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Evaluation of microstructural instability during the differential strain rate test of a superplastic alloy

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Stress (σ)–strain rate ($\dot{\epsilon}$) data of banded and elongated grain microstructures of the Pb–Sn eutectic alloy were analysed over 298 to 443 K to evaluate microstructural instability during differential strain rate tests in the superplastic region. With reference to a stable equiaxed microstructure exhibiting unique σ–$\dot{\epsilon}$ relation, banded structure is more susceptible to strain hardening while the elongated grain microstructure exhibits either strain softening or strain hardening depending on the test temperature. This flow behaviour is considered in terms of a change in grain size, represented by the cube root of the grain volume. Activation energy for grain growth calculated from the differential strain rate test data indicates that the activation energy depends on strain rate and type of microstructure.

1. Introduction

The steady-state superplastic flow is usually represented by

$$\dot{\epsilon} = \frac{AD_0Eb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{E}\right)^n e^{-Q/RT} \quad (1)$$

Here $\dot{\epsilon}$ is the steady strain rate, σ is the stress, A is a dimensionless constant, $D_0 \exp(-Q/RT)$ is the diffusion coefficient, E is Young's modulus, b is the atom size, d is the grain size, p is the grain size exponent and n is the stress exponent; kT and RT have their usual meanings. Equation 1 is based on the premise of stable microstructure exhibiting no strain hardening or softening during high temperature flow. The flow stress of superplastic materials in the as-worked state, however, becomes strain dependent up to some strain of the order of 0.3, initially [1].

Our earlier observations indicate that the Pb–Sn eutectic alloy, when deformed in the as-worked condition [2] or after annealing [3], exhibits strain dependent flow stress in high temperature deformation. This behaviour is due to a concurrent microstructural change. However, in this alloy, the uniqueness of the stress–strain rate relation is valid beyond about 0.3 strain and Equation 1 becomes applicable. Since the microstructural instability is the major cause of differences between Equation 1 and experimental observations during the transition stage (non-steady-state) of flow behaviour, it is possible to assess the microstructural instability on the basis of flow behaviour of stable microstructures. We report in this paper the results of an analysis of evaluating the change in grain size of the Pb–Sn eutectic alloy with different initial microstructures from their σ–$\dot{\epsilon}$ data obtained by differential strain rate tests.

2. Experimental procedure and results

Since, the details of the experimental procedure and the results were already published elsewhere [2–5], only a brief summary is presented here.

The Pb–Sn eutectic alloy was processed to obtain equiaxed, banded and elongated grain microstructures by different thermomechanical treatments of the cast ingots. Equiaxed microstructures were obtained by three different routes: (i) extrusion followed by annealing at 433 K, (ii) forging of the cast ingot in different directions in

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a controlled manner and (iii) extrusion followed by prestraining in tension to approximately 30% strain in the superplastic region. Banded structures were obtained by a two step working of the cast ingot with an intermediate annealing at 443 K for 30 days. Elongated grains were obtained by extrusion of a 75 mm diameter cast ingot to rods of 12.5 mm diameter followed by swaging to a final diameter of 7.5 mm.

Tensile specimens with the above microstructures, except for the equiaxed one obtained by route (iii), showed a significant influence of strain on the flow stress during constant crosshead speed (but corrected for constant strain rate) tests as well as differential strain rate tests with repeated strain rate cycling in an Instron machine. No significant change in the grain size occurred with strain in the case of equiaxed microstructures, whereas banded and elongated grain microstructures exhibited a reduction in the band length or degree of grain elongation with concurrent grain coarsening in the lateral direction.

