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On the evaluation of parameters of the constitutive equation for 7475 Al alloy

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From the mechanical data on 7475 Al alloy, it is evident that flow stress is significantly dependent on the strain during superplastic flow. This is due to its ability to strain-harden during superplasticity. The rate of increase in the flow stress is much higher at 457° C than at 517° C. This gives rise to non-unique values for the parameters of the constitutive equation. At 457° C, whereas the stress exponent (n) and activation energy for superplastic flow at $1 \times 10^{-4}\,\text{sec}^{-1}$ increase only slightly with strain, the grain size sensitivity parameter (p) and structure parameter (A) decrease significantly with strain. These changes in the constitutive parameters are associated with dislocation activity occurring within the grain interior, leading to grain elongation without significant changes in the grain size, through the parameter, $(\boldsymbol{b}/d)^p$, of the constitutive equation.

1. Introduction

The steady-state superplastic flow behaviour is usually represented by [1]

$$\dot{\varepsilon} = \frac{A\,D_0 G b}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n \exp\left(\frac{-Q}{RT}\right) \quad (1)$$

Here $\dot{\varepsilon}$ is the steady strain-rate, σ is the stress, A is a dimensionless constant, $D_0 \exp(-Q/RT)$ is the diffusion coefficient, Q is the activation energy for superplastic flow, G is the shear modulus, $\boldsymbol{b}$ is the Burgers' vector, 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 a stable microstructure, exhibiting no strain-hardening or softening during high temperature flow. For the purpose of assessing the operating deformation mechanism, parameters m, p, Q and A are estimated by the appropriate use of experimental plots. The experimental curves for $\log\sigma$ against $\log\dot{\varepsilon}$ over a wide range of strain rates show three distinct regions, each region having characteristic values of m, p, Q and A. In the superplastic region, the magnitude of m generally varies from 0.3 to 0.9, the magnitude of p varies from 1.5 to 3.0, and Q varies from that of grain-boundary to lattice diffusion. In contrast to this, different models of superplasticity usually predict invariant values of these parameters. So there is a need to understand the source of variation in the parameters of the constitutive relation. Therefore, parameters of the constitutive relation have been evaluated at various strain levels in a 7475 Al alloy, which was fine-grain processed for superplasticity and which normally strain-hardened during high-temperature flow [2].

2. Experimental procedure

The 7475 Al alloy used in this study was obtained from the Rockwell International Science Center in the form of a 2.56 mm thick sheet. The chemical composition of this alloy is given in Table I. Tensile specimens of 7 mm gauge length and 6.56 mm gauge width were machined from the as-received sheet. Detailed microstructural analysis led to the conclusion that the grain size (mean linear intercept) in the elongated (rectangular parallelepiped) microstructures may be represented by the cube root of grain volume. The average of grain size measured on the three mutually perpendicular surfaces (by drawing random lines) was comparable to the grain size estimated as a cube root of the grain volume. In the following sections the latter was used to represent the grain size. Grain sizes of 14, 25 and 30 μm were obtained by annealing in vacuum at 530° C for 24, 72, and 144 h, respectively.

Tensile tests were conducted in an argon atmosphere by using an MTS machine programmed to run in a constant strain-rate mode), and a clamp-shell radiant furnace where the temperature was controlled to $\pm$ 5° C. Metallographic specimens were prepared under selected conditions and micrographs of the longitudinal and transverse surfaces were obtained. Grain size was determined from the micrographs of the surfaces using approximately 1000 intercept lengths. The error in the grain sizes ranged below 10% of the mean value at a confidence limit of 95%.

3. Experimental results

Differential strain rate tests were conducted in the decremental order of strain rates from 5×10^{-3} to

TABLE I Compositional limits of 7475 Al alloy (wt %)

Fe	Si	Cu	Mn	Mg	Cr	Zn	Ti	Others, each	Others, total	Al
0.10 max.	0.10 max.	1.2 to 1.9	0.6 max.	1.9 to 2.6	0.18 to 0.25	5.2 to 6.2	0.06 max.	0.05 max.	0.15 max.	Remainder

