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Fracture of a tin–lead eutectic alloy in Region III of superplastic flow

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With the aid of scanning electron microscopy, cavitation and fracture behaviour in the Sn–Pb eutectic alloy, whose reduction in area of cross-section before failure is close to 100%, has been investigated in Region III of superplastic flow (where both the elongation-to-fracture and the strain-rate sensitivity index decrease with increasing strain rate). It has been demonstrated that, although it decreases, grain-boundary sliding persists in this range as the strain rate is increased. At all strain rates the final failure was due to tearing by plastic flow of the inter-cavity ligaments, but the interlinkage of cavities along the grain–interphase boundaries decreased with increasing strain rate. The features of cavitation and fracture did not differ much from an earlier study on a pseudo-single phase copper alloy, although copper alloys usually fail non-ideally, i.e., a large area of cross-section is present at fracture.

1. Introduction

A number of studies have considered the flow behaviour of superplastic alloys. In comparison, much less attention has been paid to fracture in this class of materials [1, 2]. There are two types of fracture in these alloys:

(a) "ideal" fracture in which the reduction in area is close to 100%, such as is seen in the Sn–Pb, Zn–Al and Pb–Cd systems; and

(b) "quasi-brittle" or "non-ideal" fracture in which a large area of cross-section is present at failure, such as is encountered in α–β brass and steels of different grades. In the second type of fracture, cavitation is seen at a very early stage of deformation and, in general, it increases with strain.

Copper alloys usually exhibit fracture of the second type [1, 2]. Nevertheless, cavitation behaviour in a pseudo-single phase copper alloy with a dispersed cobalt phase [3, 4] was not all that different from the behaviour of the Zn–Al eutectoid alloy [5–8], which displays failure of the first type. Subtle differences are, however, present.

According to Miller and Langdon [9], when vacancy diffusion controls the growth of cavities they are approximately spherical and randomly distributed. When power-law creep (mechanism is not identified) is dominant, the vacities are elongated and lie in strings parallel to the stress direction.

In this view, in Zn–22 wt% Al alloy in both Regions I and II of superplastic flow, power-law growth is dominant. However, in the pseudo-single phase copper alloy [3, 4] in Region I vacancy diffusion is predominant, while in Region II power-law creep controls the growth of cavities.

For Region III, the studies have been mostly confined to an examination of the surfaces of fractured samples. According to Shei and Langdon [3] in the pseudo-single phase copper alloy, small cavities are present that lie along strings parallel to the tensile axis. Cavity inter-linkage, however, was absent. On the other hand, Ahmed *et al.* [8] could see no evidence for surface cavitation in the Zn–Al eutectoid alloy.

The aim of this paper is to examine if this

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reported difference in behaviour in Region III, that depends on the type of fracture, is genuine by examining the surfaces of fractured specimens using scanning electron microscopy in place of optical metallography employed in the earlier investigations [3, 8]. Moreover, the effect of strain rate on the extent of grain-boundary sliding has also been assessed qualitatively by viewing the fracture surface. For this purpose, a nominally Sn–38 wt% Pb alloy was tested to failure (in Region III) at two different temperatures. The characteristics of flow under these experimental conditions have already been reported [10].

In view of the limited aim stated above, the present study was confined to an examination of the specimen surfaces only. Because of differences in constraints, the behaviour in the interior of specimens could be different. No attention was paid to this aspect because two earlier studies [11, 12] have considered this point. It should also be noted that neither the present paper nor the earlier work of Ahmed *et al.* [8] is concerned with identifying the stage of deformation at which cavitation commences in those alloys that exhibit ideal fracture. This last point has been the subject of comment by Hazzledine and Newbury [11] and Geckinli and Barrett [12].

2. Experimental procedure

The procedure for obtaining the strip tensile specimens of dimensions 31.75 mm × 6.34 mm × 1.50 mm, needed for the present tests, has already been described [10]. As before, the alloy had a composition (± 0.3 wt%) of 37.3 wt% Pb, balance Sn, with traces of Fe, Cu, Zn and Ti also being detected. Following annealing at 373 K for 900 sec and quenching in ice-water, a two-dimensional grain size of 4.7 ± 0.2 μm was obtained in the specimens.