Tensile specimens with varying initial microstructures were deformed by the differential strain rate test over the range of the strain rates (10^{-5} to $10^{-2}\,sec^{-1}$) and temperatures (298 to 443 K). The observed results [3–5] can be classified into two groups. Firstly, the results on equiaxed microstructures obtained by route (iii) support the uniqueness in the σ–$\dot{\epsilon}$ relation with a strain rate sensitivity index m $(= 1/n) = 0.6$, $p = 3.34$ and $Q = 44.7\,kJ\,mol^{-1}$ for $T < 408\,K$ and $81.1\,kJ\,mol^{-1}$ for $T > 408\,K$. Secondly, the equiaxed microstructures obtained by routes other than (iii) and the non-equiaxed microstructures do not support the uniqueness in the σ–$\dot{\epsilon}$ relation. Although the equiaxed microstructures are stable and significant instability occurs in the non-equiaxed microstructures, the parameters m and Q are virtually comparable. The Q-value is, however, observed to be about 20% lower in the case of strain dependent flow behaviour in comparison to the strain independent flow. The p-value for equiaxed microstructures processed by routes (i) and (ii) is approximately 2.5.

3. Analysis and discussion

The assessment of the microstructural instability during differential strain rate tests is based on the following considerations:

(a) The difference in flow stress at a given strain rate and temperature for specimens with different initial microstructures may arise from the variations in grain size and shape. By deforming specimens with elongated grains and following the microstructural change with increasing strain, it was noticed that the grain size, taken as the cube root of the grain volume, correlates reasonably well with the flow stress [6]. Similary, by considering the data of Suery and Baudelet [7] on brass, we note that the change in the grain size (calculated as the cube root of grain volume assuming rod-shaped grains) with increasing strain is in qualitative agreement with the observed strain hardening and softening at different strain rates. Thus it appears that the superplastic flow behaviour of the as-worked material in the non-steady-state region may be assessed in terms of the grain size represented by the cube root of the grain volume for non-equiaxed microstructures [8]. This approach was also adopted in a recent theoretical analysis [9] of the deformation behaviour.

(b) The observed difference in Q between the equiaxed microstructures exhibiting strain dependent (SD) and strain independent (SI) flow behaviour can be explained on the basis of the strain effect on the flow stress [10] in the former. Since SI equiaxed microstructures show an unique σ–$\dot{\epsilon}$ relation, the higher Q and the lower p values of the SD equiaxed microstructures is to be attributed to the influence of strain in the initial part of deformation (about 30% strain). The value of $p = 2.5$ for SD equiaxed microstructures in comparison to 3.34 for SI equiaxed microstructures suggests that this difference arises from microstructural details other than the grain size. It is reasonable to assume that $p = 3.34$ represents the actual inverse grain size exponent during this early part of deformation, had there been no strain effect.

(c) The similarity in the m and Q parameters of the constitutive relation for SD equiaxed and non-equiaxed microstructures suggests that the strain effect on flow behaviour might be more important than the influence of grain shape. The change in the grain size of non-equiaxed microstructures may be evaluated with reference to the SD equiaxed microstructure. Further, the difference in the parameters of the constitutive relation for SD and SI equiaxed microstructures is seen to be in terms of the magnitude of p. It is thought, therefore, that using σ–$\dot{\epsilon}$ data corresponding to SI equiaxed microstructure but p for SD equiaxed microstructures will show an unique σ–$\dot{\epsilon}$ relation under the constraint of strain dependent flow behaviour.

We now present the results of the analysis of changes in grain size during differential strain rate tests. The mean intercept length (considered as the grain size for comparison here), in the case of elongated grains, was evaluated to be 6.06 μm by considering the cube root of the volume of a cylinder with a mean diameter of 5.05 μm and length of 11.1 μm. Since the banded structure is a cluster of equiaxed grains of similar phases aligned along the axis of deformation processed rods, the grain size can be represented by the grain dimension in the transverse section. The mean intercept lengths for the banded and SI equiaxed microstructures are 4.85 and 5.57 μm, respectively.

