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The relationship between intergranular cavitation and superplastic flow in an industrial copper base alloy

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Intergranular cavitation has been observed during the superplastic deformation of a fine grain sized (1 μm) Cu–2.8% Al–1.8% Si–0.4% Co alloy when tested at temperatures ⩾500° C. High voltage electron microscopy revealed that the cavities could be nucleated at twin boundary/grain boundary intersections. The maximum elongation occurs at a higher temperature than that of the maximum strain-rate sensitivity and this is explained in terms of grain-boundary migration, at the higher temperature, which restricts the cavitation process. This explanation was put forward on the basis of texture analysis which was used to study the deformation characteristics at the temperatures of maximum elongation and strain-rate sensitivity. The final fracture mode is shown to change with test temperature: (i) at 400° C no cavitation occurs and fracture is by ductile rupture, (ii) at 500 to 550° C cavitation occurs and fracture is by the interlinkage of voids by an intergranular void sheet (IVS) mechanism and (iii) at 800° C grain growth occurs and fracture occurs by the propagation and interlinkage of grain-boundary cracks along the grain boundaries.

1. Introduction

At temperatures $\geqslant 0.5\ T_m$ (where T_m is the absolute melting temperature) certain alloys have been shown to deform in a manner which closely resembles that of hot polymers. Alloys of this type thin down uniformly along their length and do not neck-down like normal ductile metals. This behaviour is known as superplastic deformation and it is obtained in alloys with a grain or phase size < 10 μm. The maximum superplastic deformation of a particular alloy occurs under test conditions which give the maximum strain-rate sensitivity (m) as defined by

$$\sigma = \sigma_0 + k\dot{\epsilon}^m \tag{1}$$

where σ is the flow stress, k is a constant and $\dot{\epsilon}$ is the strain-rate.

The models of superplasticity, based on grain-boundary sliding are, at present, the most successful in explaining experimental observations. These models are:

(i) a combination of grain-boundary sliding and diffusion creep;

(ii) a combination of grain-boundary sliding and grain-boundary migration;

(iii) multiple combination of grain-boundary sliding, grain-boundary migration and localized dislocation motion by glide and/or climb.

Alden [1] and Dunlop and Taplin [2] present direct evidence that grain-boundary sliding has occurred during superplastic deformation. Grain-boundary sliding is also considered to be the mechanism by which intergranular cavities form during creep. The classical model of cavity nucleation is that grain-boundary sliding concentrates stress at grain-boundary irregularities (e.g. particles, ledges, the intersection of a twin boundary with a grain boundary) which results in the decohesion of the boundary/irregularity interface. Cavitation is not generally observed in superplastic alloys and it appears to occur only in copper base alloy [3-6].

Copper base alloys are particularly prone to cavitation and exhibit cavitation under a large number of high temperature treatments. To date there is no conclusive mechanism which explains this phenomena in copper alloys but it seems that the concentration of impurities in the grain boundary offers the best explanation.

Sagat *et al.* [4] have discussed measurements of ductility and show that the maximum superplastic ductility occurs when the elongation is a maximum but the reduction in area is not a maximum, i.e. localized necking has not occurred. This indicates that the maximum superplastic ductility occurs when no necking has taken place and that the final fracture is "brittle" in nature. The present results will be discussed in terms of the elongation as it has been shown for a copper base alloy [4] that the maximum elongation occurs at the maximum ductility. In order to confirm this for the present alloy measurements of elongation before the onset of necking would have to be carried out.

A previous note has established the main superplastic characteristics of flow in Alloy 638 [7] and this work has been extended to examine: (i) why the maximum elongation does not occur at the test conditions which give maximum m; (ii) where intergranular cavities nucleate; (iii) the final fracture mode at different test temperatures.

