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METALLOGRAPHIC STUDIES OF DUCTILE FRACTURE IN HIGH PURITY ALUMINIUM AND LEAD.

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Abstract

Metallographic studies were made of ductile fracture in superpurity aluminium (99.99%Al), zone-refined aluminium (99.999%Al) and high purity lead (99.9992%Pb) following tensile testing at room temperature and 85°K. By examining longitudinal sections through the necked regions in cylindrical tensile specimens and also by examining the fracture surfaces using stereomicroscope techniques it was shown that large numbers of closely spaced cavities can form in the necked region and on the fracture surfaces of each of these materials during tensile testing at 85°K provided that the grain size was sufficiently fine. At room temperature, cavitation was still observed in superpurity aluminium but was greatly reduced in zone-refined aluminium and could not be detected at all in the high purity lead. The results will be compared with those of other workers and used as a basis for the discussion of possible mechanisms of cavity initiation and hence ductile fracture.

1. Introduction.

The fact that cavities may be formed near inclusions during ductile fracture was convincingly demonstrated by Tipper(1) and Puttick(2) using mild steel, tough-pitch copper and Armco iron. Since then, cavitation associated with ductile fracture has been observed in a large number of different metals and alloys. Rogers(3), for example, observed cavities in the necked regions of heavily deformed OFHC copper and from evidence obtained from electron microfractographs concluded that inclusions, presumably of Cu_2O and Cu_2S, were associated with the cavities. Crussard et al.(4) have stressed the importance of inclusions in the ductile fracture of a wide range of alloys from steels to aluminium alloys. The inference which can be drawn from observations of this kind

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(Cottrell(5)) is that if cavities are nucleated at impurity particles and if fracture is the result of the linking up and growth of the cavities, then in materials which are of such purity that they are free from inclusions, no cavities should form and the material should rupture with 100% reduction in area. If, on the other hand, impurity particles are not essential for the nucleation of cavities, cavitation should still be observed in these inclusion-free materials and fracture should still occur before the reduction in area reached 100%. The establishment of this point by experiment is proving to be rather difficult.

Rosi and Abrahams(6) observed cavitation near the fracture surfaces of single crystals of "high-purity" silver, copper and a copper-aluminium alloy and concluded that because of the high purity of the materials, cavity nucleation on impurities or at gas pockets was not possible. There is, however, some doubt as to whether the purity of their materials (copper approximately 99.98%) was sufficiently high for them to be inclusion-free.

Koppenaal(7) supported Rosi and Abrahams' suggestion in his interpretation of experiments on single crystals of copper 10% aluminium alloys prepared from 99.999% Cu and 99.996% Al. Since cavities could be observed on specimens deformed at room temperature but not on those deformed at 77°K, Koppenaal concluded that the cavities were formed by vacancy condensation. These observations are difficult to reconcile with those of Chin et al.(8) who found that with high-purity aluminium single crystals the material thinned down to 100% necking without cavitation occurring, and also with those of Beevers and Honeycombe(9) who likewise failed to observe cavitation in single crystals of high-purity aluminium. Beevers and Honeycombe's experiments on inclusion-free polycrystalline aluminium and copper-50% nickel alloys(10) reported that these materials did not neck down to 100% reduction in area but that during the later stages of deformation, cracking, and not cavitation, occurred. They therefore rejected mechanisms for the initiation of ductile fracture in high-purity metals which involved either vacancy condensation or plastic yielding around impurity particles and proposed instead that the important factor was the stress concentration around dislocation pile-ups which led to the formation of cracks. Chin's(8) experiments on aluminium of various grades of purity ranging from 99.993% which contained some inclusions to carefully zone-refined aluminium which was claimed to be completely free of inclusions, did not support either Honeycombe's theory or the vacancy condensation theory but instead supported strongly the suggestion that nucleation of cavities occurred on inclusions. The results of the present investigation are in general agreement with the conclusion of Chin et al. in that they underline the importance of inclusions in materials of very high purity. Further, it will be shown that even in zone-refined aluminium, in which the number of detectable inclusions is very low indeed, and in very high-purity lead, cavitation can occur provided the tests are carried out at low temperatures and on fine-grained specimens.