The σ–$\dot{\epsilon}$ data obtained by differential strain rate tests in the temperature range of 298 to 443 K for banded [4], elongated grain [4] and SI equiaxed [5] microstructures were normalized to represent a mean intercept of 5 μm. The flow stress for a grain size of 5 μm at a given temperature and strain rate was evaluated from

$$\sigma_2 = \left(\frac{d_2}{d_1}\right)^{p/n} \sigma_1 \qquad (2)$$

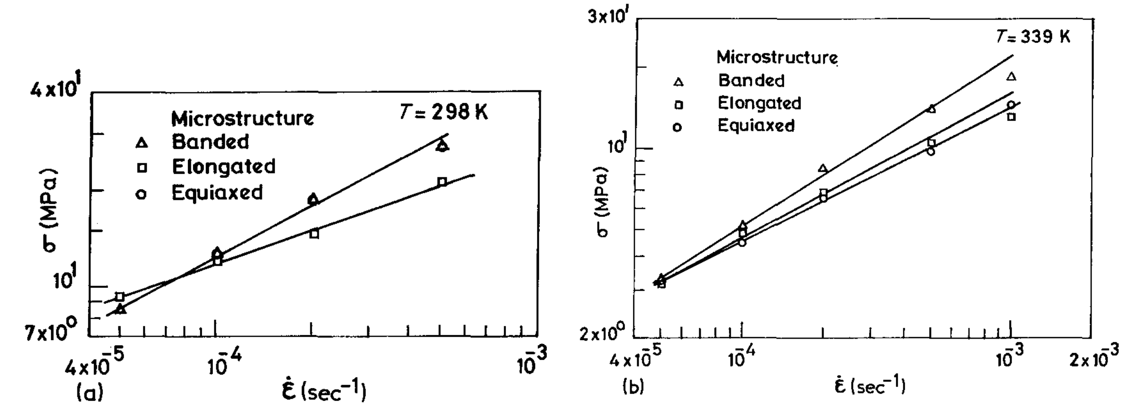
where σ_2 is the flow stress for grain size d_2 (= 5 μm), obtained from flow stress σ_1, corresponding to different grain shapes but of size d_1; $p = 2.5$ and $n = 1.67$. The σ–$\dot{\epsilon}$ data thus obtained from the original data corresponding to banded, elongated grain and equiaxed microstructures are presented in Fig. 1 for different test temperatures.

At 298 K (Fig. 1a), the σ–$\dot{\epsilon}$ data for banded and equiaxed microstructures are identical indicating that there is no grain coarsening of the banded structures during the differential strain rate test. However, the σ–$\dot{\epsilon}$ data of microstructures with elongated grains lie above those of the equiaxed ones in the beginning, but at a later stage the flow stress is lower than that of the equiaxed structure indicating strain softening due to break up of elongated grains into an equiaxed form. At 339 K (Fig. 1b) strain hardening in banded structures is seen while the σ–$\dot{\epsilon}$ data of specimens with elongated grains coincide with those of the equiaxed, suggesting that the effect of evolution of elongated grains towards an equiaxed shape is compensated by their concurrent grain coarsening. At 391 K (Fig. 1c) grain coarsening is predominant in both banded and elongated grains but the lower flow stress of the elongated grains is due to a strain softening effect resulting from evolution towards an equiaxed shape. At a still higher temperature (Fig. 1d), both the grain coarsening and evolution towards an equiaxed shape become predominant leading to a much higher strength level for the banded structures, whereas the strain effect is nullified for elongated grains. This prediction of microstructural instability is in qualitative agreement with the observed microstructural changes during constant crosshead speed tests [6].

Using the normalized σ–$\dot{\epsilon}$ data of 5 μm grain size for different microstructures and Equation 2, the instantaneous grain size was calculated during the different strain rate test. Fig. 2 shows the grain size corresponding to different strain rates at 391 K from this calculation. Figs. 3a and b illustrate the effect of the strain rate and the test temperature on the grain size for banded and elongated grain structures, respectively. At a strain rate of $5 \times 10^{-5}\,\text{sec}^{-1}$, the temperature dependent change in the grain size is shown in Fig. 4 for both banded and elongated grain structures in comparison with the stable equiaxed microstructures.