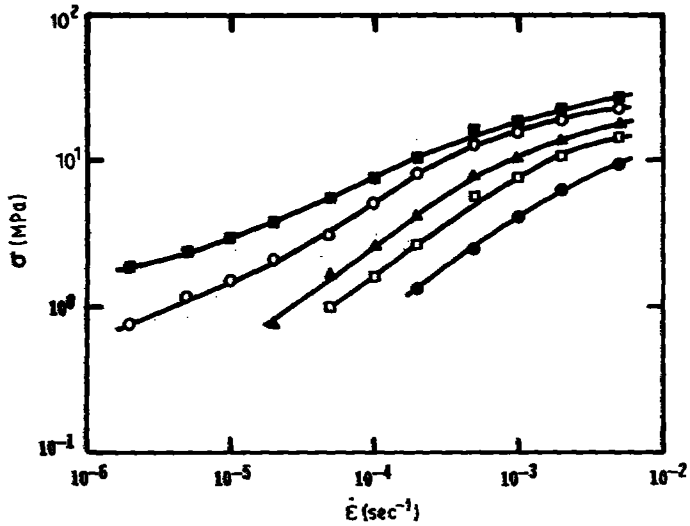

Figure 1 Stress against strain-rate behaviour for the 7475 Al alloy at various temperatures: (■) 437, (○) 457, (▲) 477, (□) 497, (●) 517° C. d_0 = 14 μm.

2×10^{-6} sec^{-1} on individual specimens at each temperature. Fig. 1 shows the σ–$\dot{\varepsilon}$ data obtained in the temperature range of 437 to 517° C. It can be seen that the data at low temperatures have a more distinct sigmoidal shape, with a narrow intermediate strain rate range representing Region II of superplastic deformation, when compared to the data in the higher temperature range. This apparent flow behaviour can be understood on the basis of microstructural changes occurring concomitantly during superplastic deformation. It can be seen that as the temperature decreases the curve is displaced to progressively lower strain rates and higher stresses. The strain-rate sensitivity was evaluated by using

$$m = \frac{\log (p_2/p_1)}{\log (\dot{\varepsilon}_2/\dot{\varepsilon}_1)} \qquad (2)$$

following the technique of Backofen *et al.* [3] by replacing cross-head velocity (V) with strain rate ($\dot{\varepsilon}$). In Equation 2, p_1 and p_2 are the loads corresponding to the strain rates $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$, respectively.

The maximum value of m determined from strain-rate change tests also decreases with temperature and occurs at a lower strain rate as the temperature is lowered. This is emphasized by the plots of m against $\dot{\varepsilon}$ (Fig. 2). The maximum value of m at 517° C is 0.8 at a strain rate of 1×10^{-4} sec^{-1} in comparison to corresponding values of 0.7 at 9×10^{-5} sec^{-1} at 457° C, and 0.43 at 2×10^{-5} sec^{-1} at 437° C.

The value for the parameter m was determined over the investigated range of strain rates for four initial grain sizes. The m against $\log \dot{\varepsilon}$ results for different grain sizes suggests the existence of a maximum in the values of m. As the grain size becomes larger, the $\dot{\varepsilon}$ at which the peak in the values of m occurs is lower. Within the strain-rate range investigated, it appears that the value of strain-rate sensitivity is smaller the larger the initial grain size (Fig. 3). At 457° C, a maximum m value of 0.7 and a maximum elongation of 840% occur at a strain rate of 1×10^{-4} sec^{-1}. There will be some emphasis in the subsequent sections on this particular strain rate while describing results for 457° C.

Separate specimens were deformed at constant strain rates and temperatures in the range of 1×10^{-5} to 5×10^{-3} sec^{-1} over 437 to 517° C to a true strain

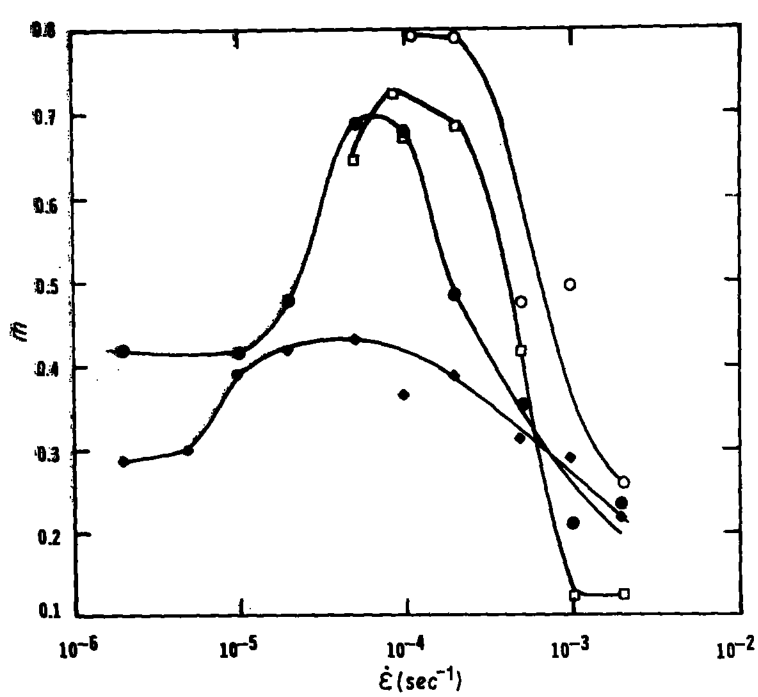

Figure 2 Plot of strain-rate sensitivity, m against strain rate, computed from Fig. 1a. T = (◆) 437, (●) 457, (□) 477, (▲) 497, (○) 517° C. d_0 = 14 μm.