Room-temperature tensile tests were carried out till fracture on an Instron Universal Testing Machine. Four constant cross-head speeds of 8.33, 16.66, 83.33 and 166.66 mm $ksec^{-1}$ (0.05, 0.10, 0.50 and 1.00 cm min^{-1}) were employed. Some tests were also performed at 348 K at a cross-head speed of 8.33 mm $ksec^{-1}$ (0.05 cm min^{-1}). In all cases the results were verified to be reproducible.

The fractured specimens were subjected to scanning electron microscopy. In the case of specimens tested at elevated temperature, the fracture surfaces were seen to be contaminated by the furnace packing material, i.e., asbestos fibre.

3. Results

The results of the room-temperature tests are first considered. The strain-rate range pertained to Region III of superplastic flow in which both the strain-rate sensitivity m, and the elongation to fracture decreased with increasing initial strain-rate (see Fig. 1).

At a cross-head speed of 8.33 mm $ksec^{-1}$ ($m = 0.40$) in a large area of the fracture surface

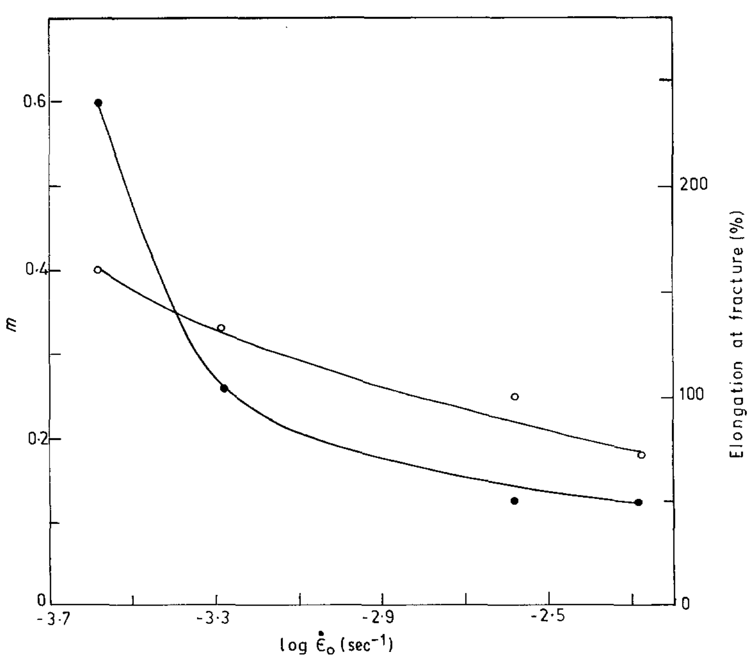

Figure 1 ○, strain-rate sensitivity, index, m, and ●, percentage elongation to fracture as a function of the initial rate of deformation. Room temperature tests.

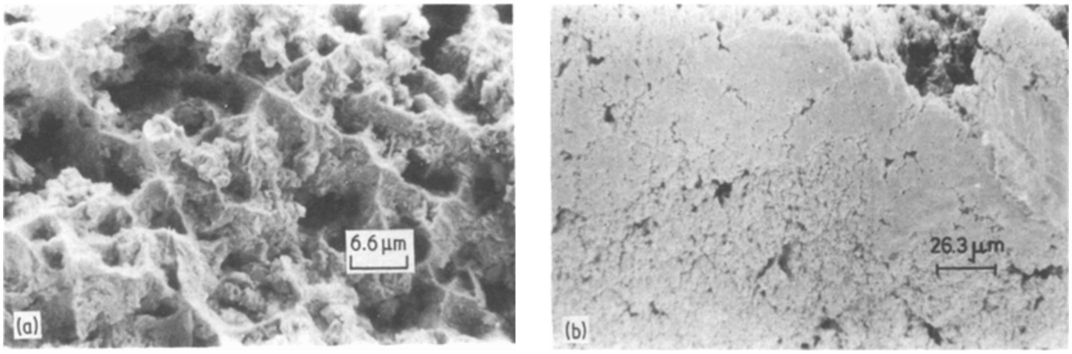

Figure 2 A specimen tested to fracture at room temperature at a cross-head speed of 8.33 mm ksec^{-1}. (a) Fracture surface and (b) side wall (gauge-length portion) of the specimen.

individual grains and triple-points were clearly identifiable. The formation of sharp tear ridges was also seen (see Fig. 2a). Final fracture appeared to be due to tearing. Examination of the wall of the fractured specimen, up to about 4 mm away from the fracture surface, revealed evidence for (surface) cavity nucleation and inter-linkage (see Fig. 2b).

