JOURNAL OF MATERIALS SCIENCE 22 (1987) 3017–3022

A criterion for cavitation of two-phase superplastic alloys

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Microduplex 60/40 brass and Zn–Al eutectoid were used as models to represent cavitating and non-cavitating classes of the two superplastic alloys, respectively. In addition, a series of alloys were produced to represent the individual phases, over a wide range of temperatures, in these model systems. Hardness, Young's modulus and tensile tests were carried out on all these alloys at temperatures between 20 and 700° C. Analysing the above data, a criterion for the occurrence of cavitation in two-phase superplastic alloys is proposed.

1. Introduction
Soon after the publication of Underwood's [1] review of superplasticity – a phenomenon whereby certain crystalline materials can be deformed to produce unusually high ductility at above $0.5T_m$ – much interest developed in this field. Since then a large number of alloys have been rendered superplastic (see reviews [2–4]) but only a few are commercially available. The reason is that many superplastic alloys based on engineering alloys (e.g. brasses and steels) often fail at relatively low elongations ($\approx 400\%$) due to grain boundary cavitation. The phenomenon of cavitation has been largely neglected (except for [5] in which it was discussed in some detail) and the various ideas put forward actually dealt with cavitation in creep. In this approach, in order to understand cavitation in two-phase superplastic alloys, two alloys were used; firstly Zn–Al eutectoid which normally does not cavitate and secondly, a microduplex 60/40 brass which does, as models of each class of behaviour. In this paper, studying the properties of the individual phases in each model system by various experimental techniques, a criterion for the occurrence of cavitation in two-phase superplastic alloys is proposed.

2. Experimental procedure
2.1. Hardness and Young's modulus tests
These have been described in detail in a previous paper [6]

2.2. Tensile tests
Round tensile specimens of 4.5 mm diameter and 25 mm gauge length with 9.4 mm BSF thread on the ends were machined from the extruded rods of each alloy. All specimens were annealed at appropriate temperatures to bring them into the annealed as opposed to extruded state. The tests were carried out on a standard Instron 4536 kg tensile testing machine at various temperatures from 100–700° C at a fixed crosshead speed of 2.5 mm min^{-1}. An air-circulating furnace with a hot zone of 17.5 cm was used for tests on Al–Zn alloys between 100 and 270° C. The specimen temperature was measured by a mercury thermometer inserted through a hole in the centre of the furnace front and was constant to within ± 1.5° C. For tests on Cu–Zn alloys at higher temperatures between 300 to 700° C a three zone furnace with a uniform hot zone 15 cm long was used. The temperature was accurate to within ± 2.5° C and measured by using a chromel/alumel thermocouple attached to the specimen. Each specimen was soaked for about 30 min at the test temperature before pulling it to failure.

2.3. Materials
In addition to the two model alloys (Zn–Al eutectoid and microduplex 60/40 brass) a series of alloys were produced of compositions corresponding to those of the individual phases in these model systems over a wide range of temperatures in the superplastic range. These special alloys were used to represent the alpha and beta phases in each superplastic alloy (see Table I). All materials were in the form of extruded bars of 15.9 mm and 25 mm diameter for tensile and hardness specimens, respectively.

3. Results
3.1. Al–Zn system
Initially, it was hoped to study the flow characteristics and activation energies of each phase, *in situ*, in the superplastic alloys using hot microhardness technique. But the *in situ* results for each phase in the Zn–Al eutectoid ($A_{\alpha\beta}$) could not be obtained because of its limited propensity for grain growth. Therefore, the results presented here are for those on special alloys made to represent the alpha and beta phases in $A_{\alpha\beta}$, over a range of temperatures. Fig. 1 shows that the hardness of the superplastic $A_{\alpha\beta}$ is lower than that of its individual phases alpha and beta over a range of temperatures (150–270° C). At the superplastic temperature (250° C), hardness (Hv) $\alpha:\beta:A_{\alpha\beta}$ is 18:15:2 Hv respectively. Fig. 2 plots the ratio of the

