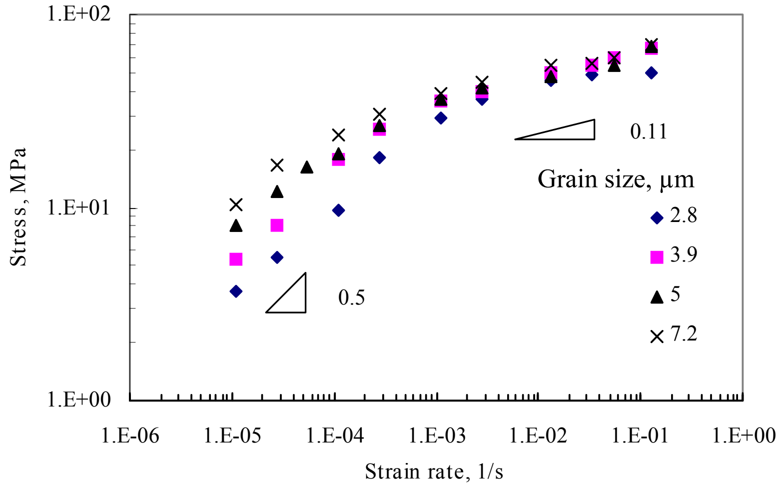

Figure 2. Strain rate dependence of flow stress for various grain sizes.

3.3 Grain size dependence of strain rate

Figure 3 illustrates the relation between the flow stress and grain size at the two deformation regions. The data points at each region fall on a straight line with a slope s representing the grain size dependence of stress ($\sigma \propto d^s$). Values of s inferred from the figure, are 1.1 and 0.18 for superplastic region and dislocation creep region, respectively. Consequently, the grain size dependence of strain rate, p (=s/m) in these two regions is calculated as 2.2 and 1.6, respectively. These values were checked by plotting $\dot{\varepsilon}$ vs. d in Figure 4, using logarithmic scale at stress of 10 MPa (superplastic region) and stress of 40 MPa (dislocation creep region). The values obtained for p are 2.3 and 1.65, respectively. These values are essentially similar to those based on s and m values.

The superplastic region extends to high strain rates with decreasing grain size. The grain size dependence of transition strain rate, $\dot{\varepsilon}_t$ between the two deformation regions is shown in Figure 5. The relationship between the transition strain rate and grain size can be described as $\dot{\varepsilon}_t \propto d^{-p}$ where p has a value of 2.5. The transition stress is weakly dependent on grain size (see Figure 1). The grain size exponent of transition strain rate at high temperature was slightly higher than the present value (p=3.3); see [Combres and Levaillant, 1990].

3.4. Threshold stress

In order to examine the possibility that a threshold stress, σ_0 exists during the superplastic behavior of the present high-purity alloy, the data points in this region for grain sizes of 2.8 and 3.9 µm are plotted as σ vs. $\dot{\varepsilon}^m$ (m=0.5) using a double linear scale(Figure 6). As seen in the figure, the datum points fall on straight lines whose extrapolation passes through the origin, giving σ_0 a value of zero. For the same alloy of commercial purity with grain size 2 µm

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tested at room temperature, it was noticed that m decreases gradually from 0.51 at $\dot{\varepsilon}$=6.3x10^{-4} s^{-1} to m=0.3 at $\dot{\varepsilon}$=3x10^{-5} s^{-1} [Zehr and Backofen, 1965]. The decrease in m value with strain rate (presence of region I) was attributed to the operation of

threshold stress process due to segregation of impurity atoms to grain boundaries [Soliman, 1994]. In the present results though extended to lower strain rates; no change in m value (~0.5) was observed.

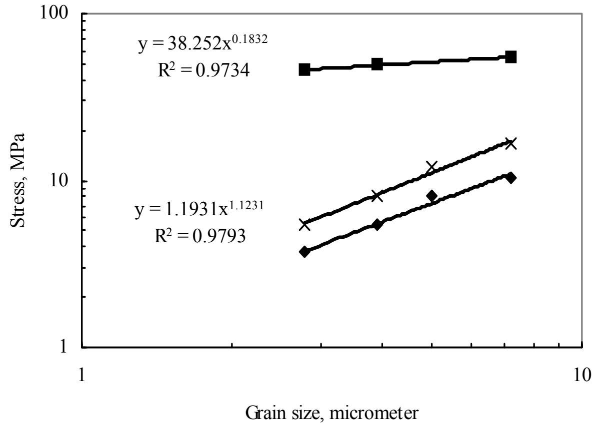

Figure 3. The relation between flow stress and grain size in region II (diamond, $\dot{\varepsilon}$=1.1x10^{-5} s^{-1} ; x, $\dot{\varepsilon}$=2.8x10^{-5} s^{-1} ; the value of s =1.1) and region III (square, $\dot{\varepsilon}$= 1.3x10^{-2} s^{-1} ; the value of s = 0.18).

