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Activation energies for superplastic tensile and compressive flow in microduplex α/ß copper alloys

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Logarithmic stress against strain rate curves have been determined at various temperatures for a superplastic commercial α/β nickel–silver alloy strained in tension, and a laboratory prepared microduplex alloy of nominally similar composition strained in compression. The shapes of the curves were found to be affected by grain growth at high temperatures and strain softening at low temperatures. After taking these factors into account, it was apparent that with decreasing strain rate in both alloys a change in deformation mechanism occurred giving rise to a Region I of low strain-rate sensitivity. By confining activation energy (Q) measurements to temperatures at which steady-state deformation occurred, it was found that Q for Region II was very similar to that measured for grain boundary diffusion in the α phase of a nickel–silver alloy of similar composition, while Q for Region I was substantially higher than that for lattice diffusion. Values of strain-rate sensitivity and Q were found to be similar for each direction of applied stress.

1. Introduction

The method of testing of superplastic materials in which microstructural change occurs during deformation [1, 2] can affect the slope of the logarithmic stress against strain rate curve. Ideally a combination of rapid testing and stable microstructure should be used for strain rate sensitivity, m, and/or activation energy, Q, determination. In previous work [3] on a microduplex Cu–Zn–Ni (nickel–silver) alloy, it was observed that little grain growth occurred during superplastic deformation due to the effective pinning action of β particles ($\sim 20\%$ volume fraction) located at α grain boundaries. Hence, such a structurally stable alloy should provide reliable values of m and Q and help to determine whether or not a Region I exists in the logarithmic stress against strain rate curves at low strain rates.

In addition to tensile tests, axisymmetric compression tests were carried out to determine whether m and Q values were dependent on the direction of applied strain. Compression tests on superplastic materials have seldom been reported in the literature [4], despite the relevance of such tests to forming operations which utilize compressive deformation.

2. Experimental details

2.1. Materials

A commercial microduplex nickel–silver (IN836) and a laboratory prepared alloy of similar composition (IN836L) were investigated. Analysed compositions of both alloys are given in Table I. The commercial alloy was supplied in sheet form of thickness 2.5 mm by the International Nickel Company. Tensile specimens of 10 mm gauge

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TABLE I Compositions (wt%) of the alloys.

Alloy	Cu	Zn	Ni	Mn	Mg	Fe	Pb	S
IN836	45.7	38.3	15.7	0.13	0.010	0.109	0.14	0.05
IN836L	44.1	40.4	15.3	0.05	0.005	0.012	0.12	0.02

length and 5 mm x 2.5 mm gauge section were milled from this. As axisymmetric compression tests require cylindrical specimens, an alloy in round bar form was prepared in the laboratory. The preparation route involved melting and casting in air to produce an ingot of 76 mm diameter, homogenization at 750° C, extrusion at 860° C to 12.7 mm diameter bar, solution treatment at 700° C followed by water-quenching to retain α and then swaging to 5.4 mm diameter bar. A pre-annealing treatment at 200° C was followed by a full microduplex anneal at 480° C to recrystallize α and precipitate β. Compression specimens of 5.4 mm diameter (D) and heights (H) of 2.7 mm, 5.4 mm, 10.8 mm and 16.2 mm were machined from the bar.

2.2. Mechanical testing

Superplastic deformation in both tension and compression was performed in air in a furnace attached to an Instron machine capable of cross-head velocities in the range 8.33×10^{-5} to 8.33×10^{-1} mm sec^{-1}. A temperature variation of $\pm 4°$ C was measured over the central 100 mm of the furnace at ~550° C. Specimens were allowed to equilibrate for ~40 min before straining. For compressive straining, specimens were held between polished stainless steel plates located in stainless steel cages. Graphite was used as lubricant.

Superplastic behaviour in tension and compression was characterized by the measurement of strain-rate sensitivity (m value) at temperatures in the range 450° C to 575° C. Logarithmic stress against strain-rate curves were determined by the strain-rate change technique in which the cross-head velocity is suddenly changed and the load before and after the change is recorded. To avoid the high initial rate of increase of flow stress with strain in tension, specimens of IN836 were first strained to 25% elongation at an initial strain rate of $8.33 \times 10^{-4} sec^{-1}$. In the case of compressive tests, specimens of IN836L were reduced in height by 25%, at an initial strain rate of 1.67×10^{-4} sec^{-1}, in order to avoid a high initial rate of decrease of stress with strain. The lowest cross-head velocity was then selected and progressively increased to the highest value required as quickly as possible in order to minimize strain softening/hardening. However, enough time was allowed at each cross-head velocity to enable steady-state conditions of flow to operate; that is, the load reached a near constant value.