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0022–2461/87 $03.00 + .12

TABLE I Composition of alloys

Alloy	Denoted by	Actual composition (wt %)	Phase represented in S.P. alloy	Temperature range (°C)
Al–78 Zn	$A_{\alpha\beta}$	Al–77.4 Zn–0.09 Fe		
Al–12.4 Zn	$A_{\alpha 1}$	Al–13.0 Zn	α	160–220
Al–22 Zn	$A_{\alpha 2}$	Al–21.8 Zn	α	230–270
Al–99.5 Zn	A_{β}	Al–98.5 Zn	β	20–270
60Cu–Zn	$B_{\alpha\beta}$	59.6 Cu–Zn		
62Cu–Zn	B_{α}	61.4 Cu–Zn	α	300–700
55Cu–Zn	B_{β}	54.6 Cu–Zn	β	300–700

hardness of the two phases against temperature between room temperature and 270° C, and at 250° C, H(α)/H(β) = 1.2.

At the temperature of superplastic deformation, U.T.S. (β)/U.T.S. (α) = 1.24, and elongations of 30% and 33% were obtained for the alpha and beta phases respectively (Fig. 3). Thus, the two phases of Zn–Al eutectoid have similar mechanical properties at the superplastic temperature. The activation energies for deformation of α-phase, β-phase and the eutectoid $A_{\alpha\beta}$ at 250° C are 29.9 ± 2 kcal mol^{-1}, 19.5 ± 2 kcal mol^{-1} and 11.3 ± 2 kcal mol^{-1} respectively (Table II).

3.2. Cu–Zn system

In order to retain the highest possible proportion of beta phase in large crystals the microduplex 60/40 brass ($B_{\alpha\beta}$) was annealed at 700° C for 118 h and then quenched in water. This was necessary because, in a preliminary experiment, the beta phase was found to be very soft and the indentation tended to distort the adjacent grain boundaries. Indentations were made in each phase, *in situ*, at different temperatures and the results so obtained are shown in Fig. 4a. The hardness of the separate alpha and beta phases (represented by special alloys) together with that of the superplastic $B_{\alpha\beta}$ is compared in Fig. 4b. Above 250° C (below 250° C), $B_{\alpha\beta}$ is harder (softer) and softer (harder) than its beta and alpha phases, respectively. Beyond 600° C the hardness of $B_{\alpha\beta}$ approaches that of its beta phase. The shape of the $H(\alpha)/H(\beta)$ against T plot in Fig. 4c for separate phases is similar to that of the *in situ* results. At the superplastic temperature (600° C), the (*in situ*) $H(\alpha)/H(\beta)$ = 6.3. Table III summarizes the mechanical properties of the two phases of $B_{\alpha\beta}$ at 600° C. At this temperature, the beta phase is highly ductile (≈ 172% elongation), whereas an elongation of only ≈ 54% could be obtained in the alpha phase; the ratio U.T.S. (α)/U.T.S. (β) = 7.2. Thus the two phases of the superplastic 60/40 brass are clearly dissimilar in their mechanical properties at the superplastic temperature. The activation energy values near superplastic temperature are 42.2 ± 2 kcal mol^{-1}, 22.0 ± 2 kcal mol^{-1} and 21.2 ± 2 kcal mol^{-1} for the alpha phase, beta phase and superplastic $B_{\alpha\beta}$ respectively (Table 2).

TABLE II Activation energy values for the alloys [6, 15]

Alloy	Activation energy Q (kcal mol^{-1})	Temperature range (T_m)*
$A_{\alpha\beta}$	11.3 ± 2	0.5–0.7
$A_{\alpha 1}$	29.5 ± 2	0.6–0.8
$A_{\alpha 2}$	29.9 ± 2	0.5–0.8
A_{β}	19.5 ± 2	0.7–0.9
$B_{\alpha\beta}$	21.2 ± 2	0.67–0.8
B_{α}	42.2 ± 2	0.6–0.8
B_{β}	43.0 ± 2	0.5–0.7
	22.0 ± 2	0.7–0.8

*T_m = melting point (k).

4. Discussion

The H-value of the individual phases of a superplastic alloy is important; firstly because it gives a measure of the ease or difficulty of plastic deformation by dislocation glide in the individual phases and secondly, the variation of hardness with temperature provides information about the rate of diffusion in each phase. This two-fold information from the hardness measurements is important in explaining cavitation during superplasticity.