TABLE II Constants for the constitutive equation used for the 7475 Al alloy

Symbol	Name	Constant	Reference
D_0	Diffusion coefficient	$1.71\,cm^2\,sec^{-1}$	21
b	Burgers' vector	2.8×10^{-8} cm	21
k	Boltzmann's constant	$1.38 \times 10^{-23}\,J\,K^{-1}$	–
R	Universal gas constant	$8.314\,J\,K^{-1}\,mol^{-1}$	–
G	Shear modulus (MPa)	2.0×10^4 at 710 K	22
		1.96×10^4 at 730 K	22
		1.92×10^4 at 750 K	22
		1.87×10^4 at 770 K	22
		1.83×10^4 at 790 K	22

of 1.4. The σ–ε behaviour (Figs 4, 5 and 6) observed in this range of conditions suggests that considerable work-hardening occurs during superplastic flow. Such a behaviour leads to a situation in which the flow stress is not invariant with strain. This is because the microstructure changes continuously during superplasticity. Hence, evaluation of parameters of the constitutive Equation 1 as a function of strain becomes important.

From the data depicted in Fig. 5, log σ against log $\dot{\varepsilon}$ plots at various levels of true strain were constructed. The slopes of such plots gave the strain-rate sensitivity, as a function of strain at various strain rates (Fig. 7). It is clear from Fig. 7 that m reaches a steady value of 0.5 beyond a true strain of 0.5, at a strain-rate of $1 \times 10^{-4}\,sec^{-1}$ and a temperature of 457° C.

Fig. 4 was used to evaluate the activation energy at various strain levels using the equation

$$Q = -2.3R \left. \frac{\partial \log (\sigma^{-n} G^{n-1} T)}{\partial (1/T)} \right|_{\dot{\varepsilon}, d} \tag{3}$$

The above equation was obtained by differentiating Equation 1 with respect to temperature and compensating for changes in the shear modulus G with temperature, and using appropriate values for the stress sensitivity parameter n $(= 1/m)$, at various strain levels. The values of the shear modulus in addition to other constant parameters used in the constitutive equation for this alloy are tabulated in Table II. From Fig. 8 the activation energies for Region II at a strain rate of $1 \times 10^{-4}\,sec^{-1}$ by using appropriate $n\,(= 1/m)$ values from Fig. 7 are 146, 173 and $166\,kJ\,mol^{-1}$ at true strain levels of 0.5, 0.7 and 1.0, respectively. Furthermore, the activation energy did not change significantly when specimens with higher grain sizes were used.

The grain size sensitivity parameter, p, can be calculated by differentiating Equation 1 with respect to grain size at constant strain rate and temperature, i.e.

$$p = n \left. \frac{\partial \log \sigma}{\partial \log d} \right|_{\dot{\varepsilon}, T} \tag{4}$$

The grain-size sensitivity parameter, p, obtained by using Fig. 6 at various levels of true strain is summarized in Fig. 9. The p value changes significantly

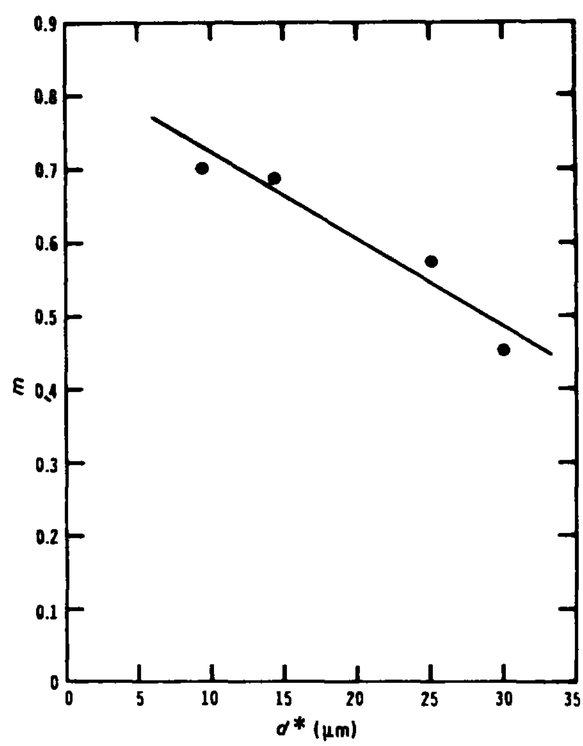

Figure 3 Plot of strain-rate sensitivity m against grain size for the 7475 Al alloy. $T = 477°$ C, $\dot{\varepsilon} = 1 \times 10^{-4}\,sec^{-1}$ (d^* = initial grain size).