2. Experimental

The material studied is an industrial copper base alloy (Coronze, CDA 638; 95% Cu; 2.8% Al, 1.8% Si, 0.4% Co) manufactured by Olin Corporation. The as-received material was 1.3 mm thick hot-rolled plate which had been thermomechanically treated [8] to give a fine grain size (1 μm). Flat tensile specimens were machined from this plate with their tensile axis in the rolling direction. The specimens used had the following dimensions: 12.7 mm gauge length, 9.53 mm gauge width, 1.3 mm gauge thickness.

Tensile tests were carried out under different conditions of strain-rate and temperature on an Instron Universal Testing Machine fitted with a push-button selector gear box. Test temperature was maintained by a three-zone, split, vertical furnace which was controlled to a tolerance of ± 3°C over a length of 150 mm. The specimen temperature was measured by a thermocouple placed in contact with the specimen gauge length. All tests were carried out in air.

The strain-rate sensitivity, m, as defined by Equation 1 was measured by subjecting a single specimen to a series of changes in cross-head speed. This resulted in stepped load versus time plots from which m was calculated by the method outlined by Backofen *et al.* [9]. Specimens tested at a constant strain-rate were sectioned for optical metallography and fracture surfaces were preserved for scanning electron microscopy.

A Quantimet (QTM) Image Analysing Computer was used to determine the area fraction of cavities present after deformation to different strains. The area fraction of cavities was measured in two hundred separate areas along the gauge length of the specimen. From these measurements the average area fraction was calculated. The average length of the microcracks was determined for directions parallel to and perpendicular to the stress axis. For each direction two hundred cracks were measured by a scale placed on the projection screen of the optical microscope. From these results the average aspect ratio (A_r) of the cracks was calculated from: A_r = (the average length parallel to the stress axis)/(the average length perpendicular to the stress axis). These measurements were carried out on specimens pulled to fracture.

Texture analysis of tested specimens was carried out on a Philips X-ray unit fitted with a rotating goniometer and oscillating X-ray detector. The specimens used were too small for a complete analysis to be made and it was only possible to scan from the rolling direction to the normal direction. From this analysis the change in intensity of the texture was obtained. The analysis was carried out to determine the orientation of the (1 1 1) type planes.

Transmission electron microscopy (TEM) was carried out on the as-received and tested material. Both 100 kV and 1 MV electron microscopy was used. Specimens for TEM were prepared by punching out 3 mm diameter discs from 0.254 mm thick material and then electro-jet polishing with $\frac{1}{3}$ nitric acid: $\frac{2}{3}$ methanol electrolyte at − 10°C. The discs obtained by punching were not "cupped" which indicates that the specimens were not deformed by the punch.

3. Results

A 100 kV transmission electron micrograph of the as-received material is presented in Fig. 1.

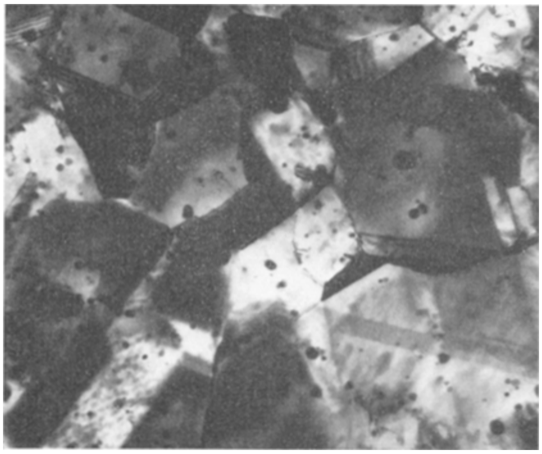

Figure 1 A 100 kV transmission electron micrograph of the as-received material. Shows extensive twinning and a non-uniform precipitate distribution (× 18 200).