4.1. Class 1: Non-cavitating superplastic alloys

Zn–Al eutectoid is the model alloy that represents this class of superplastic alloys. Fig. 5 confirms that little cavitation occurs during superplastic deformation of this alloy. We found little grain growth occurring in this alloy after superplastic deformation.

Grain boundary sliding (GBS) plays a major role in the superplastic deformation of Zn–Al eutectoid. It causes a high concentration of stress at any irregularity in the grain boundaries and its accommodation will occur more readily if all phases present can contribute to the accommodation process whether this is by diffusion or dislocation glide. The mechanical and diffusion properties of the alpha and beta phases of Zn–Al eutectoid imply that accommodation of GBS may be occurring in the former by dislocation glide predominantly, and that in the latter the diffusion of atoms may be more important. The beta phase with

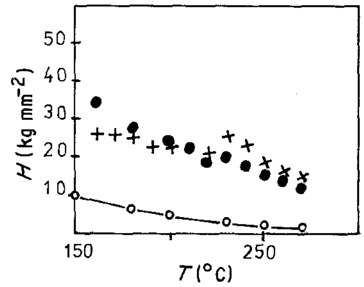

Figure 1 Hardness (H) as a function of temperature relationships for the Zn–Al eutectoid and its individual phases. Break in curve (at about 225° C) for the alpha phase is because of two alloys used to represent it over the temperature range 160 to 270° C. (O) Zn–Al eutectoid, (x) α-phase, (●) β-phase

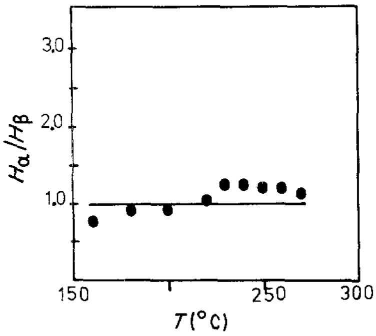

Figure 2 Ratio of the hardness of two phases of the Zn–Al eutectoid as a function of temperature.

TABLE III Mechanical Properties of the two phases of 60/40 brass at 600° C

	α-phase	β-phase
% Elongation	54	172
Hardness (Hv)		
(a) *in situ*	17.8	2.8
(b) separate phases	12.2	1.6
*UTS ($\times 10^3$ p.s.i.)	3.6	0.5

*UTS = ultimate tensile strength.

$Q = 19.5 \pm 2\,\text{kcal mol}^{-1}$ appear to be more 'active' than alpha phase with $Q = 29.9 \pm 2\,\text{kcal mol}^{-1}$. Higher diffusivity of the beta phase has been indicated in the work of Naziri and Pearce [7]. Naziri *et al.* [8], in their *in situ* experiments on Zn–Al eutectoid, observed no dislocation activity in the beta grains while some dislocation activity was observed in the alpha grains. The Q-value ($13.2 \pm 2\,\text{kcal mol}^{-1}$) for the eutectoid is close to the value for grain boundary diffusion in zinc, indicating the important role played by the beta phase during superplastic deformation, the proportion alpha : beta being 28 : 72 at 250° C.

At a lower temperature of 190° C, the two phases of the $A_{\alpha\beta}$ are less ductile and have higher flow stress than at 250° C. At this temperature, although the hardness of the alpha and beta phases is similar, the ductility and U.T.S. values are not. However, the relative difference in the properties of the two phases become less significant as the grain size is decreased since the strains developed in the individual grains as a result of greater GBS become small. As a consequence less accommodation over the grain or phase boundary area is needed. Moreover, the contribution from grain boundary diffusion to accommodation processes is increased because a larger grain boundary area is now available. This is probably the case in the deformation of the very fine and stable structured Zn–Al eutectoid which shows superplasticity without any significant amount of cavitation. Walser *et al.* [9] developed a thermal mechanical process to obtain a very fine (< 1 μm) mixture of cementite in ferrite. Although the cementite particles are much harder than the ferrite matrix (at 700° C, H(Cementite)/H(Ferrite) = 97 Hv/22 Hv = 4.4, [10]), such a steel (1.6% C) can be deformed up to 750% elongations at 650° C and cavitation does not occur during its superplastic deformation.