Experimental Procedure.

The materials used in this investigation were superpurity aluminium* (99.99% Al) in which the major impurities were 0.002% Si, 0.002% Fe and 0.003% Cu, zone-refined aluminium* (99.999% Al) in which the major impurities were 0.00004% Si, 0.00005% Fe and 0.00001% Cu. Examination of carefully prepared sections of these materials indicated that they were not completely homogeneous for in each of them "spots" of unknown origin could be observed with the optical microscope. That these spots were related to impurities in the metals was suggested by the observation that there were 170 mm^{-2} on the superpurity aluminium but only about 12 mm^{-2} on the more highly purified zone-refined aluminium. High-purity lead (99.9992% Pb) which contained 0.0003% Cu and 0.0001% of each of Bi, Sb, Fe and Zn and 0.00005% oxygen was also used. This was vacuum melted, scrubbed with hydrogen at 550°C and then cast into ingots in an atmosphere of hydrogen at a pressure of 10^{-2} mm Hg.

Cylindrical tensile specimens which had a gauge length of 1¼ in. and a diameter of 7/32 in. were prepared from each of these materials by swaging from rods of approximately ½ in. diameter to ¼ in. diameter and then machining in a lathe. Specimens of superpurity aluminium were swaged and machined at room temperature before being annealed at 450°C for 1 hour to produce a grain size of 0.07 mm and then electropolished in a perchloric acid - alcohol solution (for some tests the annealing treatment at 450°C was omitted and tensile testing was carried out in the "as swaged" condition). The zone-refined aluminium was annealed at 250°C for 15 mins. to produce a grain size of 0.03 mm and at 250°C for 1 hour to produce a grain size of 0.3 mm.

Tensile testing was performed in hand operated rigid frame straining devices which could be immersed in suitable baths to enable tests to be done over a wide range of temperatures. The machines were very hard so that close control on strain could be exerted even when necking was well advanced. This meant that straining could be stopped just before fracture and the severely necked specimen mounted in epoxy resin to enable longitudinal sections through the necked region to be prepared for metallographic examination. With other specimens straining was continued to fracture so that the fracture surfaces could be examined with a stereomicroscope.

Results.

(a) Superpurity Al 99.99%. The presence of cavities in the necked regions of specimens of superpurity Al is well illustrated by Fig. 1 which

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* Kindly supplied by the Research Laboratories, British Aluminium Co. Ltd., Chalfont Park, Gerrards Cross, Bucks., England.

is a metallographic section through a well developed neck in superpurity Al deformed at room temperature. Cavities of this kind have been observed in this material over a wide range of conditions of grain size and testing temperature but two features appear to be significant. First, as the test temperature was decreased from 373°K to 85°K, the extent of cavitation increased markedly. This is shown in Figs. 2 and 3 which are photomicrographs of longitudinal sections through the fracture on specimens deformed at 373°K and 85°K respectively. The difference in the number of cavities in the fracture surfaces can be seen by comparing the stereofractograph Fig. 4 from the specimen deformed at 373°K with Fig. 5 from a specimen deformed at 85°K. Figs. 4 and 5 were obtained using material which had been annealed prior to tensile testing to produce a grain size of 0.07 mm (approximately 30 grains per cross section) but very similar results were obtained with "as swaged" material which was not annealed before testing.

The second feature was that cavitation was observed only when the grain size of the specimens was small compared to the diameter of the specimen. When the grain size was such that there were on the average only 5 or 6 grains in the cross section, the mode of fracture changed to the knife-edge type; under these conditions no cavities could be detected on the fracture surface and only rarely could they be seen on metallographic examination of transverse sections through the necked region.