In isothermal annealing, the grain coarsening is represented [11] by $\bar{d} = (Kt)^{n'}$ where $\bar{d}$ is the grain size, t is the annealing time and, K and n' ($\leqslant 0.5$) are constants. If n' is independent of temperature and K varies exponentially with temperature ($K = K_0\,e^{-Q_m/RT}$), the activation energy Q_m for grain growth can be determined from the gradient ($-n'Q_m/R$) of an isochronal plot of $\ln \bar{d}$ against $1/T$. These concepts of isothermal and isochronal annealing can also be applied to the dynamic case of annealing under stress during the differential strain rate tests of the present work. It may be noted that in the differential strain rate test, the variable time, strain and strain rate are simultaneously involved and d against $\dot{\epsilon}$ data at any strain rate corresponds to a particular strain and time. Thus the activation energy for grain growth of the Pb–Sn eutectic during dynamic annealing is calculated at a given strain rate (with inherent cumulative strain) for both elongated grain and banded microstructures. The n' for this purpose is calculated on the basis of the "rule of mixtures" using the data of pure elements [11] ($n' \simeq 0.5$ for zone refined tin and $n' \simeq 0.4$ for zone refined lead. The value of n' thus obtained is 0.474 for the eutectic composition. Fig. 5 shows Arrhenius plots for banded and elongated grain microstructures at a strain rate of $5 \times 10^{-4}\,\text{sec}^{-1}$.

The activation energy values for grain growth during differential strain rate tests thus evaluated from the above considerations are listed in Table I



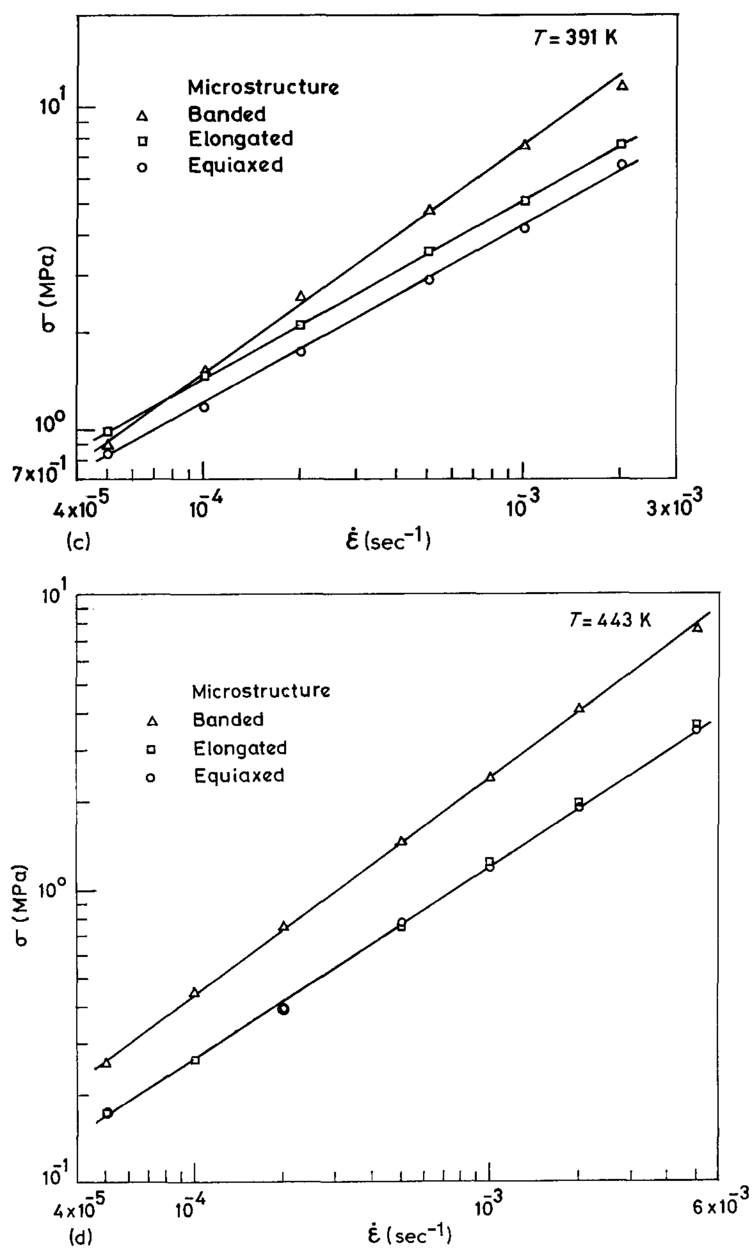


Figure 1 σ–$\dot{\varepsilon}$ behaviour of banded, elongated grain and stable equiaxed microstructures, all with an initial grain size of $5\,\mu m$ at (a) 298 K, (b) 339 K, (c) 391 K and (d) 443 K.