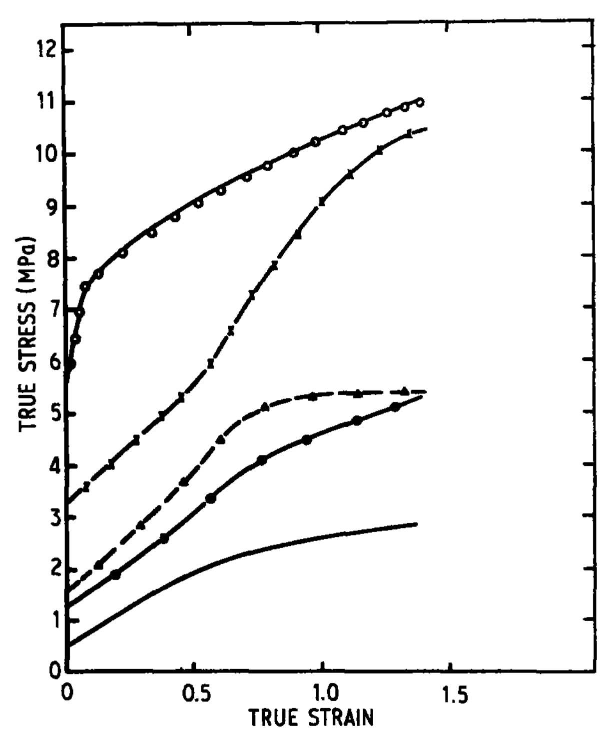

Figure 4 True stress against true strain plot for different temperatures at a strain rate of $1 \times 10^{-4}\,sec^{-1}$, with initial grain of $14.0\,\mu m$, up to a maximum strain of 1.4. T = (○) 437, (x) 457, (▲) 477, (●) 497, (——) 517° C. $d = 14\,\mu m$.

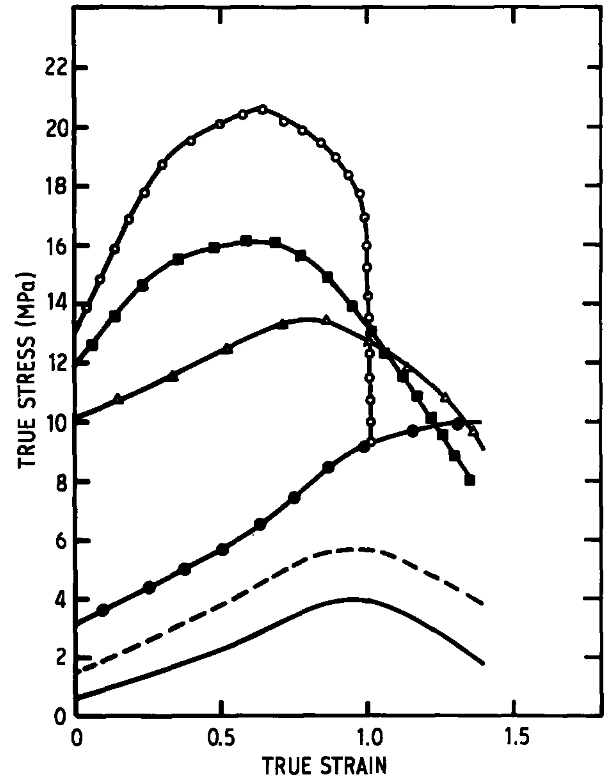

Figure 5 True stress–true strain plot for different strain rates at 457° C with an initial grain size of 14.0 μm, up to a maximum strain of 1.4. Values of $\dot{\varepsilon}$ (sec^{-1}) as follows: (——) 1×10^{-5}, (– – –) 5×10^{-4}, (●) $1 = 10^{-4}$, (△) 5×10^{-4}, (■) 1×10^{-3}, (○) 5×10^{-3}.

with true strain at a strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$ and temperature of 457° C. The value of p changed from a value of 2.16 at a strain level of 0.5 to 1.26 at a strain of 1.0, although no change in the grain size occurred after the high-temperature flow.