There were some inclusions present in the as-received alloy $A_{\alpha\beta}$ (Fig. 6) which did not change shape nor cracked after deformation. A few cavities were found to be associated with such inclusions (Fig. 7). An analysis in the SEM showed these impurities to be rich in iron. Woodthorpe and Pearce [11], while studying the fatigue behaviour of this alloy, also encountered these regularly shaped inclusions and identified them as $FeAl_3$ type. The size of these inclusions was too small to allow measurement of their hardness variation with temperature using the present machine. However, Petty [12] has measured the hot hardness of $FeAl_3$ and gives 750 Hv at 20° C falling to 650 Hv at 250° C, and inferred that slip processes were difficult in it. Thus, the presence of very hard $FeAl_3$ type inclusions introduces a small amount of cavitation in this

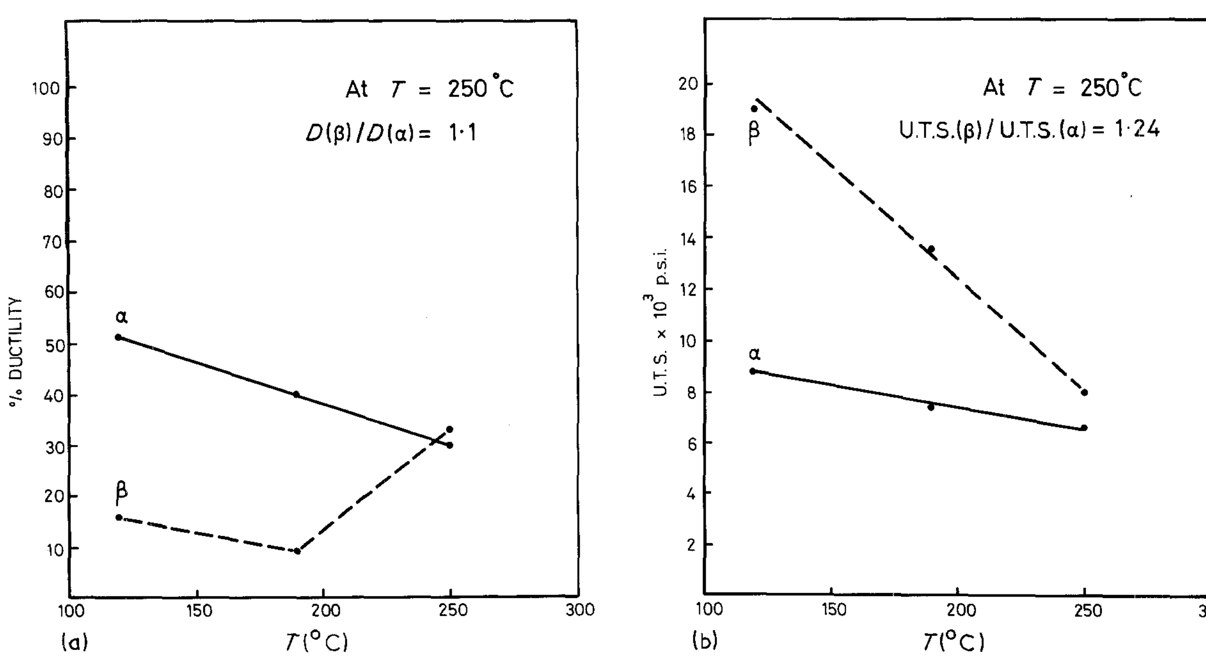

Figure 3 (a) Variation of ductility with temperature for the two phases of Zn–Al eutectoid. (b) Variation of U.T.S. with temperature for the two phases of Zn–Al eutectoid.