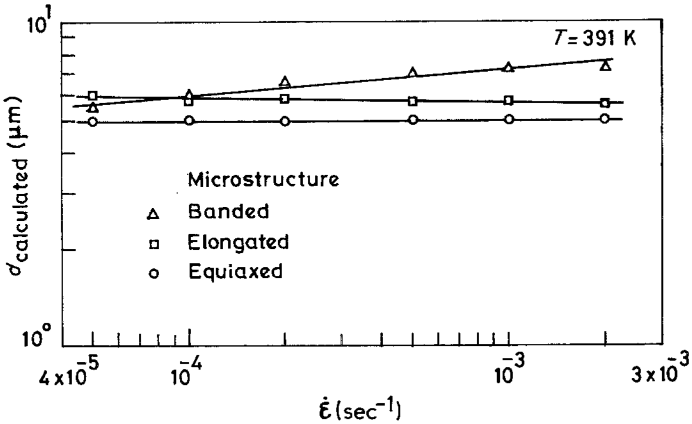


Figure 2 Plot of change in grain size from Equation 2 during a differential strain rate test.

for different strain rates in the superplastic region. These are significantly lower than any of the relevant activation energies corresponding to various processes either for Pb–Sn alloys or pure lead or tin listed in Table II. This difference can be noticed even in comparison with the activation energy for grain boundary migration of pure lead or tin during static annealing. It is, however, worth noting that the activation energy for grain boundary migration is generally based on describing the experimental curve $\bar{d}$ (t) by only a single set of parameters K, $\bar{d}_0$ and t_0 (initial time), whereas in reality the curve is composed of several regions. Simpson *et al.* [19] have shown that $\log \bar{d}$ against $1/T$ plot for pure lead shows four regions but higher values of Q (about 26.3 to 53.3 kJ mol^{-1}, taking $n' = 0.4$) are attributed to normal grain growth. It is expected that the grain boundary migration is more difficult in two phase alloys in comparison to single phase alloys. The observed low activation energies of the present study appear to be a consequence of stress enhanced diffusion