The parameter A at various levels of strain was determined by plotting all the available experimental data at appropriate strains, using the relation

$$\log\left(\frac{\sigma}{G}\right) \text{ against } \log \frac{\dot{\varepsilon}kT}{DGb}\left(\frac{d}{b}\right)^{p}$$

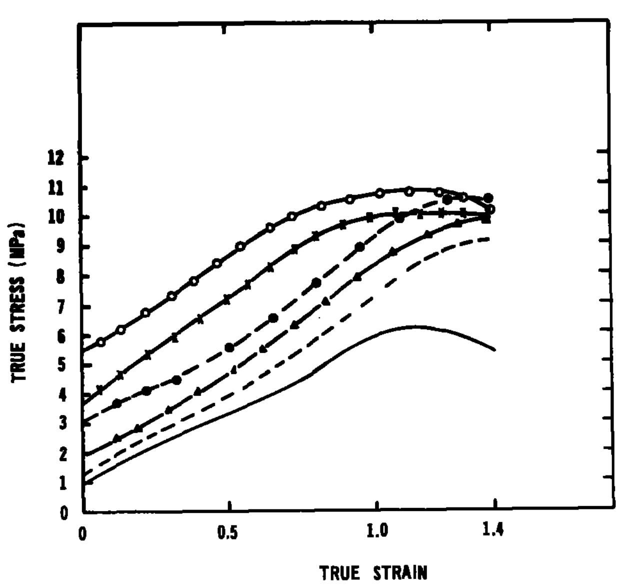

Figure 6 True stress–true strain plot for different grain-sizes at 457°C and at a strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$, up to a maximum strain of 1.4. Values of $d_0(\mu\text{m})$ as follows: (——) 9 (as-received), (– – –) 10, (▲) 11, (●) 14, (x) 25, (○) 30.

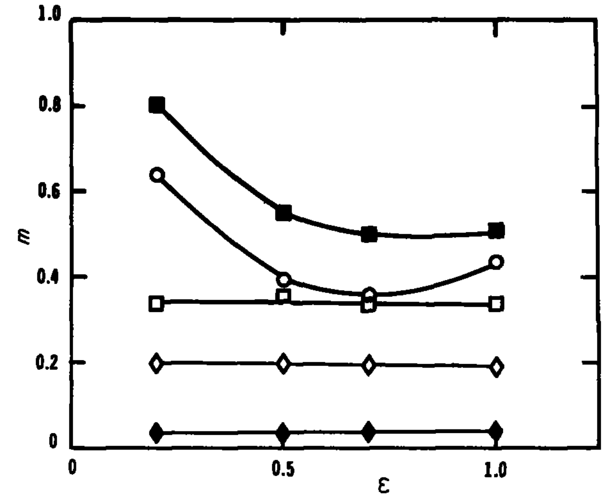

Figure 7 Plot of m against true strain at 457° C and at various strain-rates $\dot{\varepsilon}$ (sec^{-1}): (○) 5×10^{-5}, (■) 1×10^{-4}, (□) 5×10^{-4}, (◇) 1×10^{-3}, (◆) 5×10^{-3}.

as shown in Fig. 10, at a test temperature of 457° C and strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$. Extrapolation of the lines in Fig. 10 to $(\sigma/G) = 10^0$ yielded values for A equal to 5.6×10^6, 9.3×10^3 and 2.28×10^2 at strain levels of 0.5, 0.7 and 1.0, respectively. Significant decrease in the value of A occurred during the superplastic flow as a consequence of the ability of the material to strain-harden. Table III summarizes the values of m, n, p, Q and A at various levels of true strain.

4. Discussion

During high-temperature steady-state deformation the strain usually does not play any role in characterizing the flow behaviour. However, if changes in microstructure and flow behaviour persist, then the σ–$\dot{\varepsilon}$ behaviour will depend on the strain. The parameters of the constitutive relative calculated from the relation for steady-state behaviour will therefore be misleading if such changes are not taken into account. Some qualitative [4] and quantitative [5] attempts have been made to illustrate this aspect. The values of m

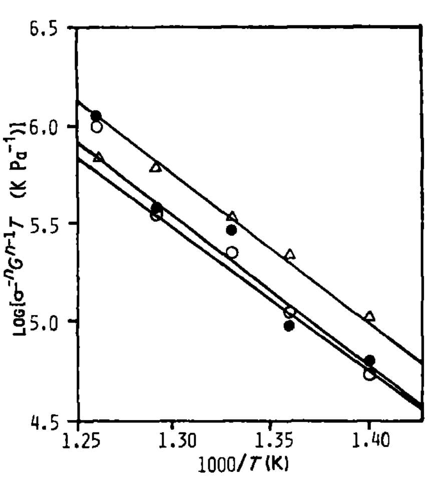

Figure 8 Activation energy plot for Region II (strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$). (△) $\varepsilon = 0.5$, $Q = 146.06\,\text{kJ mol}^{-1}$; (○) $\varepsilon = 0.7$, $Q = 172.89\,\text{kJ mol}^{-1}$, (●) $\varepsilon = 1.0$, $Q = 166.35\,\text{kJ mol}^{-1}$. Mean $Q = 161.79\,\text{kJ mol}^{-1}$.