TABLE I The influence of temperature on the strain-rate sensitivity for a strain-rate of 3.9×10^{-2} min^{-1}

Temperature (° C)	Strain-rate sensitivity (m)
400	0.25
450	0.35
500	0.46
550	0.34
600	0.32

TABLE II Influence of test temperature on elongation (%) and aspect ratio of micro-cracks after pulling to fracture at 3.9×10^{-2} min^{-1}. A t-test showed that the aspect ratios, except those at 500 and 550° C, were different with 95% confidence

Temperature (° C)	500	550	600	700	800
Elongation (%)	215	318	284	93	40
Aspect ratio	1.19	1.28	0.9	0.69	0.53

This shows no dislocation sub-structure with a high density of twins and precipitate distributed on the grain boundaries and in the grains. Table I records the influence of temperature on the strain-rate sensitivity (m) and percent elongation at fracture for a strain-rate of 3.9×10^{-2} min^{-1}. These results show that the maximum m (0.46) occurs at 500° C whereas the maximum elongation occurs at 550° C. It has previously been established [7] that $m = 0.46$ is the maximum value of m for this material. The aspect ratio of the micro-cracks as a function of elongation (%) is recorded in Table II, where it is observed that the maximum elongation corresponds to the highest aspect ratio. A statistical t-test was carried out in order to determine whether or not the A_r values were significantly different. This analysis showed that the A_r values for 500 and 550° C were not significantly different but all the other A_r values were significantly different with 95% confidence. Fig. 2a to d are optical micrographs of typical areas in the gauge length of specimens pulled to fracture at different temperatures. From these micrographs the change in aspect ratio of micro-cracks with test temperature can be observed. The fracture surfaces of these specimens are presented in Fig. 3a to d: these are scanning electron micrographs and indicate a change in the final fracture mode for the different test temperatures studied.

The influence of strain (% elongation) on the extent of cavitation at 500 and 550° C is recorded in Fig. 4. For both temperatures the extent of cavitation is observed to increase with increasing strain. Fig. 5a and b record the change in texture as a function of strain for test temperatures of 500 and 550° C, respectively. These results show that the strength of texturing is decreased as the strain is increased.

1 MV transmission electron micrographs of specimens strained to 7.7 and 123% elongation at 500° C are presented in Figs. 6 and 7 respectively. These micrographs show a build-up of a dislocation substructure as the elongation is increased. Fig. 6 shows a cavity at the junction of a twin boundary with a grain boundary.

4. Discussion

Most electron micrographs of superplastically deformed alloys show that the dislocation density is not markedly increased above that of the annealed alloy. The electron micrographs of the as-received material (Fig. 1) and the superplastically deformed material (Figs. 6 and 7) show that the as-received material (which had a final anneal of 1 h at 590° C [8]) had a very low dislocation density (Fig. 1). This was not increased by deformation up to − 7% elongation (Fig. 6) but was markedly increased by deformation up to − 120% elongation (Fig. 7). Superplastic deformation has been shown to be a result of a grain-boundary sliding/grain-rotation mechanism which results in grains changing their neighbours [10, 11]. In the present material the dislocation substructure (Fig. 7) at relatively high