(b) Zone-refined Aluminium 99.999% Al. Similar experiments to those reported above were carried out using zone-refined aluminium. Similar trends were observed but cavitation was less extensive than with the superpurity material strained under equivalent conditions. Coarse grained specimens deformed at room temperature failed by a shearing off mode which does not encourage cavitation and only rarely were cavities seen. Very fine grained specimens in which there were 5×10^3 grains in the cross section ruptured at room temperature with practically 100% reduction in area and again no cavities were observed on the fracture surface. There was, however, no doubt that in specimens in which there were 5×10^3 grains in the cross section very extensive cavitation occurred when the deformation was carried out at 85°K (Fig. 6). The nature of the fracture did not conform with any one of the standard types of ductile fracture (cup and cone, double cup, knife edge etc.) but instead displayed characteristics of nearly all these types at one point or another. Cavities were not formed over the entire fracture surface but were found most frequently on those parts which were comparatively flat and mainly transverse to the stress axis. Rough estimates of the cavity population in these parts were obtained by counting the number of cavities observable on stereofractographs and for this material there were between 10 and 20 cavities per square millimeter.

When the grain size was increased until there were only 500 grains in the cross section of the tensile specimen the fracture tended to become more predominantly of the irregular knife edge type and the area over which cavitation occurred was very greatly reduced (Fig. 7). The density of cavities on this area was estimated and found to be about 20 per square millimeter.

On further increasing the grain size until there were only 5 or 6 grains in the cross section specimens deformed at 85°K failed with an irregular knife-edge fracture and only rarely could cavities be detected on the fracture surfaces.

(c) Lead. In order to establish whether these same trends were observable in other metals of very high purity a series of experiments was carried out using lead. Lead cast in air contains many oxide inclusions and when this material is strained at room temperature, cavitation at oxide particles may be so extensive that the fracture is of the transverse-planar type and the fracture surfaces have a honeycomb-like appearance. When strenuous efforts were made to eliminate the oxide particles by the hydrogen-casting technique described in Section 2, cavitation was practically non-existent and the material ruptured with almost 100% reduction in area. However, when this same hydrogen-cast material was strained at 85°K, marked cavitation again occurred and the extent of cavitation on the fracture surfaces (Fig. 8) was comparable to that produced at low temperatures in fine grained superpurity aluminium (Fig. 5). The grain size in the specimen shown here was such that there were approximately 100 grains in the cross section.

The effect of grain size on the incidence of cavitation in lead at 85°K was also similar to that found with aluminium, for in specimens which contained only twenty grains in the cross section, failure was of the irregular-knife-edge type and few cavities were observed on the fracture surfaces (Fig. 9).

4. Discussion.

It is clear from the present results that provided the conditions of deformation are favourable, cavities are formed in the necked regions and on the fracture surfaces of materials of very high purity. Three factors have been found to favour cavity formation:-

(i) The presence of impurities. By using zone-refined aluminium instead of the less pure superpurity aluminium, a very marked reduction in the extent of cavitation was obtained and it must be concluded from this that a further reduction in cavitation would be achieved if material of even higher purity was used. The present results are therefore exactly analogous to those discussed by Cottrell(5) on tough-pitch and oxygen-free copper except that they apply to materials in which the impurity levels are orders of magnitude lower.

(ii) The use of fine grained specimens, i.e. a large number of grains in the cross section. This appears to be a necessary, although not a sufficient, requirement for cavity formation. Availability of the high-purity materials limited the number of experiments which could be carried out on the effect of grain size but it would seem that unless the number of grains in the cross section was high, the stress distribution across the necked region was so unsymmetrical that knife-edge-type fractures occurred before cavities could be formed. It has not been possible to accurately determine the number of grains per cross section below which cavities will not be formed but it is clear that the figure for lead is quite different from that for aluminium. For lead the critical number is between 25 and 100 whereas for aluminium it is probably fairly close to 500 grains per cross section. This probably explains why Chin et al.(8) were unable to detect cavities in zone-refined aluminium deformed at 85°K for although the possibility that their material was of much higher purity than that used here cannot be ignored, it would seem from the present results that the grain size which they used (approximately 100 grains per cross section) was too large.