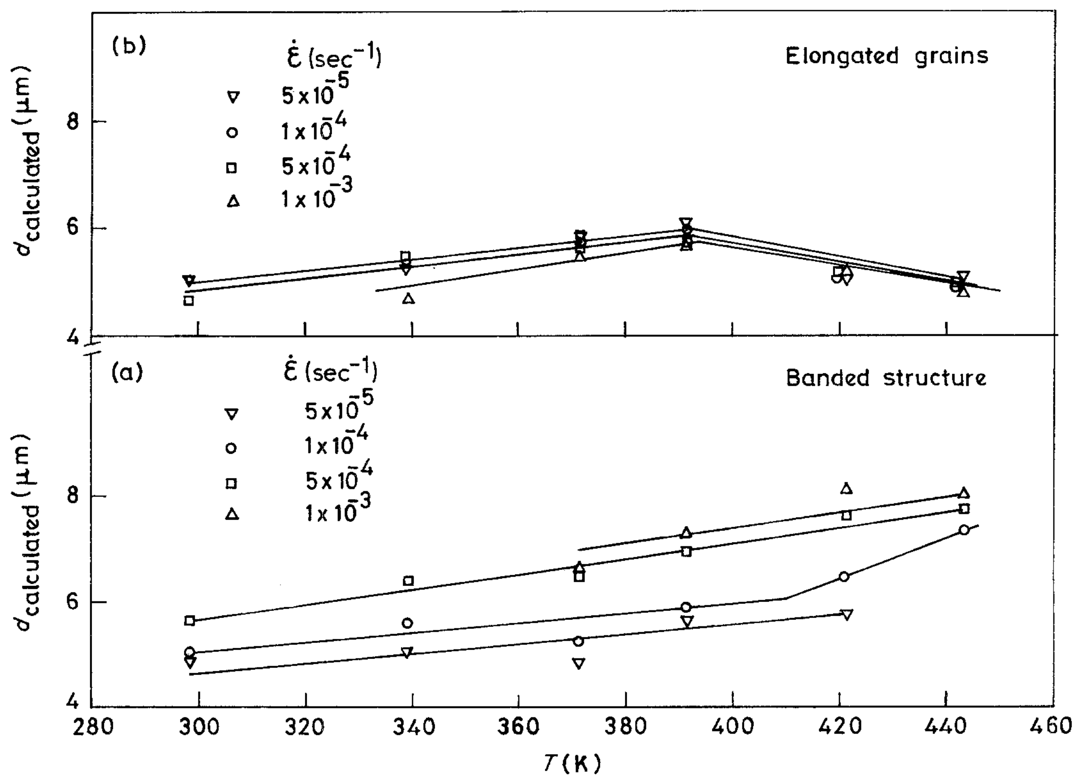


Figure 3 Effect of test temperature on grain size during a differential strain rate test of banded (a) and elongated grain (b) microstructures.

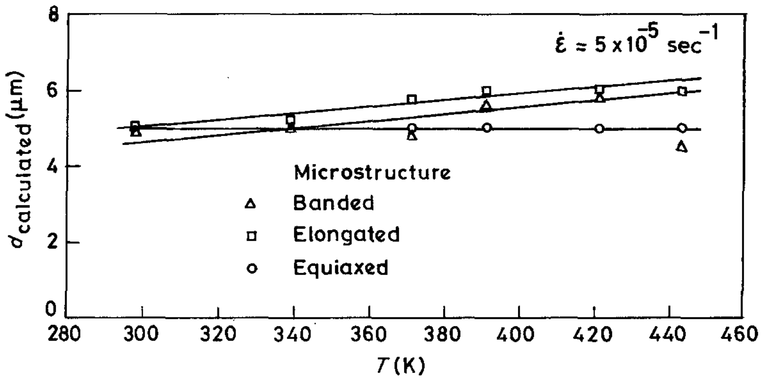


Figure 4 Grain size change of banded and elongated grain microstructures at $\dot{\epsilon} = 5 \times 10^{-5}$ sec^{-1} as a function of temperature during a differential strain rate test.

in the differential strain rate tests. In Pb–Sn alloys (containing 30 to 61 wt% Sn) which were undergoing superplastic deformation, the diffusivity of tin by the radioactive tracer technique was more than ten times that of the undeformed superplastic materials and more than a thousand times that of the nonsuperplastic alloys of the same composition [20]. The present observations of a lower activation energy for the grain growth during differential strain rate tests are in conformity with the trends of the above reported data.

It is seen from Table I that the banded structures exhibit somewhat lower activation energy for grain growth than the elongated grains in general. This may be partly due to grain coarsening in the banded structure being more akin to that in single phase alloys, Another point of interest, in this regard, is that microstructural instability in the case of elongated grains involves not only grain coarsening but also break up of the elongated grains into equiaxed ones.

The observed increase in the activation energy for grain growth with increasing strain rate may have its origin in the change of diffusivity due to grain coarsening arising from cumulative strain at the preceding strain rates in a differential strain rate test. An increase in grain size by a factor of over two has been reported in a differential strain rate test in earlier work [21] on the Pb–Sn eutectic. In the present investigation, a maximum increase in grain size by a factor of 1.6 (Fig. 3a) is noted at 443 K. In a Sn–1% Bi alloy the enhanced grain growth due to dynamic annealing is a maximum at an intermediate strain rate [22]. From a study of grain growth in Ni–Cr–Fe alloys [23] during superplastic deformation, it is reported that the grain size increases approximately linearly with test time regardless of the strain rate or elongation.