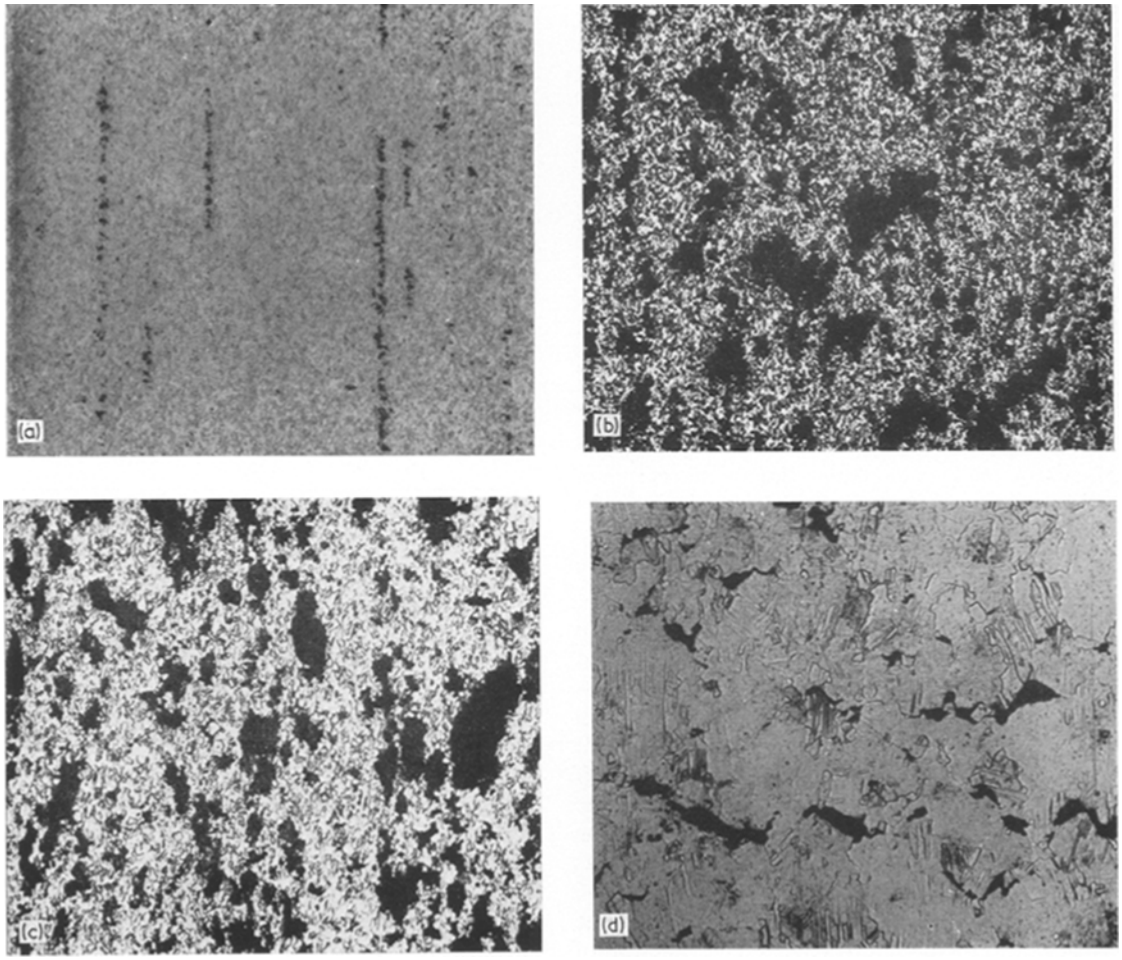

Figure 2 (a) In this series of micrographs the stress axis is in the vertical direction. Typical area of the gauge length of a specimen pulled to fracture at 400° C and 3.9×10^{-2} min^{-1}. No cavitation present ($\times$ 93). (b) Typical area of the gauge length of a specimen pulled to fracture at 500° C and 3.9×10^{-2} min^{-1}. Shows extensive grain-boundary cracking ($\times$ 93). (c) Typical area of the gauge length of a specimen pulled to fracture at 550° C and 3.9×10^{-2} min^{-1}. Shows extensive grain-boundary cracking ($\times$ 93). (d) Typical area of the gauge length of a specimen pulled to fracture at 800° C and 3.9×10^{-2} min^{-1}. Shows grain-boundary cracks and an increase in grain size ($\times$ 93).

elongations indicates that intragranular deformation has made a significant contribution to the overall deformation. This observation indicates that the grain-boundary sliding/grain-rotation mechanism has been inhibited due to the presence of grain-boundary particles which will restrict grain-boundary mobility and to intergranular cavities which will reduce the area of grain boundary on which grain-boundary sliding can take place.