(iii) Deformation at low temperatures. With zone-refined aluminium and high-purity lead, cavitation was observed only after deformation at liquid air temperatures and even with the very fine grained specimens of aluminium, no cavities at all were observed after fracture at room temperature. This trend towards increased cavitation at low temperatures was also shown with superpurity aluminium. These observations, like those of Chin et al.(8) are clearly contrary to the predictions of a mechanism of cavity formation involving vacancy condensation and it must be concluded that such a mechanism is unacceptable. Chin et al. interpreted a similar effect to this which they found in inclusion populated aluminium as being due to the flow stress of the aluminium increasing more rapidly than the particle-matrix cohesion as the temperature is lowered. An alternative explanation is that, as Chen(11) has suggested, high stresses are built up around inclusions by dislocations piling up against them. At low temperatures these high stresses result either in fracture of the inclusion or in a breakdown of inclusion-matrix cohesion whereas at room temperature cross slip relieves the stress before cavities can be produced.

The conclusion to be drawn from these observations is that if it is proposed to test, with the utmost rigour, the theory that materials which are free from inclusions do not exhibit cavitation and rupture with 100% reduction in area, the experiments must be carried out at low temperatures and on very fine-grained specimens.

Many attempts were made to detect cracks of the type which Beevers and Honeycombe(10) reported finding on heavily deformed high-purity aluminium but none was successful. The observations reported here appear to be consistent with cavitation being associated with impurity particles in the matrix and little support can be given to their suggestion that cracks, nucleated in the absence of inclusions, play an important part in the ductile fracture of high-purity metals.

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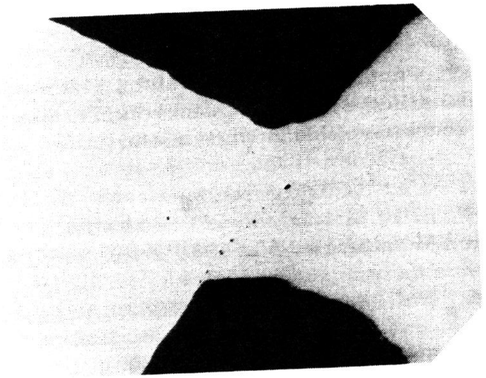

Figure 1. Metallographic section of neck in superpurity aluminium deformed at room temperature. 40X

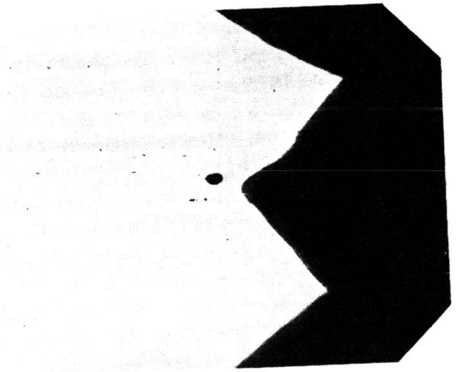

Figure 2. Longitudinal section through fractured superpurity aluminium deformed at 373 °K. Fine grained. 40X

Figure 3. Longitudinal section through fractured superpurity aluminium deformed at 85 °K. Fine grained. 40X

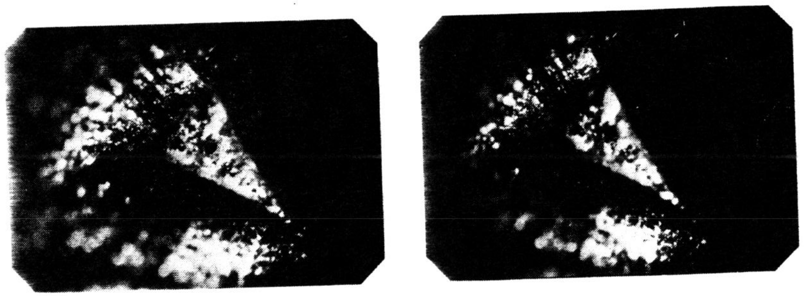

Figure 4.