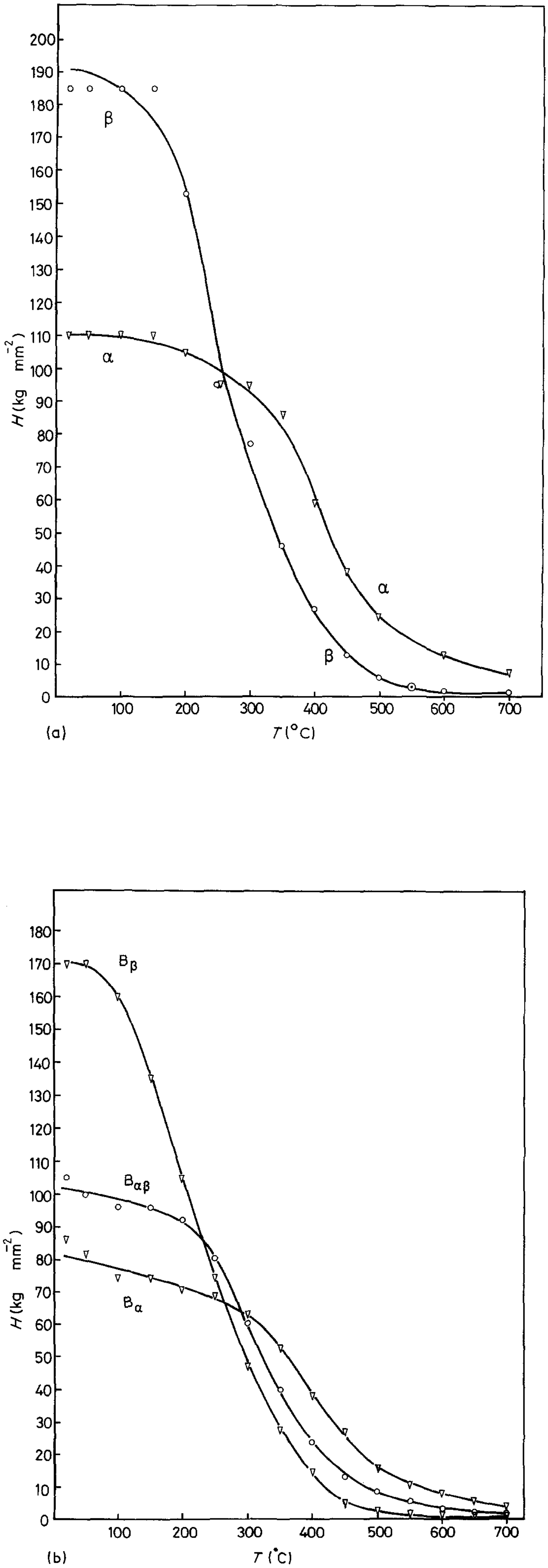

Figure 4 (a) Hardness as a function of temperature curves for the *in situ* alpha and beta phases of the 60–40 brass. 118 h at 700° C, quench water, load = 10 g, time of loading = 15 sec, atmosphere-vacuum. (b) Hardness as a function of temperature curves for the 60–40 brass ($B_{\alpha\beta}$) and its separate phases (represented by B_α and B_β alloys). All three alloys [illegible] h 600–620° C anneal. (c) Ratio of the hardness of two phases of the 60–40 brass as a function of temperature. (A) *in situ*, (b) separate phases represented by special alloys.

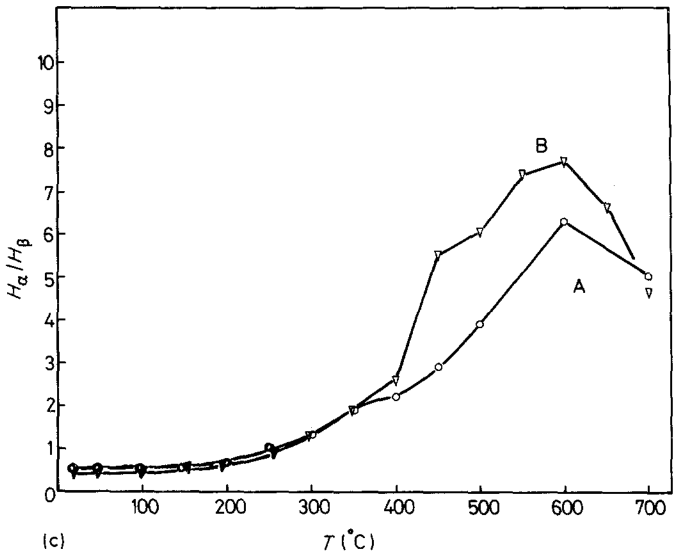

Figure 4 Continued.

otherwise normally non-cavitating superplastic Zn–Al eutectoid because of incomplete accommodation of stress concentrations at the inclusion/matrix interfaces during deformation.