In a parallel publication [12] the present authors have shown that intergranular cavities nucleate at grain-boundary particles when coarse grain sized Alloy 638 is tested under creep conditions. However, in the present condition Alloy 638 does not exhibit a preferential grain-boundary precipitate (Fig. 1) and in order to account for the high density of cavitation, cavities must nucleate on grain boundaries at other sites as well as at particles. From Fig. 1 it is observed that this alloy exhibits a high density of twins and it was decided to use 1 MV TEM in order to establish whether cavities could nucleate at the intersection of a twin boundary and a grain boundary. An extensive examination was not carried out in order to determine the

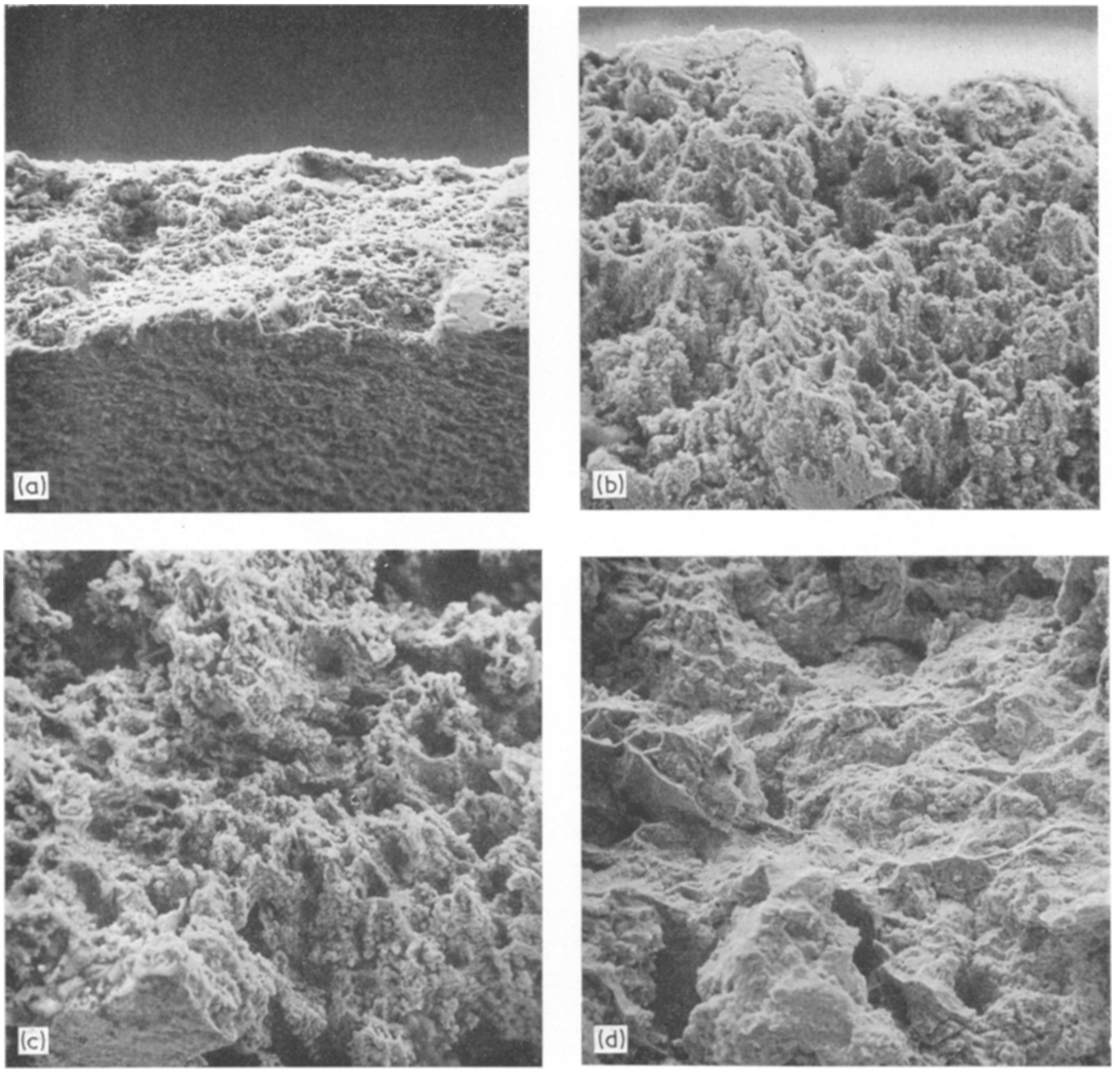

Figure 3 (a) Fracture surface of specimen shown in Fig. 2a. Shows ductile fracture (× 189). (b) Fracture surface of specimen shown in Fig. 2b. Shows areas of ductile fracture between adjacent cracks (× 150). (c) Fracture surface of specimen shown in Fig. 2c. Shows areas of ductile fracture between adjacent cracks (× 177). (d) Fracture surface of specimen shown in Fig. 2d. Shows an intergranular fracture (× 167).