At a cross-head speed of 16.66 mm ksec^{-1} ($m = 0.33$) the area occupied by the tear ridges had increased. Holes left behind by grains (or clumps of grains) pulling apart along grain boundaries could also be seen (see Fig. 3a). As a result of the latter process, some grains in the interior of the tensile specimen could be seen. Grain-boundary decohesion was also visible at these boundaries. The walls of the fractured specimen were highly rumpled but cavity formation was less pronounced (see Fig. 3b).

At a cross-head speed of 83.33 mm ksec^{-1} ($m = 0.25$) holes left by individual grains or clumps of grains occupied even less area and tearing was predominant (see Fig. 4a). The wall of the fractured specimen, however, was still "granular" and uneven but evidence for cavity formation (surface delamination) was slight (see Fig. 4b).

At the highest cross-head speed of 166.66 mm ksec^{-1} ($m = 0.18$) the area occupied by inter-granular separation of grains (or clumps of grains) was the least and tearing was dominant (see Fig. 5a). The walls of the fractured specimen were relatively smooth and a few gaps (cavities) could be seen only very close to the fracture surface (see Fig. 5b).

Following deformation at a test temperature of 348 K and a cross-head speed of 8.33 mm ksec^{-1} ($m = 0.40$) the fracture surface was highly contaminated by the furnace packing material, transferred to the specimen by the water medium in which the high temperature tests were conducted [10]. Therefore, it was not possible to identify clearly the details of cavitation and fracture in the high-temperature tests. However, it does appear that here also the grains pulled apart along their grain boundaries (see Fig. 6). By examining the

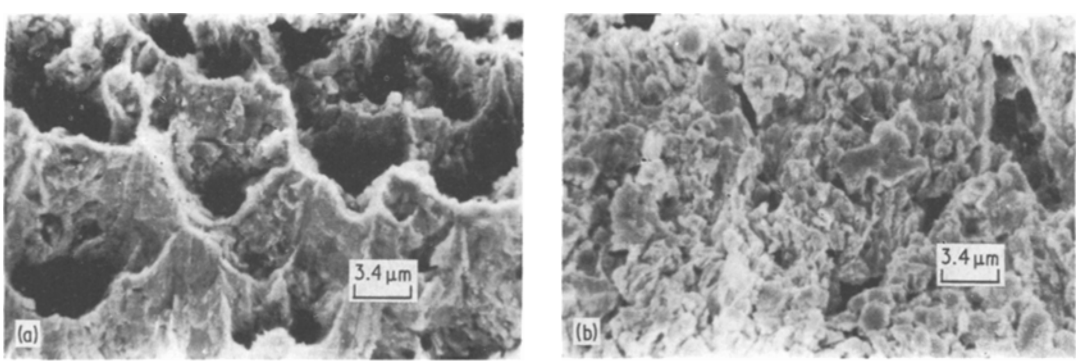

Figure 3 A specimen tested to fracture at room temperature at a cross-head speed of 16.66 mm ksec^{-1}. (a) Fracture surface and (b) side wall of the specimen.