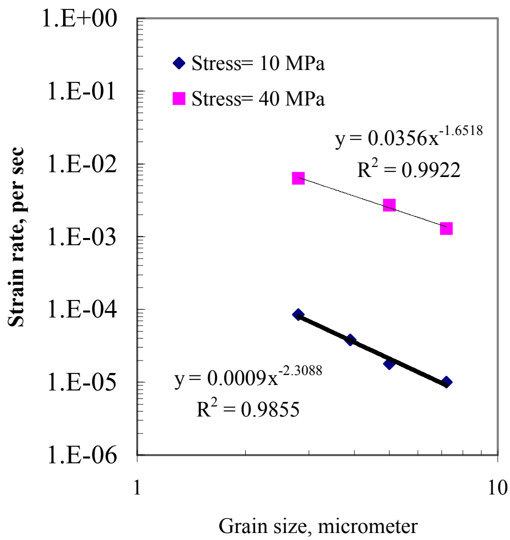

Figure 4. Strain rate vs. grain size at stress of 10 MPa (superplastic region) and stress of 40 MPa (dislocation creep region).

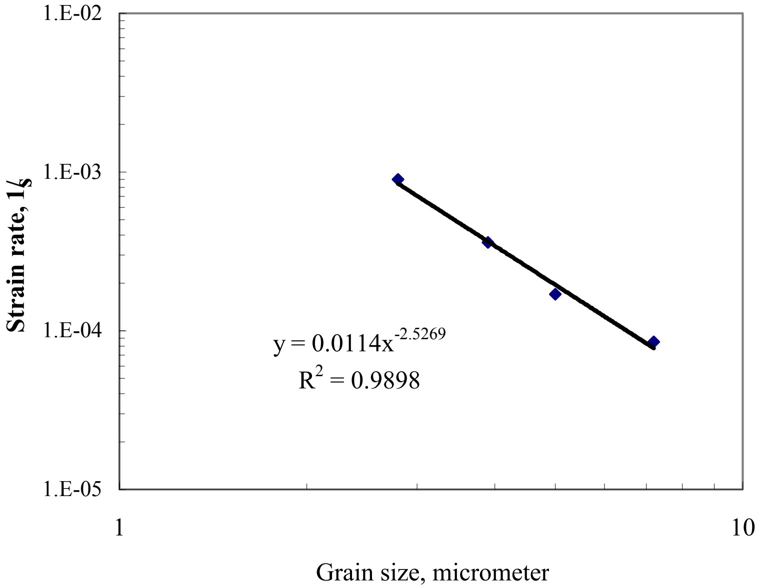

Figure 5. The relation between transition strain rate and grain size.

3.5. Apparent activation energy

In addition to the experiments performed at room temperature, few experiments were conducted at T=273 K at a strain rate of 0.033 s^{-1}. The apparent activation Q_a for dislocation creep region was roughly estimated using the values of flow stress at strain rate of 0.033 s^{-1} at temperatures of 273 K and 295 K. The value of Q_a was calculated from the relation:

$$Q_a = \left(\frac{R}{m}\right)\left(\frac{\partial \ln \sigma}{\partial (1/T)}\right)_{\dot{\varepsilon}} \qquad (2)$$

Where R is the gas constant (=8.31 kJ mol^{-1} K^{-1}) and m is the strain rate sensitivity (=0.11). The value obtained for Q_a is 45 kJ/mol. This value is very similar to those previously reported for the superplastic region [Mohamed and Langdon, 1975, Lam et al., 1979, Grivas et al., 1979], but it is almost one half of that reported for dislocation creep region [Lam et al., 1979, Kashyap and Murty, 1981].

4. DISCUSSION

The high temperature behavior of polycrystalline materials may, in general, arise from operation of different deformation mechanisms that act either independently or in a sequential manner where the fastest controls the deformation behavior for independent processes and the slower controls the behavior in sequential processes. Therefore, under favorable combinations of material parameters and experimental conditions, a single diffusion-controlled process dominates resulting in the presence of a distinct deformation region whose strain rate may be represented by the Dorn equation of the form:

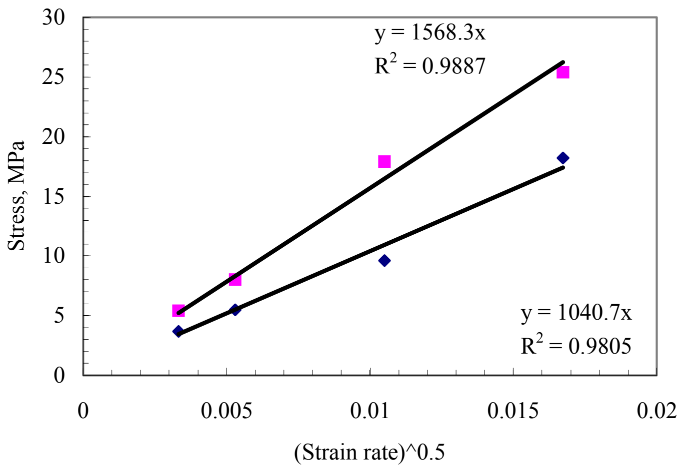

Figure 6. A linear plot of stress vs. $\dot{\varepsilon}^{0.5}$ in region II for grain size 2.8 µm(diamond) and 3.9 µm (square).

$$\dot{\varepsilon} = A\frac{DGb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n \quad (3a)$$

where A is a dimensionless constant, *k* is a Boltzmann's constant, *G* is the shear modulus, *b* is the magnitude of the Burger's vector and *D* is the diffusion coefficient given by

$$D = D_0 \exp\left(\frac{-Q}{RT}\right) \quad (3b)$$

where D_0 is the frequency factor, *Q* is the activation energy of the diffusion process that controls the deformation mechanism. According to equation 3 any high-temperature deformation process is completely defined by finding the appropriate values for these parameters A, p, n (=1/m) and Q. Comparison between the experimental values of these parameters and those established for basic deformation mechanisms may lead to identification of the rate controlling process.

The deformation behavior of superplastic materials has been extensively studied; see for example [Gifkins, 1982 and Langdon, 1991] and, as a result, three deformation regions have been established, and designated as I, II and III at low, intermediate and high strain rates, respectively. In region II (superplastic region), m~ 0.5, p~ 2, Q= Q_{gb} (activation energy for grain boundary diffusion) and maximum ductility is observed. In regions III and I, m decreases to a lower value ≤ 0.2, Q> Q_{gb}, p~ 2(region I) or p~ 0 (region III) and ductility is less. Region I has not been observed by many investigators [Lam et al., 1979, Kashyap and Murty, 1981, Schneibel and Hazzledine, 1983] when using superpure materials. It has been established recently that region I does not represent a genuine change in the rate controlling mechanism, but rather arises from operation of a threshold process due to segregation of impurity atoms on the grain boundaries [Mohamed, 1983, Yan et al., 1994].

In the present investigation two distinct regions are identified based on m and p values (Figure2). At intermediate strain rates a superplastic mechanism is operative whereas at high strain rates a dislocation controlled creep mechanism dominates. The present values of m and p observed in the superplastic region are in good agreement with those reported previously [Mohamed and Langdon, 1975, Grivas et al., 1979, Lam et al., 1979, Schneibel and Hazzledine, 1983]. In region III, m= 0.11 and p= 1.6. Region III has received little attention previously [Grivas et al., 1979, Lam et al., 1979, Kashyap and Murty, 1981] and the reported m values are in the range of 0.1 to 0.18. The present value is similar to that reported by one report [Kashyap and Murty, 1981]. On the other hand the value of p=1.6 is slightly higher than those reported previously [Soliman, 1994 (p=1.4) and Kashyap and Murty, 1981 (p=1)]. Finally, it was noted [Lam et al., 1979] that there was an apparent grain size dependence of the creep rate in specimens with small grain size. However, this dependence was not carefully examined and attributed to the fact that these specimens were not subjected to the same thermal processing as other specimens of large grain size.

Having determined the grain size exponent, p, it is interesting to examine the relation between the flow stress, σ_m (the peak stress in the true stress-strain curve as listed in Table 1) and the grain size compensated strain rate $\dot{\varepsilon}(d/b)^p$, using double logarithmic scale as shown in Figure 7 a and b for region II (p=2) and region III (p=1.6), respectively. Included also in this figure the tensile test data conducted previously under similar experimental conditions, but with slightly different grain sizes [Soliman 1994 and 1995a]. Inspection of the figure shows that the datum points coalesce on straight lines having slope of m~ 0.5 and~ 0.11 for region II and region III, respectively. This suggests that the estimated values for p in both regions are reasonably accurate.