frequency of cavity nucleation at grain boundary/twin boundary intersections but Fig. 6 shows that cavities can nucleate at such sites. The high density of cavitation can, therefore, be explained by considering cavities to nucleate at twin boundary/grain-boundary intersections and at grain-boundary particles. Intergranular cavity nucleation is widely accepted to be a result of grain-boundary sliding, which is also considered to be important in superplasticity [10, 11]. From Fig. 2a to d it is observed that cavitation only occurs at temperatures $\geqslant$ 500° C and this suggests that for the strain-rate used grain-boundary sliding is only operative at temperatures $\geqslant$ 500° C.

The reason why the maximum elongation does not occur at the same temperature as the maximum m will now be discussed. It must be remembered that, as pointed out in the introduction, the maximum elongation does not necessarily mean that we have maximum ductility and it may well be that the maximum ductility

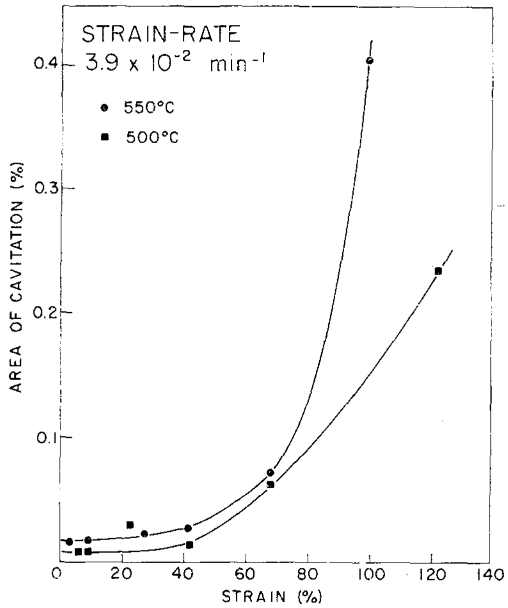

Figure 4 The influence of strain (%) on the area of cavitation in specimens tested at 500 and 550°C at 3.9×10^{-2} min^{-1}. For both temperatures the area of cavitation is observed to increase with strain.

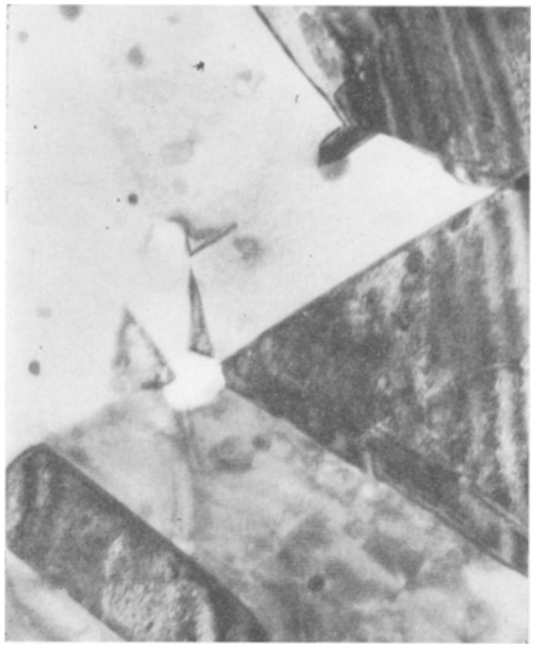

Figure 6 A 1 MV transmission electron micrograph of a specimen strained to 7.5% elongation at 500°C at 3.9×10^{-2} min^{-1}. An intergranular cavity is observed on the intersection of the twin boundary with the grain boundary (× 32 000).