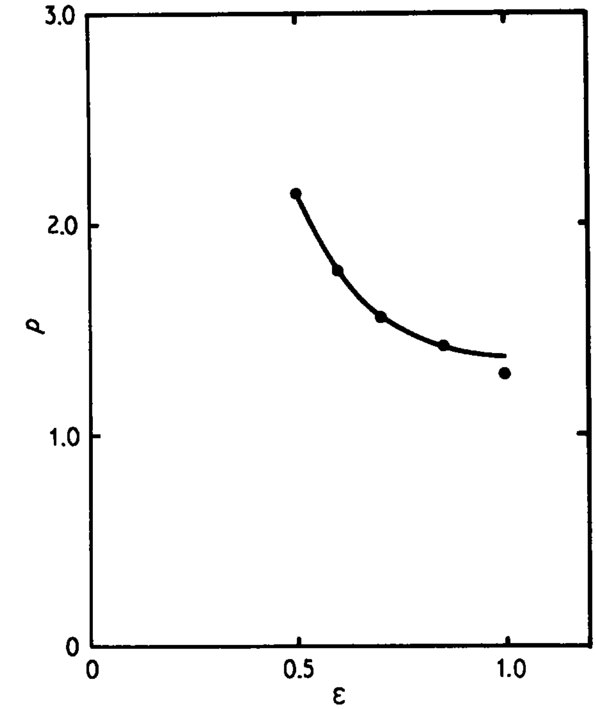

Figure 9 Plot of grain-size exponent (p) against true strain at a temperature of 457° C and strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$.

obtained from the change in strain-rate tests and the values obtained from the constant stain-rate tests are often not in good agreement. This implies that there is a large-scale change either in the defect structure or in the grain size during superplastic deformation. It is to be remembered that parameters of the constitutive Equation 1, calculated from constant strain-rate test results, are not influenced by the cumulative strain prehistory, as in the case of differential strain-rate tests. It has been found [6] that no change in the grain size takes place in this alloy during high-temperature flow. However, change in grain shape (as evidenced by grain elongation) does occur, and has been associated with increasing dislocation activity with strain. In this 7475 Al alloy, dislocations interact with chromium-rich particles in the matrix during superplastic flow, thereby producing an increase in dislocation density with increase in strain. Consequently, the alloy strain-hardens [6].

These microstructural changes are reflected by significant changes in parameters A and p (as evident from Table III). Changes in n occurred to a smaller extent. At 457 and 516° C the maximum value of m in Region II is lower than that in an earlier investigation [2]. Also, at 457° C it can be seen from Fig. 2 that as the strain rate is decreased from 1×10^{-4} to

TABLE III The variation of parameters n, p, Q and A with superplastic strain

Parameter	ε		
	0.5	0.7	1.0
m (From Fig. 9b)	0.549	0.531	0.520
n	1.82	1.88	1.92
p	2.16	1.56	1.30
Q (kcal mol^{-1})	146.06	172.89	166.89
A	5.6×10^{6}	9.3×10^{3}	2.28×10^{2}