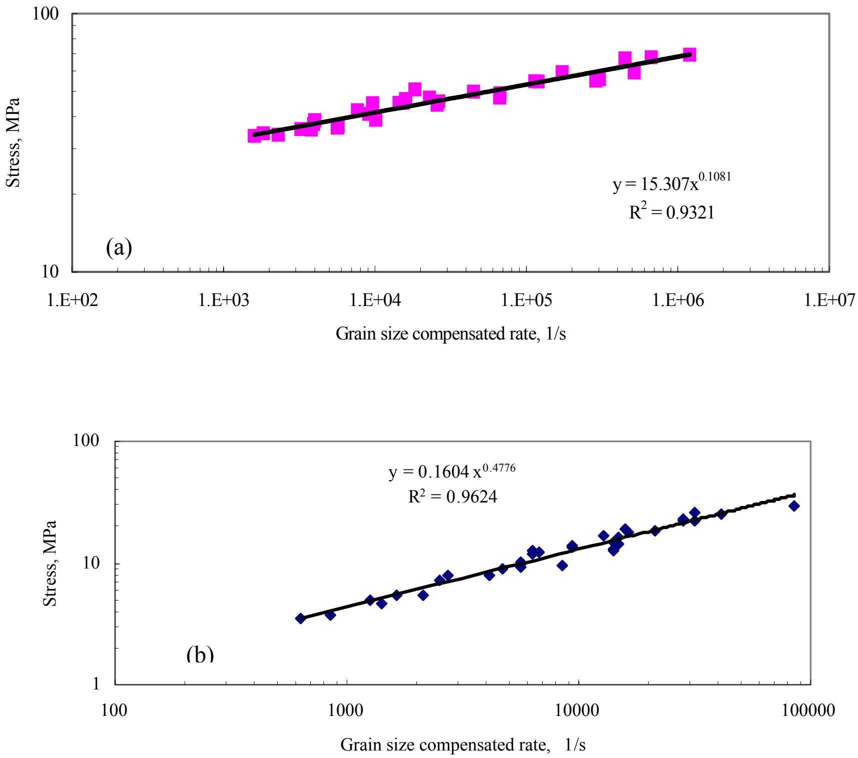

Figure 7. Flow stress vs. grain size compensated strain rate for (a) dislocation creep region, (b) superplastic region.

The apparent activation energy for creep was determined in region III only and the value obtained (45 kJ/mol) is very close to that observed in region II (controlled by grain boundary sliding)[Lam et al., 1979,Grivas et al., 1979, Mohamed and Langdon, 1975]. However the low value of m=0.11 refutes the possibility that grain boundary sliding is rate controlling in this region, and suggests that some form of dislocation creep is rate controlling. For lattice diffusion controlled dislocation creep, the activation energy is equal to~ 87 kJ/mol [Lam et al., 1979, Grivas et al., 1979] ,which are twice the present value and the stress exponent n observed by those investigators has the value of 5.7 and 7.1, respectively. The origin of region III may be attributed to the operation of dislocation creep controlled by pipe diffusion, which has activation energy, about one half of that for self diffusion [Soliman 1993,1995b]. The high value of n~ 8.9 and low activation energy support this possibility; in case of pipe-diffusion controlled dislocation creep the value of n increases by 2 over the value of n (5.7 to 7.1) for lattice-diffusion controlled dislocation creep. The grain boundary sliding that controls the deformation process in the superplastic region [Valiev and Langdon, 1993] and dislocation creep are two independent mechanisms and the faster controls the deformation behavior [Gifkins 1982]. In fine grained materials, the contribution of grain boundary sliding to the total strain at high strain rates, represent a small fraction (~0.2); the remainder is contributed by dislocation creep [Drury et al. 1989]

The role of dynamic recrystallization (as it appears from the shape of the true stress-strain curves especially at high strain rates) and its effect on the evolution and refinement of grain size requires further investigation.

5 CONCLUSIONS

1. The deformation behavior of Pb-Sn eutectic with grain size in the range from 2.8 to7.2 μm, when studied using compression tests in the strain rate range of 10^{-5}-10^{-1} s^{-1} at room temperature, can be divided into two regions: pipe-diffusion controlled dislocation creep region at high strain rates and superplastic region at intermediate strain rates.

2. In superplastic region, the strain rate sensitivity m was observed to be 0.5, while the grain size exponent p is 2.2.

3.In dislocation creep region the observed values for m and p are 0.11 and 1.6, respectively. The observed activation energy (45 kJ/mol) in this region is very close to that for dislocation core diffusion (pipe diffusion).

4. The transition strain rate $\dot{\varepsilon}_t$ between the two deformation regions increases with decreasing the grain size d. This dependence on grain size is represented as $\dot{\varepsilon} \propto d^{-p}$, where p=2.5.

5. The present data on Pb-Sn eutectic lend support to the suggestion that the origin of region I is related to a threshold stress which is resulting from impurity-atoms segregation to the grain boundaries.

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