Stereofractograph of specimen fractured at 373 °K. Fine grained superpurity aluminium. 22X

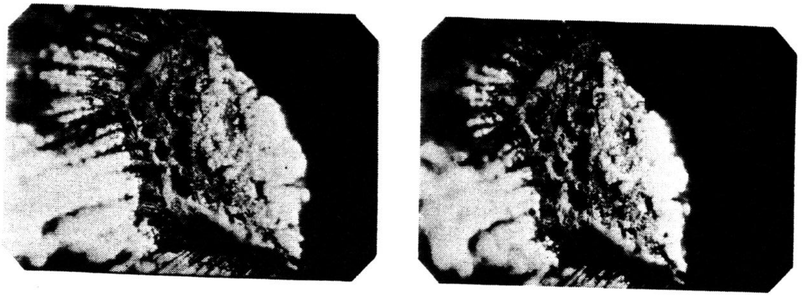

Figure 5. Stereofractograph of specimen fractured at 85 °K. Fine-grained superpurity aluminium. 22X

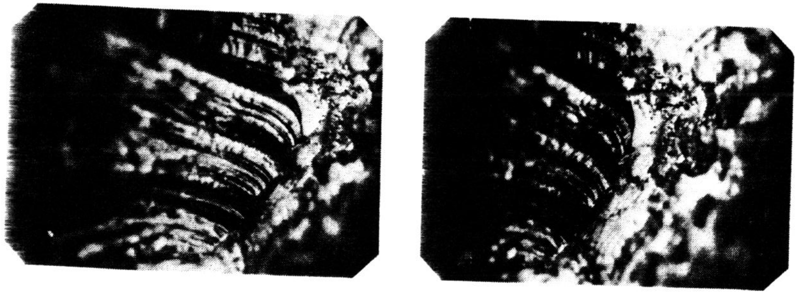

Figure 6. Stereofractograph of specimen fractured at 85 °K. Fine-grained zone-refined aluminium. 22X

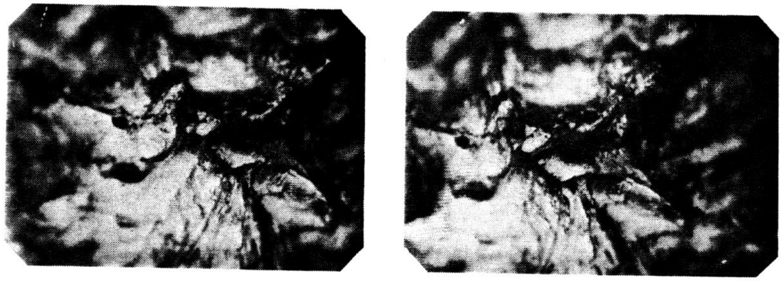

Figure 7. Stereofractograph of specimen fractured at 85 °K. Medium grain size zone-refined aluminium. 22X

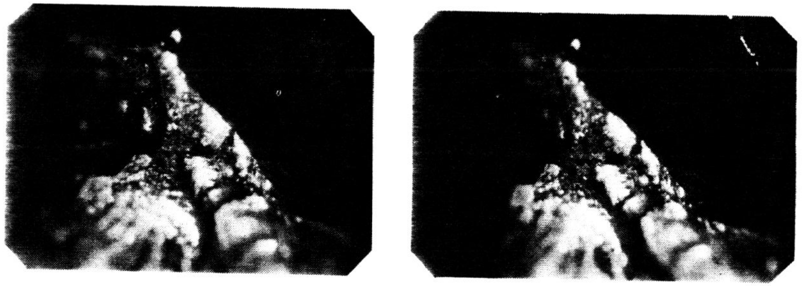

Figure 8. Stereofractograph of specimen fractured at 85 °K. Fine-grained high purity lead. 22X

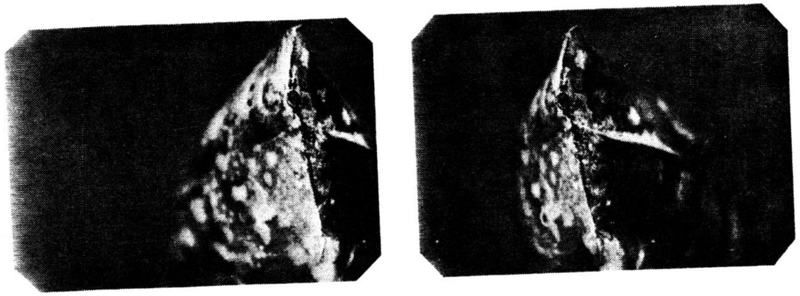

Figure 9. Stereofractograph of specimen fractured at 85 °K. Coarse-grained high-purity lead. 18X