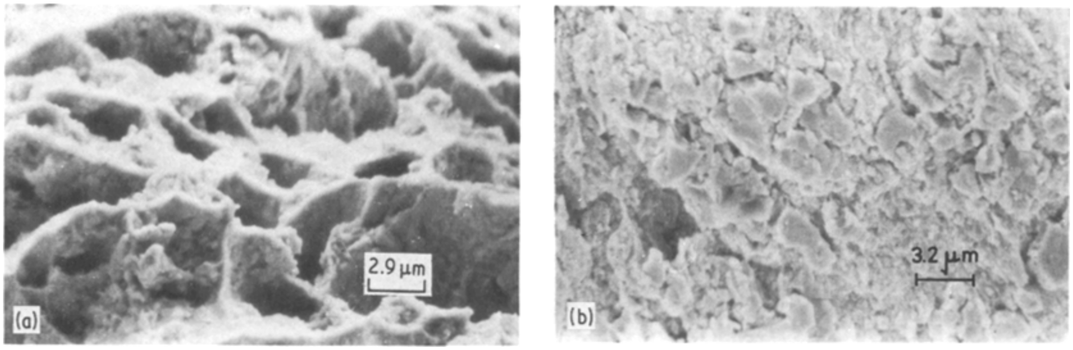

Figure 4 A specimen tested to fracture at room temperature at a cross-head speed of 83.33 mm ksec^{-1}. (a) Fracture surface and (b) side wall of the specimen.

"grain" coming out of the fracture surface (shown in Fig. 6) at a higher magnification it was established that its size and shape closely resembled that of the initial grain.

4. Discussion

It has already been pointed out [10] that the values of m obtained in the present work are in good agreement with those reported in the earlier studies. On the other hand, the elongations encountered in these experiments are appreciably lower than those reported by other workers. This observation appears to be directly attributable to the much lower thickness to gauge-length ratio employed in the present case and this point has been discussed in the earlier paper [10]. The present results dealing with cavity formation in the gauge portion and fracture surface of specimens tested to fracture in Region III of superplastic deformation are analogous to those reported by Ahmed *et al.* [8].

Using optical microscopy, Langdon and co-workers [5, 7, 8] obtained evidence for surface cavitation in the gauge-length portion of Zn–Al eutectoid alloy specimens tested to fracture in Regions I and II of superplastic flow. Their claim [7] that Ishikawa *et al.* [5] were the first to demonstrate that cavities may form in materials that pull-down to a point does not take into account two earlier and more elegant investigations [11, 12] involving scanning electron microscopy. Both Hazzledine and Newbury [11] and Geckinli and Barrett [12] have paid greater attention to surface cavitation than Langdon and co-workers [5, 7, 8] and they also noted [11, 12] that, at an early stage in deformation, small gaps developed between surface grains on account of significant sliding. When the strain rate was in the optimal range, these microcavities were readily filled by sub-surface grains. However, if the strain rate was greater than the optimal rates of deformation, fewer sub-surface grains appeared and the grain-boundary gaps grew to form cracks between surface grains as they moved apart (compare this with the work of Ahmed *et al.* [8], who did not report any surface cavitation in the latter range

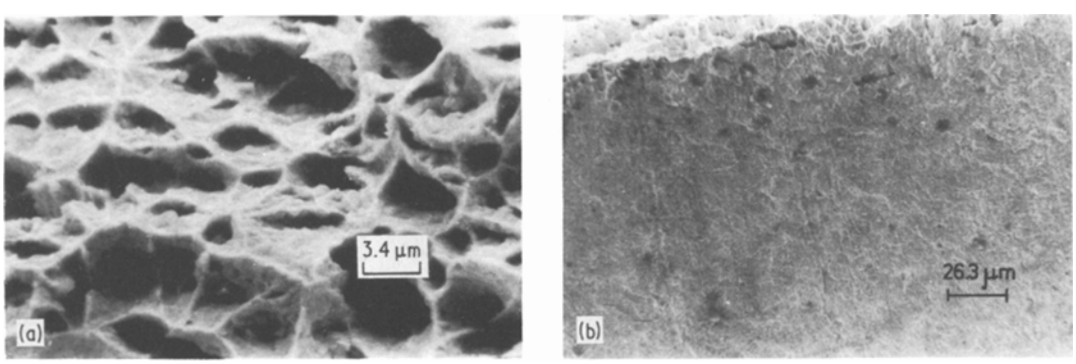

Figure 5 A specimen tested to fracture at room temperature at a cross-head speed of 166.66 mm ksec^{-1}. (a) Fracture surface and (b) side wall close to the fracture surface.