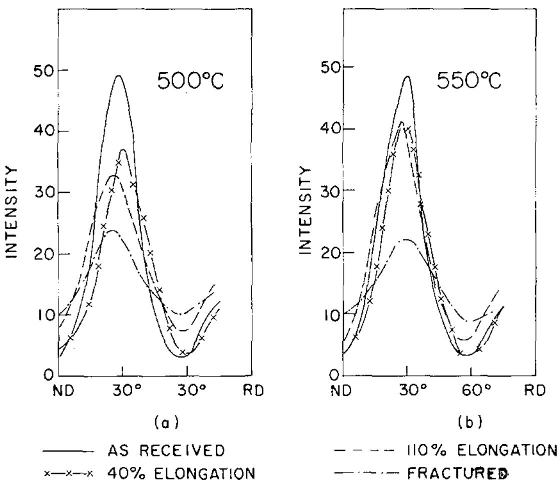

Figure 5 (a) and (b) These figures show the breakdown in texture as the elongation is increased for test temperatures of 500 and 550°C.

and maximum m both occur at the same temperature. The cavitation characteristics at 500 and 550°C are recorded in Fig. 4 where it is observed that the cavitation is more extensive at 550 than at 500°C for a given strain. This observation does not explain why the percent elongation is greater at 550 than at 500°C. However, the texture analysis (Fig. 5a and b) shows that the texture starts to break down at lower elongation at 500 than at 550°C. The change in texture at 500°C can be explained in terms of extensive grain-boundary sliding and grain rotation whereas at 550°C some grain-boundary migration has occurred which has restricted the breakdown of the texture by restricting grain-boundary sliding and grain rotation. The influence of this grain size instability on the cavitation process will be that cavity nucleation will be inhibited due to the stress concentrations being relaxed by the grain-boundary migration. To verify this point a further study of the cavitation behaviour at 500 and 550°C would have to be carried out for elongations $< 60\%$. The preceding argument would suggest that higher elongations would be required to nucleate cavities at 550 than at 500°C. The result which shows that the maximum elongation does not occur at the test conditions which give the maximum value of m indicates that in superplastic alloys which exhibit inter-

Figure 7 A 1 MV transmission electron micrograph of a specimen strained to 123% elongations under the same conditions as Fig. 6. This micrograph shows that a dislocation sub-structure has been formed as a result of the superplastic deformation (× 17 400).

granular cavitation the test conditions must be optimized in order to obtain the best balance between a high m-value and restricted cavitation. Further experimental work would have to be carried out in order to establish the influence of cavitation on the maximum ductility.

The optical micrographs (Fig. 2a to d) and the scanning electron micrographs (Fig. 3a to d) of the fracture surfaces of specimens tested at different temperatures indicate that the final fracture mode changes with temperature. For test temperature $< 500°$C where intergranular cavitation does not occur (Fig. 2a) the final fracture mode is observed to be by ductile rupture (Fig. 3a). Under test conditions where intergranular cavitation does occur i.e. at temperatures $\geqslant 500°$C then the final fracture mode is a result of crack or void growth and interlinkage (a void has $A_r \geqslant 1.0$ and a crack has $A_r \leqslant 1.0$). By this definition the final fracture mode for test temperatures 500 to 600°C is by the interlinkage of voids (Table II). This final fracture mode has been previously suggested [13], as the final fracture mode of the fine grain sized material tested under creep conditions. The similarity between void interlinkage and the void sheet mechanism for ductile rupture was pointed out by Fleck *et al.* [13] and this has been expanded in a parallel publication [14] where the McClintock [15] model for ductile rupture has been modified to explain final fracture by the interlinkage of voids and this fracture mode has been called the intergranular void sheet (IVS) mechanism.