Further tests were performed at the temperature of highest m value in which specimens were deformed to a high strain in compression at various constant strain rates.

2.3. Metallography

Microstructures of specimens of IN836 and IN836L were examined using optical and scanning electron microscopy. After polishing to 0.25 μm diamond finish, the specimen surface was swab-electroetched in citric acid ($100 g l^{-1}$, 5 to 15 V, 10 to 20 sec). Grain size measurements were made on specimens deformed to various extents at the highest test temperatures of 575° C in tension and 570° C in compression. A total of ~500 grain and phase boundary intercepts were counted for each specimen with no attempt being made to differentiate between grain and phase boundaries. An average grain size was therefore measured for the duplex structure. Grain sizes are quoted as $1.75\bar{L}$, where $\bar{L}$ is the mean linear intercept.

3. Results

3.1. Tensile testing

Logarithmic stress against strain rate curves are plotted in Fig. 1. The curves have the sigmoidal shape often reported for superplastic materials, with three distinct regions of flow, particularly at higher test temperatures. An additional experimental point, (full circle) corresponding to the stress value obtained at an elongation of 25%, just prior to selection of the lowest cross-head velocity as described, has also been plotted. The height of the curve above this point represents the effect of grain growth in raising the flow stress during the time (or strain) before this particular strain rate was again selected. Alternatively, the height of the point above the curve represents the effect of a strain softening process in lowering the flow stress during the test. It can be seen that the the effect

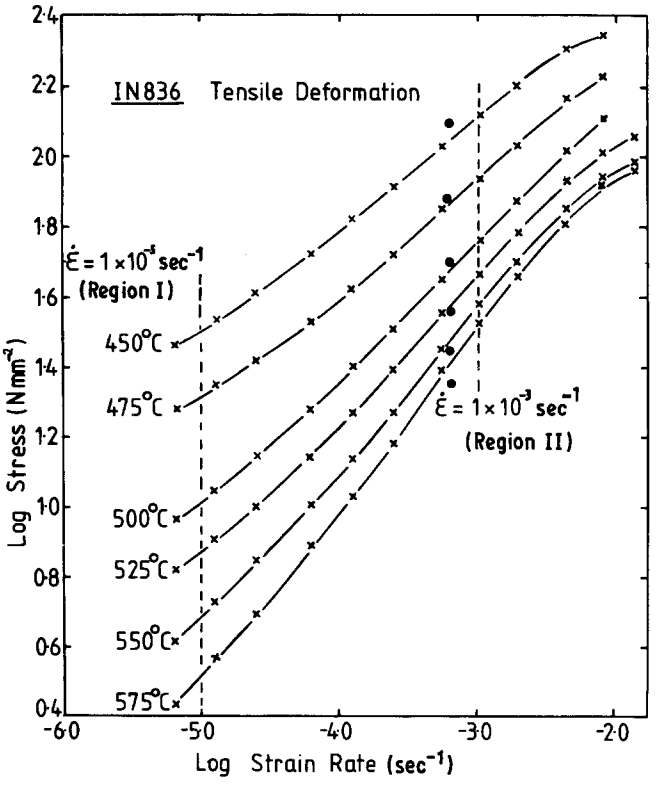

Figure 1 Stress–strain rate data at various temperatures for IN836 deformed in tension.

of grain growth is small except at the highest test temperature. Values of m determined at strain rates in Regions I and II are given in Table II.

Activation energies for deformation in Regions I and II were determined by assuming an equation of the form:

$$\dot{\epsilon} = A \exp(-Q/RT), \quad (1)$$

where $\dot{\epsilon}$ is strain rate, A is a constant, Q is activation energy, R is the gas constant and T is temperature. Q was calculated by measuring stress levels corresponding to lines of constant strain rate (Fig. 1) and using:

$$Q = \frac{R}{m}\left(\frac{\mathrm{d}\ln\sigma}{\mathrm{d}(1/T)}\right)_{\dot{\epsilon}}. \quad (2)$$

The value of Q was obtained from the slope of an Arrhenius plot (Fig. 2) by substituting an appropriate value of m for the region of interest.

Mohamed *et al.* [5] have criticized Equation 1 in that it does not include the effect of any variation of shear modulus with temperature. Unfortunately, there are no data available for an alloy of composition sufficiently close to that of IN836/IN836L to make a reliable estimate of this effect. However, values of Q previously obtained using Equation 2 have often been similar to those obtained from an analysis which includes the effect of shear modulus [5].