Thus an indirect assessment of microstructural changes from the differential strain rate test data appears promising, in general. However, changes in the flow stress during superplastic flow are not always accounted by the corresponding change in

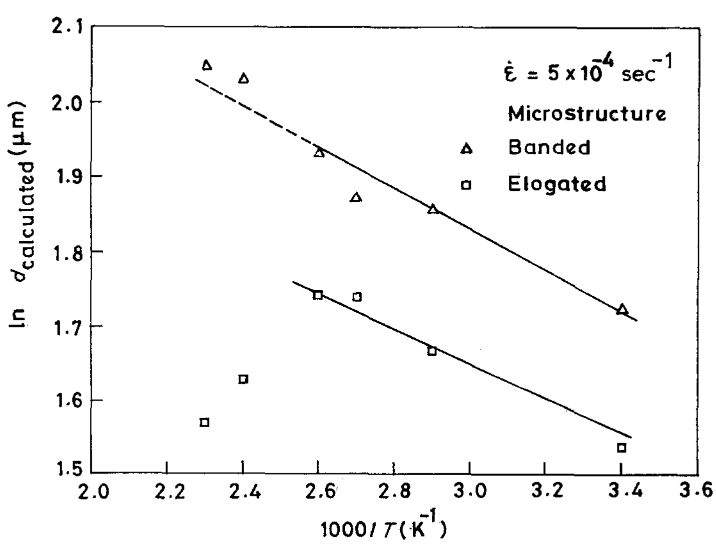


Figure 5 Arrhenius plot for determining activation energy for grain growth of banded and elongated grain microstructures during differential strain rate tests.

TABLE I Activation energy for microstructural instability during differential strain rate tests of banded and elongated grain microstructures

Strain rate (sec^{-1})	Activation energy ($kJ\ mol^{-1}$)	
	Banded microstructure	Elongated grains*
5×10^{-5}	6.3	8.9
1×10^{-4}	6.1	11.6
5×10^{-4}	11.2	11.2
1×10^{-3}	14.5	26.7

*Data up to 391 K were considered.

the grain size [2, 24, 25]. Detailed microstructural studies in relation to static and dynamic annealing are necessary to corroborate the kinetics of microstructural instability of a superplastic material assessed in this study from differential strain rate tests.

4. Conclusions

Superplastic flow behaviour of banded, elongated grain and equiaxed microstructure of the Pb–Sn eutectic shows substantial influence of strain on the flow stress. However, equiaxed microstructures obtained by prestraining (about 30% strain) of the deformation processed alloy show steady-state deformation. The parameters of the constitutive relation for superplastic deformation for the first types of microstructures are not significantly dependent on the structure. The $\sigma-\dot{\epsilon}$ data for different microstructures were normalized for the initial grain size of $5\,\mu m$ to evaluate instability in these structures during differential strain rate tests in the superplastic region. The $\sigma-\dot{\epsilon}$ data, with reference to those for stable equiaxed microstructures, indicate substantial grain growth leading to strain hardening in the banded structures whereas evolution towards an equiaxed shape, leading to strain softening is equally significant in the elongated grain microstructures. An attempt to estimate the activation energy for grain growth during a differential strain rate test shows that they range from 6.3 to 14.5 and 8.9 to $26.7\,kJ\,mol^{-1}$ depending on the strain rate for the banded and elongated grain microstructures, respectively. These values are significantly lower than that for grain boundary migration during static annealing of pure lead or tin.

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TABLE II Activation energy values for diffusion, creep, grain boundary sliding and grain boundary migration in Sn, Pb and Pb–Sn alloys

	Activation energy ($kJ\ mol^{-1}$)		
	Sn	Pb	Pb–Sn
Q_1	106.2 ± 3.8 [12]	107.5 ± 1.0 [12]	100.6 [12]
Q_{gb}	40.0 ± 2.9 [12]	65.8 (487–533 K) [12]	–
Q_{gbs}	79.6 [13]	54.5 to 108.9 [13]	–
Q_c	46.0 ($T/T_m \leq 0.8$)	116.9 [15]	100.6 to 117.3 [15]
	109.2 ($T/T_m \geq 0.8$) [14]	–	(4.4 to 19.4 at% Sn)
Q_m	33.1 [16]	25.1 [17, 18]	–
	(425–500 K)	(473–593 K)	

Subscripts of the activation energy, Q, have the following meaning; 1 = lattice diffusion, gb = grain boundary diffusion, gbs = grain boundary sliding, c = creep, m = migration of single boundaries.

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