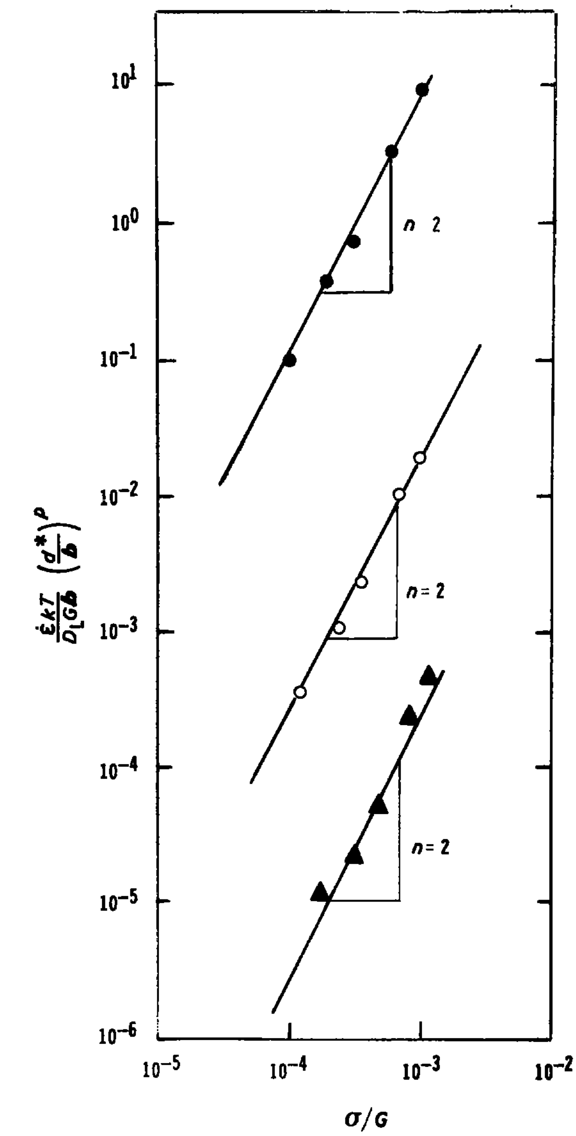

Figure 10 A plot of the non-dimensional constitutive equation for Region II at a temperature of 457° C for various strain levels: (●) 0.5, (○) 0.7, (▲) 1.0 (D_L = lattice diffusivity; d^* = initial grain size).

$1 \times 10^{-5}\,\text{sec}^{-1}$, m decreases from 0.7 to 0.42 and remains constant as the strain rate is further reduced. This value is much higher than is usually reported for Region I [7, 8]. Backofen *et al.* [9] showed that a gradually falling strain rate sensitivity could be explained by the presence of a threshold stress. It seems likely that the fall in m from 0.7 to 0.42 is due to a gradual change in the controlling deformation mechanism. At high strain rates, i.e. in Region III, the value of m is approximately 0.25. This agrees closely with values obtained by many other workers for this regime [10] and is characteristic of dislocation creep. It has been found that as the temperature was decreased and the $\log \sigma$ against $\log \dot{\varepsilon}$ curve moved to the left, the maximum value of m decreased and the strain rate at which maximum m occurred also decreased. These trends are similar to those found in nearly all other superplastic materials [10]. When m is evaluated from uniaxial tests in the strain-rate range of 1×10^{-5} to $5 \times 10^{-3}\,\text{sec}^{-1}$ (as seen from Fig. 7), m decreases with increasing strain at a test temperature of 457° C and strain rate of $1 \times 10^{-4}\,\text{sec}^{-1}$, to 0.54, 0.53 and 0.52 at strain levels of 0.5, 0.7 and 1.0; i.e. it reaches an approximately constant value after a strain of 0.5.

The activation energy at a strain rate of $1 \times 10^{-4}\,sec^{-1}$ in the temperature range 437 to 517° C increased to a very small extent with strain, as seen in Fig. 8 and Table III. The existence of a non-unique value of activation energy could be due to several possibilities. One of them is the existence of microstructural changes in terms of dislocation activity, which (through a process of strain-hardening) influences the stress term in the numerator of Equation 3. These dislocations appear to be emitted from grain boundaries [11, 12] and are generated at ledges and protrusions under large stress concentration due to grain-boundary sliding [13]. Small changes as a function of strain in the value of n ($= 1/m$, obtained from Fig. 7), which is again correlated with the dislocation activity, change the numerator of Equation 3 significantly. Most models for superplastic deformation predict the activation energy in Region II to be equal to that for the grain-boundary, or interphase boundary, diffusion. From Fig. 8, however, the Q values are close to activation for lattice diffusion in aluminium solid solutions [14]. Such a high value for activation energy is attributed to interactions between dislocation and chromium-rich dispersoids in the matrix. The chromium-rich dispersoids not only pin grain boundaries but also impede dislocation motion within the grains. Three sequential processes are expected to take place in this alloy during superplastic flow with respect to dislocations at 457° C: (a) emission of dislocations at a grain boundary, (b) overcoming of the chromium-rich particles by these dislocations in the matrix by climb, and (c) annihilation of dislocations at a grain boundary. The rate-controlling process which is the slowest of the three processes is the climb motion of the dislocations over the chromium-rich particles. This process is associated with an activation energy equal to that for lattice diffusion. An identical situation exists in an Al–Mg alloy containing chromium [15]. The substructure, particularly at higher strain, consisted of predominantly dense tangles of fairly homogeneously distributed dislocations [6] that show little tendency for being confined to well-defined slip planes. The formation of such dislocation tangles clearly reflects the ability of the dislocations to readily climb away from obstacles. An examination of the substructure shows that the plastic flow leading to the formation of the dislocation entanglement is aided by the presence of non-shearable particles present in the microstructure. The interaction of dislocations with the chromium-rich dispersoids leads to dense tangles around each particles which act as effective barriers to dislocation motion. The dislocations have to bypass these barriers by climbing over them. The increase in concentration and mobility of vacancies at elevated temperatures can accelerate dislocation climb [16] and movement of dislocations conjectured here during superplastic deformation.