3.2. Compression testing

Interfacial friction at the ends of the test cylinder contacting the platen resulted in measured values

TABLE II Values of strain rate sensitivity index, m, for IN836 in tension.

Temperature (°C)	Region I ($\dot{\epsilon} = 1 \times 10^{-5}$ sec^{-1})	Region II ($\dot{\epsilon} = 1 \times 10^{-3}$ sec^{-1})
450	0.27	0.33
475	0.25	0.34
500	0.32	0.40
525	0.33	0.43
550	0.40	0.50
575	0.46	0.56
Average	0.35	0.45

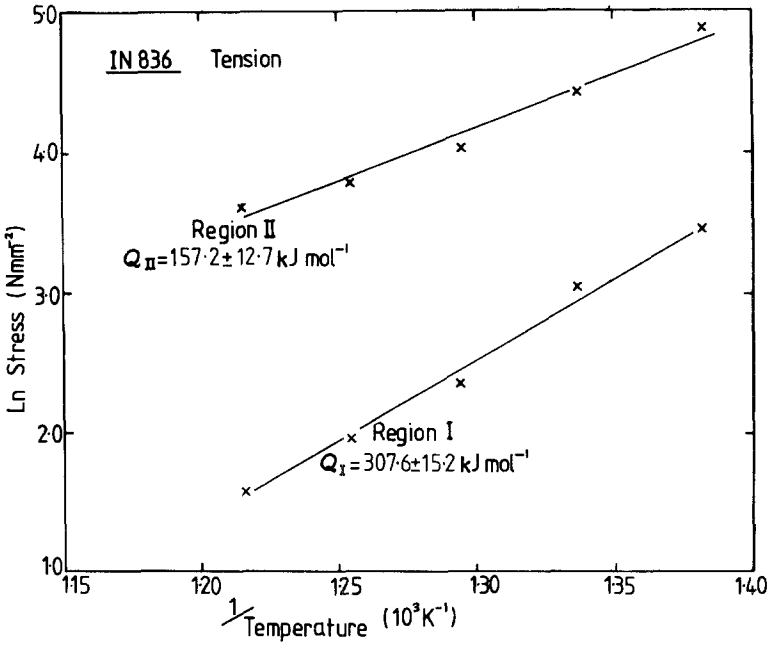

Figure 2 Logarithmic plots of stress against reciprocal temperature for IN836 tested in tension.

of stress having a frictional contribution. The Cook and Larke procedure [6] was adopted to correct for this effect. To do this, true stress–true strain curves for specimens of various D/H ratios, deformed at a constant strain rate and temperature, were plotted (Fig. 3). The stresses corresponding to give strain were then plotted against D/H in Fig. 4. These constant strain curves were extrapolated to obtain the stresses required to produce given strains in an infinitely high cylinder. Cook and Larke [6] assume that such stresses represent the resistance to homogeneous compression, although Polakowski disputes this [7]. However, he concludes that the stresses represent a minimum resistance to compression and have a well-defined physical significance. Such a curve for $D/H = 0$ is plotted in Fig. 3. The specimens used to obtain the data in Figs. 3 and 4 are shown in Fig. 5. A curve of true stress against true strain for a specimen of IN836 strained in tension at the same constant strain rate is also plotted in Fig. 3.

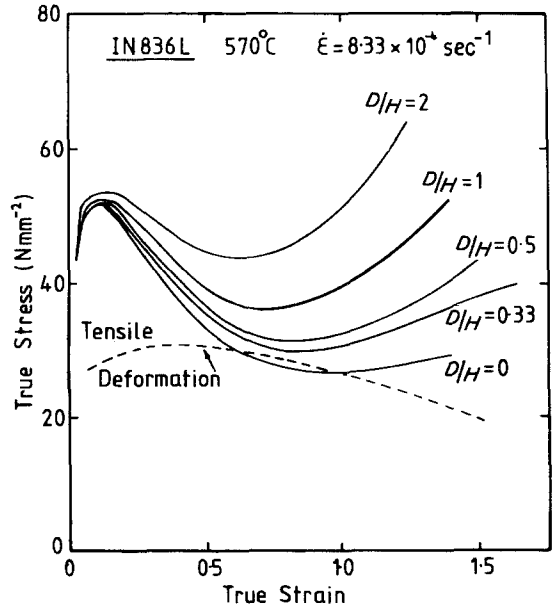

Figure 3 True stress against true strain curves for specimens of IN836L of various D/H ratios deformed in compression at 570° C.