The important parameters which control the onset of fracture by this mechanism are: the strain-rate sensitivity of the material, and the void size, spacing and geometry and the following inequality for the onset of fracture has been developed [14] from the McClintock [15] inequality for ductile rupture:

$$\frac{1}{\sigma} k\epsilon^n\dot{\epsilon}^m \left[\frac{n}{\epsilon} + \frac{m}{\dot{\epsilon}}\left(\frac{\mathrm{d}\dot{\epsilon}}{\mathrm{d}\epsilon}\right)\right] < \sqrt{\frac{3}{8}}\left(\frac{1+Z}{1-Z}+1\right)^{\frac{1}{2}} F_{Zb^2}\left(\frac{2b^{\mathrm{i}}}{lb^{\mathrm{i}}}\right)^2 \qquad (2)$$

where σ is the applied stress, ϵ and $\dot{\epsilon}$ are the strain and strain-rate respectively, n is the strain-hardening coefficient, m is the strain-rate sensitivity, Z is the void eccentricity, F_{Zb^2} is the void growth factor, $2b^{\mathrm{i}}$ is the initial horizontal width of the void, lb^{i} is the initial horizontal spacing between voids. This fracture criterion can be applied to fracture in the temperature range 500 to 600°C. The scanning electron micrographs Fig. 3b and c show areas of ductile rupture between adjacent "holes". The areas of ductile rupture are considered to be a result of the fracture of internal necks and the "holes" are the cross-section of voids. These observations are evidence that the final fracture mode is by the IVS mechanism for test temperatures of 500 and 550°C.

Fig. 3d shows that the fracture surface of a specimen tested at 800°C is completely intergranular. Such a fracture is a result of cavity growth and interlinkage to form grain boundary cracks which then propagate and interlink along the grain boundaries. At 800°C grain growth is observed to have taken place (Fig. 2d). This effect will markedly influence the cavitation process as cavities will not be able to nucleate until the grain size has stabilized as grain-boundary migration will relax any stress concentrations at grain-boundary irregularities. The observation that there are no cavities in the bulk of the grains is evidence that cavity nucleation did not occur until the grain size stabilized. Fleck *et al.* [13] have previously indicated that the coarser the grain size the easier it is for grain-boundary cracks rather than voids to be formed and this is considered to be the case for the specimen tested at 800°C.

The change in fracture mode with test temperature is a result of the influence of test temperature on the grain stability of the material. For test conditions where the grain size of the material remains stable (500 to 600°C) the cavities are opened up mainly parallel to the stress axis as a result of the high strains and the pinning action of the triple junctions on the growth of the cavities along the grain boundaries [13]. Final fracture then occurs by the IVS mechanism. For the test conditions where the grain size is unstable (800°C) cracks are formed along the grain boundaries in a direction mainly perpendicular to the stress axis. Under these conditions fracture occurs by crack propagation and interlinkage along the grain boundaries. From the results presented in Table II it is indicated that measurements of A_r can be used to determine the final fracture mode. For $A_r \geqslant 1$ fracture will occur by the IVS mechanism whereas for $A_r \leqslant 1$ fracture will occur by the propagation and interlinkage of cracks along grain boundaries.

5. Conclusions

1. During superplastic deformation intergranular cavities can be nucleated at the intersection of a twin boundary with a grain boundary.

2. The maximum elongation occurs at a different temperature than the maximum strain-rate sensitivity.

3. The cavitation process and the value of the strain-rate sensitivity must be optimized in order to obtain the maximum elongation.

4. The final fracture mode in superplastic material which exhibits intergranular cavitation is by the intergranular void sheet (IVS) mechanism.

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