From Fig. 9 it is clear that p decreases with increasing strain. This decrease cannot be explained by simply considering Equation 4. Since in this work the size of grains did not change with strain, and the stress term in Equation 4 did increase with strain due to strain hardening, hence one should anticipate an increase in the value of p with increasing strain. This is contrary to the results depicted in Fig. 9. However, in this investigation, the grains became elongated with progressive straining. The current results may possibly be explained by considering that the creep rate is a function of the diffusion path length and thus varies with grain shape as well as grain size. Raj and Ashby [17] treated the case when the grain aspect ratio deviates from the equiaxed condition in the context of diffusion creep. In the current investigation on superplasticity, the ultimate annihilation of dislocations at the grain boundaries is the recovery process, and it may indeed be influenced by the boundary condition of diffusional transport. The latter will be influenced by changes in grain shape.

The value of A can be calculated once the other parameters in Equation 1 have been determined. Table III indicates that in Region II the A value decreases with increasing level of strain. If theoretical values of p, n ($= 1/m$) and Q are assumed in Equation 1, a decrease in A at constant strain rate can only arise from an increase in the flow stress level. An increase in the flow stress was indeed observed as a consequence of strain-hardening.

The value of A in Fig. 10 was obtained by using experimental values of n, p and Q. Because of the changes in these parameters as a function of strain, changes in the A value are related to changes in the flow stress level. Table IV illustrates this point. Here the normalized values for the expressions related to grain size, activation energy and stress in the constitutive Equation 1 are compared as a function of strain. For each specimen the value of strain rate, grain size and temperature are the same. Values of n, p and Q at various strain levels are taken from Table III and used in Equation 1. A change in the value of p from 2.6 at a strain equal to 0.5 to a value of 1.26 at a strain equal to 1.0 increases the parameter $(b/d)^p$ from 8.22×10^{-11} to 1.31×10^{-6}, i.e. by a factor of 1.54×10^4. In addition the change in activation energy from $146\,kJ\,mol^{-1}$ at $\varepsilon = 0.5$ to $166\,kJ\,mol^{-1}$ at $\varepsilon = 1.0$ has reduced the parameter $\exp(-Q/RT)$ by a factor of 350. A change in the value of $n = 1.82$ at a strain of 0.5 to $n = 1.92$ at a strain of 1.0 increases the parameter $(\sigma/G)^n$ by a factor of only 1.14. The major influence on the value of A as a function of strain comes from the values of parameters $(b/d)^p$ and $\exp(-Q/RT)$. On comparing the parameters $(b/d)^p$ and $\exp(-Q/RT)$ as a function of strain in Table IV, an increase in the parameter $(b/d)^p$ is much more significant than a decrease in

TABLE IV Effect of superplastic true strain on the numerical values in Equation 1*

Parameter	Strain		
	0.5	1.0	Ratio
$(b/d)^p$	8.22×10^{-11}	1.36×10^{-6}	1.54×10^4
$\exp(-Q/RT)$	3.886×10^{-11}	1.39×10^{-12}	3.57×10^{-2}
$(\sigma/G)^n$	3.4×10^{-7}	3.9×10^{-7}	1.14

* In Equation 1: $\dot{\varepsilon} = 1 \times 10^{-4}\,sec^{-1}$, $d = 14\,\mu m$ and $T = 730\,K$.

exp $(-Q/RT)$. Changes in $(b/d)^p$ are associated with grain elongation without a significant change in the grain size. Dislocation activity in the grain interior involving glide and climb processes bring about grain elongation. It has been suggested [8] that when the number of dislocations arriving at the boundary from the grain interior increases, grains would elongate. Other microstructural features such as size of the intermetallic particles within the grain interior would be unimportant, as no change in the particle size within the grain interior occurred when the alloy was statically annealed for 144 h at 530° C. In addition, some investigators suggest [19, 20] that a difference in thermomechanical treatment yields different substructures which may be responsible for different values of A. This can also be anticipated with regard to the influence of thermomechanical treatment on the values of m and p. Because of the interrelationship amongst the various rate parameters, the exact values of A determined at various strain levels may indeed be slightly in error. However, there is no ambiguity with respect to the trend in the A value to decrease with increasing levels of strain.

5. Conclusions

1. The flow stress in 7475 Al alloy is significantly dependent on the strain during superplastic flow. Strain hardening is more predominant at 457° C than at 517° C.

2. The source of strain hardening is from interaction of dislocations with the hard chromium-rich intermetallic particles and the consequent increasing density. This gives rise to non-unique values for the parameters of the constitutive equation during superplasticity.

3. The values of the stress exponent (n) and activation energy for superplastic deformation (Q) increase only slightly with strain. The values of the grain-size sensitivity parameter (p) and structure parameter (A) decrease significantly with strain. These changes in the constitutive parameters are associated with dislocation activity within the grain interior.

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