Specimens of D/H ratio 1.0 were used to obtain logarithmic stress against strain rate data. The frictional force at each strain at which the cross-head velocity was changed was estimated from Fig. 3, and then subtracted from the measured stress. In this way corrected logarithmic stress against strain rate curves were obtained using the assumption that the frictional force was independent of strain rate and temperature for a given strain, and these are plotted in Fig. 6. The curves were similar in shape to those obtained for IN836 in tension except that the cross-head velocities used were too low to allow Region III to be observed. Values of m determined from these curves are given in Table III.

Activation energies for deformation in Regions I and II were determined using stress levels corresponding to the lines of constant strain rate shown in Fig. 6. Arrhenius-type plots are shown in Fig. 7.

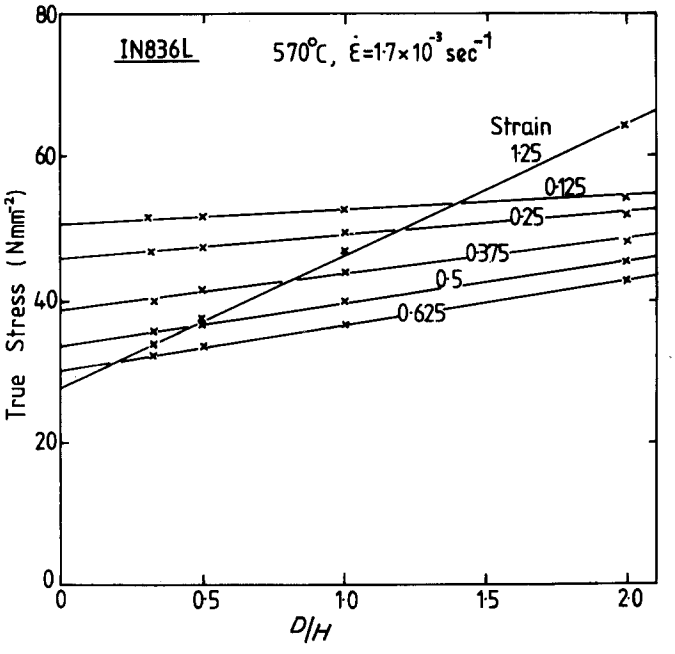

Figure 4 Plots of true stress against D/H ratio for different compressive strains in IN836L.

3.3. Metallography

Grain size measurements at the highest test temperature revealed that little grain growth occurred during tensile and compressive straining. The as-received *average* grain size of IN836 was 1.8 μm with a volume fraction of β of $\sim$0.15 to 0.2. The diameter of the average β particle was $\sim$1 μm and therefore was much smaller than the diameter of the average α grains. After equilibration for 40 min at 575° C and tensile deformation to the position at which the first datum point was recorded in Fig. 1, the average grain size had increased marginally to 1.9 μm. At the position at which the third datum point was recorded the grain size was 2.0 μm. After the final velocity increment was made the grain size had further increased to 2.5 μm. It was apparent that grain growth was mainly confined to movement of α/α grain boundaries away from β particles and that the β phase size remained stable (Fig. 8). The modest rate of grain growth recorded at the highest test temperature would suggest that coarsening is negligible at lower test temperatures.

The laboratory prepared alloy, IN836L, had an

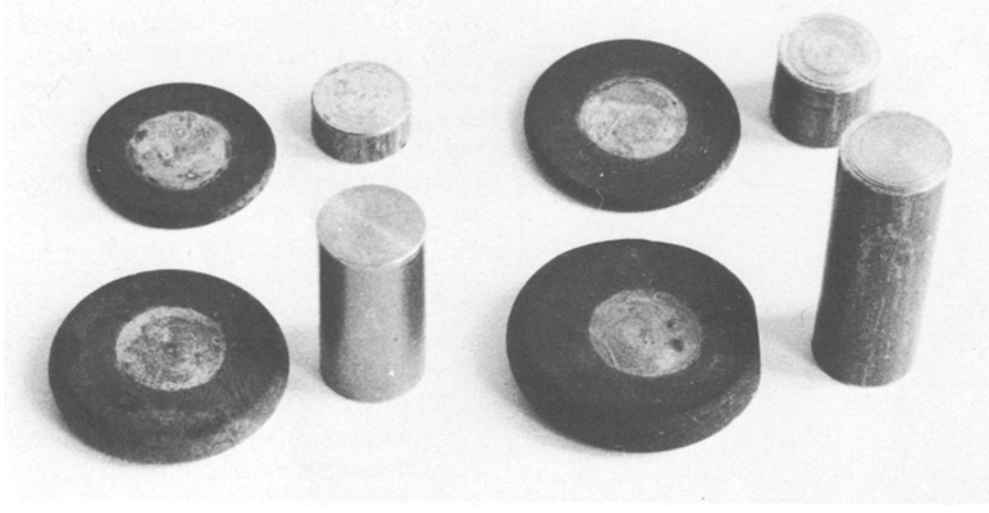

Figure 5 Compression specimens of IN836L of various D/H ratios before and after deformation.