4.2. Class II: Cavitating superplastic alloys

This class of superplastic alloys is represented by the microduplex 60/40 brass ($B_{\alpha\beta}$). Fig. 8 shows cavitation in this alloy after superplastic deformation at 600° C. The amount of cavitation decreased with increasing temperature at a fixed strain rate. The cavity sites were mainly in alpha/beta boundaries, although some cavities were also situated at alpha/alpha, beta/beta and within the beta grains. The activation energy value for $B_{\alpha\beta}$ near its superplastic temperature (600° C) is 21.2 $\pm$ 2 Kcal mol^{-1}. Since GBS is the predominant mode of superplastic deformation in this alloy [13] and sliding occurs mainly at alpha/beta interfaces, this value of Q could be referred to as the "activation energy for interphase diffusion" and is similar to the activation energy for deformation of the beta phase, the latter being 22 $\pm$ 2 kcal mol^{-1}. At 600° C, the beta phase is in a disordered state and interdiffusion of copper and zinc atoms takes place within it [14]. Thus so far as the diffusion accommodation of interphase boundary sliding is concerned, a major contribution will come from the beta phase side of the alpha/beta interface, whereas the alpha phase with an activation energy of 42.2 $\pm$ 2 kcal mol^{-1} will remain relatively inactive. A lower value of Q for the beta phase could also imply that a thermally-activated motion of dislocations will occur more easily in beta than in the alpha phase which requires a higher value of activation energy. The mechanical properties of the two phases indicate that the plastic deformation of the beta phase occurs much more easily than alpha phase at the superplastic temperature, 600° C. Thus, even the dislocation–accommodation of sliding at the alpha/beta interfaces would be easier on the beta phase side compared with alpha phase side of the interfaces in $B_{\alpha\beta}$ alloy.

Clearly, the phases of the cavitating microduplex 60/40 brass at the superplastic temperature are "incompatible" (i.e. they have different activation energies and mechanical properties). Therefore, the alloy cavitates because of unequal contributions made by the two phases to the accommodation of interphase sliding during superplastic deformation. Moreover, grain growth occurs during superplastic deformation which leads to higher stresses arising from sliding at the interfaces and makes the accommodation processes even more difficult. Therefore, we propose the following criterion for the occurrence of cavitation in two-phase superplastic alloys.

It seems that cavitation will not occur during

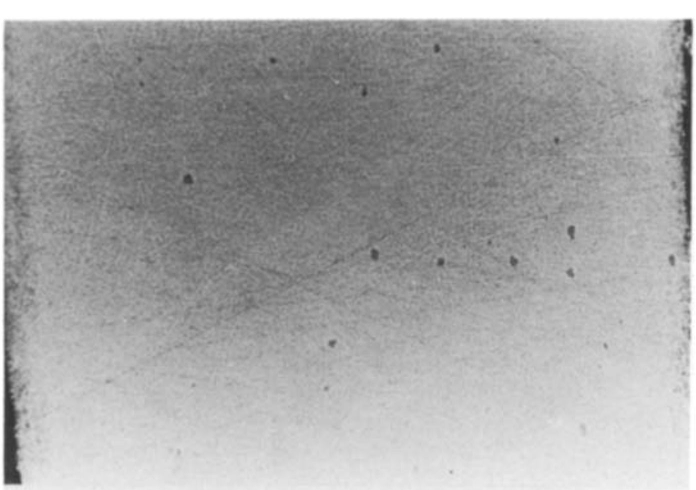

Figure 5 Optical micrograph of Zn–Al eutectoid deformed superplastically to 400 pct. elongation at 250° C (0.7 Tm), strain rate = 1.6 $\times$ 10^{-3} sec^{-1}. There is little cavitation. The $FeAl_3$ type inclusions are shown. Tensile axis is vertical. ($\times$ 140)