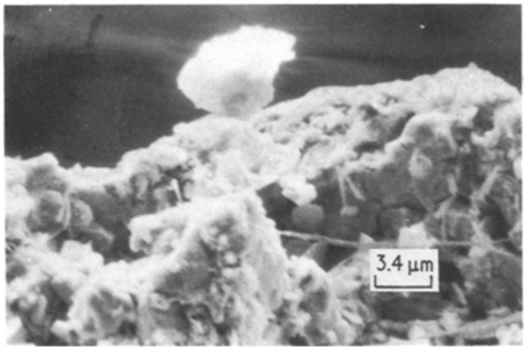

Figure 6 Side view of a fracture surface obtained at 348 K using a cross-head speed of 8.33 mm ksec^{-1}. A single grain is seen pulling out of the fracture surface.

of strain rates). The present work has confirmed this last observation of Hazzledine and Newbury [11] and Geckinli and Barrett [12] and is contradictory to the finding of Ahmed *et al.* [8].

In passing, it is also noted that Hazzledine and Newbury [11] have further stated that during optimal flow in materials that exhibit ideal fracture, the formation of grain-boundary cavities in the interior of specimens is absent. This statement is verified by Humphries and Ridley [13] whose very precise density measurements showed negligible change in the density of Sn–Pb eutectic alloy specimens with increasing strain. In view of the above, the assertion of Gifkins and Langdon [14, 15] that any theory of superplastic flow should include the effects of cavitation from the very early stages is rather strange. The present authors favour the view that cavitation is a prelude to fracture.

Figs 2b, 3b and 4b show that the surface of the samples are considerably roughened by superplastic deformation. This is normally interpreted as evidence for grain-boundary sliding [1]. As stated in the previous section, it could also be verified that grain-boundary sliding is present in the grains of the interior as well. These observations are not surprising because Geckinli and Barrett [12] have already shown that, even when the strain rate and grain size were outside the optimal range for superplastic flow, extensive grain-boundary sliding persisted. However, as the strain rate increased beyond the upper permissible limit for optimal flow, the contribution from sliding decreased with increasing strain rate. This is borne out by Figs 2 to 5. For example, from Figs 2a, 3a, 4a and 5a it follows that with increasing strain rate the area occupied by the tear ridges had increased, i.e., the contribution from grain deformation had increased.

As has been pointed out [16], tearing is a mechanism of local fracture, often found at a discontinuity in the crack advance by another fracture mechanism. In the present case, as the alloy contained no precipitates, cavities are expected to be nucleated at points of stress concentration along the boundaries, e.g., triple-points, interphase boundaries and grain-boundary obstacles arising from local complexities in atomic arrangement, due to significant grain-boundary sliding. As the strain rates pertained to Region III, sufficient time was not available for all the cavities to link-up, only along the grain–interphase boundaries and thereby give rise to purely intergranular fracture. Instead, the intercavity ligaments have fractured by plastic flow. As the importance of grain-boundary sliding (in Region III) decreased with increasing strain rate, the area occupied by the regions that have failed by tearing had increased.

5. Conclusions

Based on the present study, pertaining to Region III of superplastic flow, the following conclusions could be drawn:

(a) Grain-boundary sliding persists in Region III of superplastic deformation, although its importance decreases with increasing strain rate.

(b) At all strain rates, the final failure was due to tearing by plastic flow. However, the proportion of grains that had linked up along their boundaries decreased with increasing strain rate. This inclusion is in agreement with that stated in (a).

(c) As seen by Shei and Langdon [3] in the case of a pseudo-single phase copper alloy, in the Sn–Pb eutectic alloy (which exhibits ideal fracture), cavities could be seen in the gauge-length portion up to a distance of 4 mm from the fracture surface (in specimens tested to fracture). As the present surface observations were similar to those reported by Hazzledine and Newbury [11] and Geckinli and Barrett [12] no need was felt to examine points (on the gauge-length) further removed from the fracture surface. Thus, the failure of Ahmed *et al.* [8] to see cavitation on the surface of Zn–Al eutectoid alloy specimens tested in Region III to fracture is attributed to the incomplete nature of the study and the poor resolution available in optical microscopy.

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