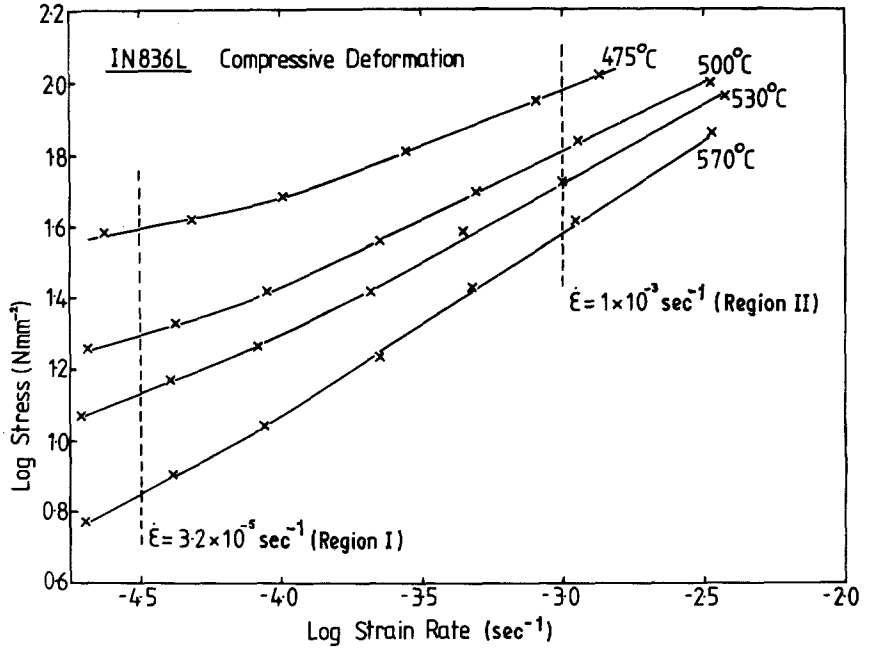

Figure 6 Stress–strain rate compression data at various temperatures for IN836L after correction for friction.

average grain size of 2.8 μm in the microduplex annealed condition and a β volume fraction of ~0.35. The sizes of the α and β phases in this alloy were very similar. After equilibration at 570° C for 40 min, and compressive deformation to the position at which the first datum point was recorded in Fig. 6, the grain size had increased to 3.4 μm and a similar rate of coarsening was observed in both phases. After the final velocity increment was made the grain size had further increased to 4.0 μm. However, it was noted that the grain size increased from the central regions of the specimen, as shown in Fig. 9a (for which these measurements are quoted) to the barrelled regions, where circumferential tensile stresses occurred (Fig. 9b).

4. Discussion

Figs. 1 and 6 show that Regions I and II exist in both tensile and compressive deformation in the nickel–silver alloys tested. It can be seen that there is a gradual transition between each region at each test temperature leading to a continuous change of shape. This might be expected if different deformation mechanisms competed near the boundaries between the different regions. In addition, the measured values of m decrease with decreasing temperature. The latter observations have been reported in other alloys systems, including α/β brass [8], Al–6Cu–0.4Zr [9], zinc–aluminium eutectoid [10] and lead–tin alloys [11], although some workers have reported sharp changes of slope and have also observed that temperature has little or no effect on m [12, 13]. Reduction of strain rate sensitivity with decreasing temperature is probably a reflection of decreasing amounts of grain boundary sliding accommodated by diffusional or dislocation processes and a corresponding increase in slip and/or recrystallization processes.