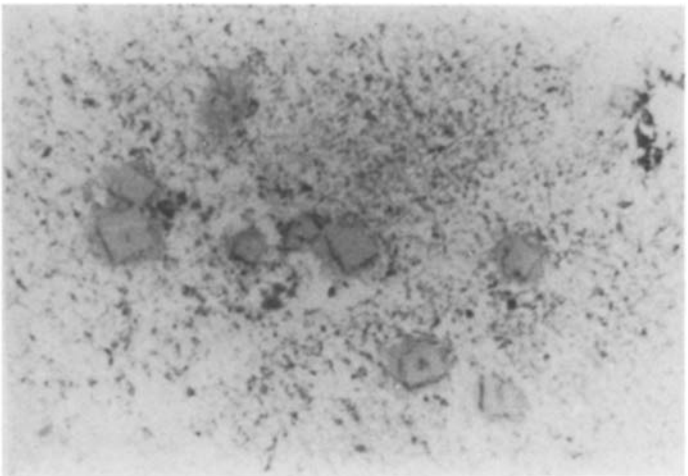

Figure 6 Optical micrograph of Zn–Al eutectoid in the as-received condition, showing the presence of $FeAl_3$ type inclusions. (× 1400)

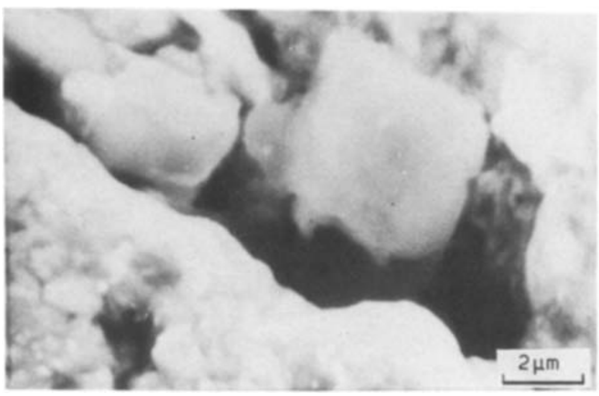

Figure 7 Scanning electron micrograph of fracture surface of specimen of Zn–Al eutectoid deformed at 250° C (0.7 Tm) showing decohesion of $FeAl_3$ type particles to form cavities.

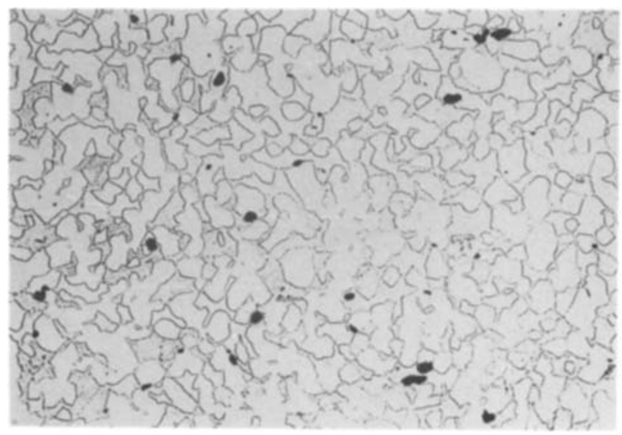

Figure 8 Optical micrograph of duplex 60–40 brass after 170% deformation at 600° C (0.74 T_m), strain rate = $1.6 \times 10^{-3}\,sec^{-1}$ showing cavitation mainly at the alpha–beta boundaries. Tensile axis horizontal. Grain size ≈ 40 μm, initial grain size was ≈ 7 μm. (× 84.4)

superplastic deformation of a superplastic alloy consisting of two phases which are ultrafine (< 1 μm) and stable, whether these are "compatible" with each other or not. A superplastic alloy (1 μm < grain size < 10 μm) will show cavitation during deformation whenever its two phases are "incompatible", that is, they have different mechanical and diffusion properties, at the superplastic temperature. On the other hand, because of the similarities in properties of the two phases (i.e. "compatible" phases) of a superplastic alloy (grain size < 10 μm), they will make equal contributions to accommodate the strains arising from GBS, as a result cavitation will not occur. This assumes that rapid growth does not occur at the superplastic temperature.

Acknowledgements
This work was carried out at the Cranfield Institute of Technology during the period 1976–80. We are very grateful to the SERC for their financial support of this project.

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Received 16 October 1986
and accepted 22 January 1987