TABLE III Values of strain rate sensitivity index, m, for IN836L in compression

Temperature (° C)	Region I ($\dot{\epsilon} = 3.2 \times 10^{-5}$ sec^{-1})	Region II ($\dot{\epsilon} = 1 \times 10^{-3}$ sec^{-1})
475	0.18	0.30
500	0.24	0.40
530	0.34	0.45
570	0.44	0.51
Average	0.30	0.41

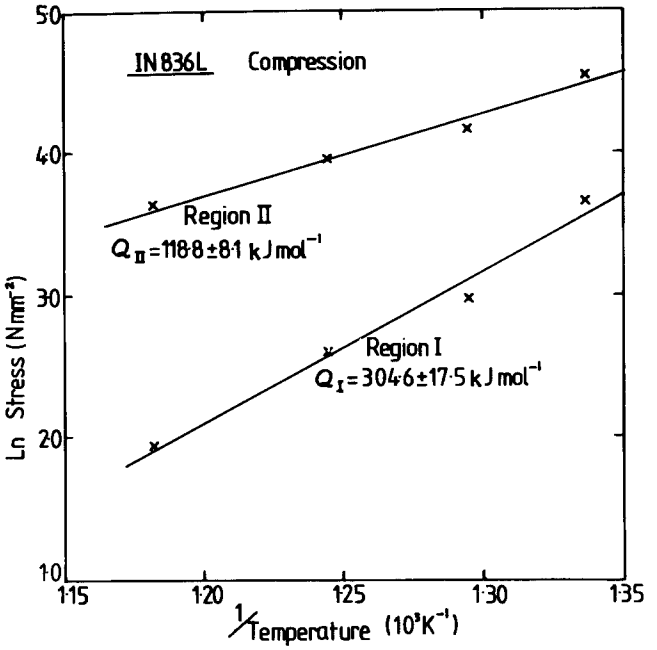

Figure 7 Logarithmic plots of stress against reciprocal temperature for IN836L tested in compression.

By adopting a rapid strain rate change technique for an alloy system having high structural stability, the effect of grain growth on the shape of the logarithmic stress against strain rate curves was minimized. However, it can be seen that, for tensile deformation, the 575° C curve is significantly displaced to a position above the initially measured flow stress and therefore should be ignored for Q determination. At temperatures of between 500 and 550° C the initial flow stress coincides with the stress–strain rate curves, but at lower temperatures a significant strain softening process causes a divergence. In addition, m values fall to 0.34 and 0.33 in Region II and the general level of flow stress increases dramatically. This loss of superplasticity is reflected in measured Q values. For the temperature regime 500 to 550° C Q values for Regions I and II (Q_I and Q_{II}) were 234.8 ± 4.6 kJ mol^{-1} and 98.1 ± 1.4 kJ mol^{-1}, respectively. However, for the temperature regime 450 to 550° C, Q_I and Q_{II} give the much higher values of 307.6 ± 15.2 kJ mol^{-1} and 157.2 ± 12.7 kJ mol^{-1}, respectively. A similar result occurs in compression with Q_I and Q_{II} for the regime 550 to 570° C determined as 234.8 ± 19.8 kJ mol^{-1} and 110.0 ± 3.0 kJ mol^{-1}, respectively. However, for the interval 475 to 570° C values of Q_I and Q_{II} are higher at 304.6 ± 17.5 kJ mol^{-1} and 118.8 ± 8.1 kJ mol^{-1}, respectively. It is only over the former narrow temperature range, 500 to 550° C, there-

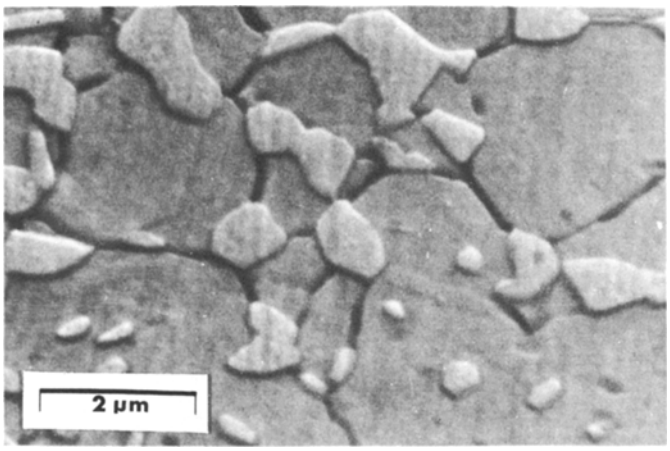

Figure 8 Microstructure of IN836 after a strain rate change test in tension at 575° C.

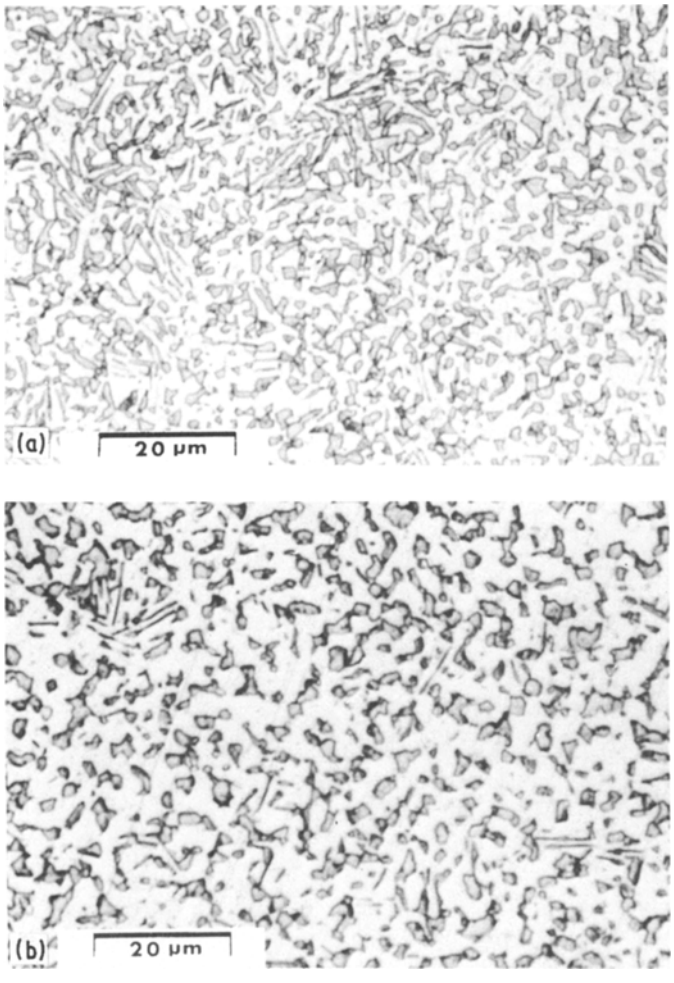

Figure 9 Microstructure of IN836L after compression to a strain of at 570° C in (a) central region and (b) barrelled region of compression specimens.

fore, that Q values can be measured with confidence and in fact, over this range values for tensile and compressive deformation are quite similar.

Activation energies for lattice diffusion, Q_L, of copper, zinc and nickel in the α phase of nickel–silver of similar composition to IN836 and IN836L, have been determined to be 173 ± 13, 184 ± 2 and $189 \pm 1.5\,kJ\,mol^{-1}$, respectively [14]. There are no data available of activation energies for grain boundary diffusion, Q_B, in the α phase, or for diffusion in the β phase. However, as the bcc lattice of the β phase is relatively open compared to the fcc lattice of the α phase, it should have lower Q_L and Q_B. Higher rates of self-diffusion have, in fact, been observed for the β phase in α/β brass [15] and values of Q_L in β brass [16] are lower than Q_L in the α phase of nickel–silver.

Schelleng and Reynolds [17] determined Q_{II} for tensile deformation of IN836 to be 144.6 kJ mol^{-1}. They concluded that, as values of Q_L for the α phase were greater than this, then creep deformation in the β phase was the rate controlling mechanism. However, the low volume fraction of β phase and the fact that it exists as "islands" in a matrix of α grains in this alloy make this unlikely. If it is assumed for the α phase in IN836 that $Q_B \sim 0.6\,Q_L$, then Q_B will be $\sim 109\,kJ\ mol^{-1}$, which is close to the experimentally determined values of Q_{II} for the temperature regime 500 to 550° C in tension and for the temperature range 500 to 570° C in compression.

Padmanabhan and Davies [4] found that logarithmic stress against strain rate curves for the superplastic Al–33% Cu eutectic coincided for both tensile and compressive deformation. Hence activation energies were also the same, and it was

concluded that there was no change in the deformation mechanism on changing the sign of the applied stress. Logarithmic stress against strain rate curves for IN836L in compression lie at slightly higher stress levels than those for IN836 in tension. A greater initial grain size and/or a difference in cavitation behaviour on changing the direction of applied strain may account for this. However, values of Q_I and Q_{II} are quite similar for both materials.

It can be seen from Fig. 3. that compression testing of IN836L produces a high initial decrease of stress with strain before steady-state deformation takes place. This contrasts markedly with the slight initial increase of stress with increasing strain during tensile deformation of IN836. A similar phenomenon has been observed in compression testing of superplastic Al–33% Cu eutectic [4], even though the apparent effect was greatly reduced by an increasing strain rate during the test, necessarily incurred by the application of constant cross-head velocities. However, it has also been observed that flow stress increases linearly with strain during constant strain rate tests [18] and in constant velocity tests [1] in tension for the same alloy. The apparent initial decrease in flow stress with increasing strain would therefore seem to be merely a product of the compression test itself rather than a material phenomenon, and may be in part due to a high initial sticking friction before sliding friction conditions operate. However, a rapid rate of dynamic recrystallization after the peak stress would produce the same effect. This may also be the origin of the strain softening process which occurs in IN836 in tension at lower test temperatures. It may be noted that flow stresses in both tension and compression for IN836 and IN836L become very similar after a strain of 0.5 at 570° C.

The rapid increase of stress with compressive strain at higher strains is an effect of friction, and in the corrected curve for compressive deformation of a specimen of $D/H = 0$ this effect is almost eliminated.

In general, Q is found to increase from a value close to Q_B in Region II to a value close to Q_L in Region I [5, 10, 19], although some workers have reported $Q_I \sim Q_{II}$ in zinc–aluminium eutectoid [20]. Most workers report a sudden change in Q on crossing the I/II boundary, although Maulik and Padmanabhan [21] have shown that there is, in fact, a gradual change in Q with strain rate in lead–tin eutectic, zinc–aluminium eutectoid and aluminium–copper eutectic.

Shei and Langdon [19] determined Q_I and Q_{II} for a Cu–2.8 Al–1.8 Si–0.4 Co alloy as 179 kJ mol^{-1} and 144 kJ mol^{-1}, respectively, which compares favourably with Q_L and Q_B for pure copper (203 kJ mol^{-1} and 120 kJ mol^{-1}, respectively). Langdon [22] has provided evidence for Region I over a wide range of grain sizes and temperatures for this alloy which would preclude any influence of grain growth. His observations are in essential agreement with the behaviour of IN836 over the temperature range 500 to 550° C. Further work by Arieli *et al.* [23], at low strains in the same alloy, has shown that Region I under such conditions cannot be accounted for by grain growth, but is, in fact, the result of dislocation climb-controlled creep. Determination of Q_I and Q_{II} by these workers yielded very similar results to those quoted by Shei and Langdon [19].

In the present work Q_I is substantially higher than Q_L for the α phase. A similar result was obtained by Menzies *et al.* [24] for nickel-base superalloy IN-100. The authors pointed out that activation energies for steady-state creep are also often much higher than for volume diffusion in nickel-base alloys.

The reason why the determined value of Q_I is so high is difficult to explain. Measured values of Q for grain boundary migration in impure materials have frequently been found to be greater than Q_L [25] and it has been suggested [26] that particles which retard boundary migration could be responsible. The activation energy for grain boundary migration in commercial purity α/β brass has been determined to be ~ 260 kJ mol^{-1} [27], which is of similar magnitude to the values of Q_I determined in the present work. In the case of the nickel–silver alloys the β particles are located at α grain boundaries and act as very efficient pinning agents.

Arieli *et al.* [23] conclude that in Region I lattice diffusion will control climb of dislocations around obstacles impeding their motion in the essentially single phase copper-based alloy. The situation in α/β nickel–silver is more complex and dislocation climb must take place in interphase boundaries. Gittus [28] indicates that a threshold stress will operate, due to pinning interaction between interphase boundary superdislocations and dislocated boundary ledges. The activation energy is that for interphase diffusion.

As discussed previously Q_I values for both alloys are, in fact, very similar for an equivalent temperature regime. This suggests that the precise values do have significance. However, it may be difficult to interpret a particular Q value in terms of a rate-controlling mechanism if several processes are operating concurrently. In addition, in multicomponent systems with more than one phase, there are a large number of possibly relevant diffusion coefficients, including those for various types of phase and grain boundary.

5. Conclusions

(1) Evidence for the existence of Region I in microduplex α/β nickel–silver alloys has been found using a procedure which minimizes the influence of strain hardening/softening on the shape of the logarithmic stress against strain rate curve. However, it was only over a relatively narrow temperature regime that these parameters did not effect Q value determination.

(2) A large variation of m value with temperature occurs in nickel–silver in Regions I and II. In addition these Regions do not have distinct boundaries with respect to strain rate. This would occur if there were two or more competing deformation mechanisms within the transition zone.

(3) Changing the sign of the applied strain did not significantly effect the shape of the logarithmic stress against strain rate curve, the stress levels involved, or measured m and Q values other than would have been expected from small differences in initial microstructure between the alloys used for tensile and compressive tests.

(4) The value of Q_{II} obtained compared favourably with reported values of Q_B, assuming that $Q_B \sim 0.6Q_L$. The value of Q_I was substantially higher than Q_L and the general observation of $Q_I > Q_{II}$ was found to be true for nickel–silver.

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